REMEDIAL INVESTIGATION / FEASIBILITY STUDY
WORK PLAN
REVISION 4.0

FORMER VERMONT BOSCH SITE
FOUNTAIN INN, SOUTH CAROLINA

Prepared for:

ROBERT BOSCH TOOL CORPORATION

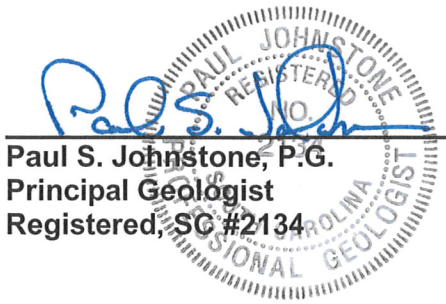
1800 West Central Road
Mount Prospect, Illinois 60056

Prepared by:

AMEC Environment & Infrastructure, Inc.
555 N. Pleasantburg Drive, Suite 202
Greenville, South Carolina 29607

AMEC Project 6251121007.01.01

May 31, 2012



Paul S. Johnstone, P.G.
Principal Geologist
Registered, SC #2134

Christopher H. Bruce
Senior Professional

TABLE OF CONTENTS

LIST OF TABLES

Table

LIST OF FIGURES

Figure

LIST OF ACRONYMS

AMCE	AMEC Environment & Infrastructure, Inc.
AOC	Area of Concern
ARAR	Applicable or Relevant and Appropriate Requirement
AST	Aboveground Storage Tank
BRA	Baseline Risk Assessment
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CERCLIS	Comprehensive Environmental Response, Compensation and Liability Information System
COC	constituent of concern
FSAP	Field Sampling and Analysis Plan
HASP	Health and Safety Plan
MACTEC	MACTEC Engineering and Consulting, Inc.
MCL	Maximum Contaminant Level
MCLG	Maximum Contaminant Level Goal
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NHPA	National Historic Preservation Act
NOAEL	No Observed Adverse Effect Level
O&G	oil and grease
OSHA	Occupational Safety and Health Act
PAH	polynuclear aromatic hydrocarbon
PQL	Practical Quantitation Limit
QA/QC	Quality Assurance/Quality Control
QCSR	Quality Control Summary Report
RAA	Remedial Action Alternatives
RAO	Remedial Action Objectives
RBC	Risk-Based Concentrations
RBTC	Robert Bosch Tool Corporation
RCRA	Resource Conservation and Recovery Act
RGO	Remedial Goal Options
RI/FS	Remedial Investigation and Feasibility Study
RSL	Regional Screening Level
RUD	Rural Development District
SCDHEC	South Carolina Department of Health and Environmental Control
SDWA	Safe Drinking Water Act
SMDP	scientific/management decision point
SVOC	semi-volatile organic compound

LIST OF ACRONYMS - Continued

TAL	Target Analyte List – A list of 24 inorganic compounds (metals, metalloids, and cyanide) that are defined by the USEPA Contract Laboratory Protocol (CLP) Scope of Work.
TCLP	Toxicity Characteristic Leaching Procedure
TT	Treatment Technique
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UST	Underground Storage Tank
VAC	Vermont American Corporation
VOC	volatile organic compound
WQC	Water Quality Criteria

1.0 INTRODUCTION

This document presents the Remedial Investigation and Feasibility Study (RI/FS) Work Plan (Work Plan) for the Robert Bosch Tool Corporation (RBTC) Former Vermont Bosch Site (Site) located in Fountain Inn, South Carolina. This Work Plan describes the proposed work to be conducted by AMEC Environment & Infrastructure, Inc. (AMEC); formerly MACTEC Engineering and Consulting, Inc. (MACTEC), on behalf of RBTC under Voluntary Cleanup Contract (VCC) #05-5613-RP. RBTC, a division of Robert Bosch, LLC, is the successor to Vermont American Corporation (VAC), who manufactured screwdrivers and spade bits at the Site. This Work Plan details the planned scope of investigation activities and feasibility study required to attain an appropriate level of understanding of the nature and extent of contaminants at the Site, assess potential risks to human health and the environment, and develop and evaluate remedial action alternatives.

1.1 BACKGROUND

The Site is located at 800 Woodside Avenue in Fountain Inn, Greenville County, South Carolina. A site location map is included as **Figure 1**. The Site is located northwest of the intersection formed by South Carolina Highway 418 (McCarter Road) and Woodside Avenue. Access to the site is from either South Carolina Highway 418 (McCarter Road) or Woodside Avenue. The site is presently developed with a 124,793 square foot former manufacturing facility where screwdrivers and spade bits were manufactured.

The plant is sited in the approximate center of the property. Parking areas are located southeast of the plant followed by a mowed grassy field with a stand of trees between the parking area and McCarter Road. Northeast of the plant are landscaped areas, mowed grassy fields, and Woodside Road. Northwest of the plant are a mowed grassy field and woodlands. Southwest of the plant are a tank containment area, access road, and hazardous waste accumulation area with mowed grassy areas in between.

1.2 PREVIOUS PLANT OPERATIONS

The site was developed with the manufacturing plant in 1984 and operations commenced in 1985 as Rosco Tools, a division of VAC which subsequently became RBTC. Screwdrivers were manufactured initially and spade bit manufacturing was added in 1992. Nickel plating and associated wastewater pretreatment was present in the facility from 1985 to the early 1990s. A self-contained vapor degreaser was used at the facility from 1985 to the early 1990s. Manufacturing operations ceased in November 2003 and the facility was vacant until it was sold in September 2005 to Fountain Inn Investments I, LLC (assignee of Liberty Property Development Corporation).

Three primary manufacturing processes were performed at the site: manufacture of screwdriver handles and other specialty items; screwdriver head manufacturing; and spade bit manufacturing. The process areas discussed below are shown on **Figure 2**.

1.2.1 Screwdriver Handles and Other Specialty Items

Raw materials included flake cellulose acetate in 50 pound bags, scrap plastic from outside sources, plasticizer (Diethyl Phthalate, or DEP), and various colorants and powders. The cellulose acetate and scrap plastic were stored in Raw Materials Storage, the DEP was stored in two 6,000-gallon aboveground storage tanks (ASTs) located in a Tank Containment Area outside the plant, and the colorants and powders were stored in the Compounding Room. The DEP was transported to the Compounding Room from the Tank Containment Area by overhead piping.

The cellulose acetate and/or scrap plastic were combined with the DEP to form plastic pellets in two compounding lines (one for virgin cellulose acetate and one for scrap plastic). The plastic pellets underwent a water quench prior to being stored in four 30,000-gallon silos located outside the Compounding Room.

Water vapor containing DEP was condensed and accumulated in a settling tank in the Compounding Room. The DEP was skimmed off the settling tank, pumped into drums, and recycled in the compounding process. Initially, from 1985 to 1996, the water from the settling tank was discharged directly to the sanitary sewer. After 1996, water from the

settling tank was periodically pumped into drums for off-site disposal. The settling tank bottoms were cleaned out approximately every two years, and containerized in drums for off-site disposal. Plastic sweepings and DEP waste entrained in oil dry were accumulated and containerized in drums for off-site disposal. An exhaust blower system was used to remove vapors from the Compounding Room. A baghouse located outside the Compounding Room collected cellulose acetate dust, which was disposed in a dumpster on the site. Bag filters in the piping were containerized in drums for off-site disposal.

The plastic pellets in the silos were pumped to extruding machines that used electric burners to heat and melt the plastic, which were extruded as plastic rods that ultimately would become screwdriver handles or other specialty items. Flexible hoses were located on the extruders to add striping to the rods. The hoses were cleaned with acetone. The spent acetone was accumulated for subsequent distilling and reuse. Each extruder had a quench trough fed with a water hose. Overfills were collected in floor drains that discharged to the sanitary sewer.

The finished rods were transported to Rod Handle Storage prior to further processing in the Handle Machining Lathes. The Handle Machining Lathes used an aqueous coolant stored in a sump located on each lathe. The containers were pumped out manually and transported to an oil/water separator located in the Grinding Area. The oil from the separator was accumulated in drums and subsequently transferred to the Hazardous Waste Accumulation Building pending off-site disposal. The water from the separator was discharged to the sanitary sewer. The oil/water separator was previously located near the Quality Control (QC) lab located in the mezzanine under the stairs near the Acetone Room.

Plastic turnings from the lathes were captured by an overhead vacuum system that transported the waste plastic to the Grinding Area where the plastic was ground to a usable size and then transferred to the Plastic Scrap Regrinding Room. Floor sweepings in the Handle Machining Lathe area were collected and disposed in a dumpster. The sump bottoms at each lathe were drummed and accumulated for subsequent off-site disposal.

Some finished rod handles were further processed in the Drilling area with pneumatic drilling machines to drill special holes. Plastic turnings were captured in the same fashion as at the lathes. Floor sweepings in the Drilling area were collected and disposed in a dumpster. After drilling the holes, the rod handles were processed in the Handle Wash area where they were first placed in a tumbler to remove plastic burrs and then washed in a 180-gallon barrel washer containing water and detergent. The rod handles were then placed in a 120-gallon rinse tank. The wash and rinse water was discharged to sanitary sewer every couple of days. Bag filters in the wash line and rinse line were disposed in a dumpster.

Following drilling and washing, the vast majority of the rod handles went to the Polishing Room where they were polished with acetone. The acetone was located in a sump in the Acetone Line that was heated to create vapors. The handles were transported through the vapors on racks in a conveyor belt system. The majority of the acetone vapor condensed and returned to the sump while other vapors discharged to the air. An acetone dip tank and paint dip tank were also located in the polishing room. The acetone dip tank was used to polish mallet heads and other specialty items. The paint dip tank was used to paint ends and noses for special order items.

Virgin acetone was stored in a 6,000 gallons underground storage tank (UST) located in the Tank Containment Area and initially piped to the Acetone Line via underground piping. At a later date, the underground piping was abandoned in place and replaced with aboveground piping.

The acetone sump was drained once a week into drums that were transferred to the Hazardous Waste Accumulation Building. The drums were initially disposed off site. Later, the acetone was reclaimed by an outside contractor who came in approximately every 90 days to distill the acetone. The recycled acetone was pumped to the UST and the still bottoms were containerized in drums and stored in the Hazardous Waste Accumulation Building until they were disposed off site.

After polishing, most handles were transferred to the Handle Storage area. Some handles were painted using a dip tank in the Painting area or with a Binks spray paint system also located in the Painting area. In the early 1990s, the Binks spray paint system was

transferred to another RBTC facility. The paint used in the dip tank and Binks system was an oil-based paint thinned with acetone. Cleaning of spray nozzles and other parts was performed using acetone. The paint/acetone mixture from painting and cleaning was containerized in drums, transferred to the Hazardous Waste Accumulation Building, and recycled by an outside contractor.

On some handles and specialty items, printing was affixed to the plastic materials. Hot stamp printing using a foil tape was performed in the Hot Stamp area. Spent foil was accumulated and disposed in a dumpster. In the Ink Pad Printing area, a pad printer using organic ink was used to print the plastic materials. The ink was thinned with ink-specific thinners. Isopropyl alcohol was used to clean inks from the plastic materials so that the plastic wouldn't be affected. Acetone was used to clean printer components and was accumulated in a satellite accumulation area nearby. The spent acetone was processed as described previously. Waste paint rags and plastic cups and cardboard coated with paint were containerized in drums in a satellite accumulation area before they were transported to the Hazardous Waste Accumulation Building for off-site disposal.

1.2.2 Screwdrivers

Raw materials consisted of coiled carbon steel. Flat-head screwdrivers were fashioned in the Press area using mechanical, hydraulic presses. Phillips-head screwdrivers were fashioned in the ESCO Manufacturing area using cutting oils. Steel scrap from the processes was collected in totes and when full were transported to the Scrap Metal Rolloff for off-site disposal. Waste oils were drummed and transported to the Hazardous Waste Accumulation Building for off-site disposal.

Vapor degreasing was performed from 1985 to 1992/1993 in a self-contained system that initially utilized 1,1,1-Trichloroethane and later Freon 113. Vapor degreasing took place in what is now the ESCO Manufacturing area. At the time vapor degreasing operations were discontinued, the floor drains in the area were plugged. The vapor degreasing was replaced by an aqueous two-stage alkaline dip degreaser.

The screwdrivers were then processed in a heat treat line located in the Heat Treat area. From 1985 to 1988/89 heat treating was accomplished using a vacuum furnace.

Following heat treatment in the vacuum furnace, the screwdrivers were quenched in an oil bath then processed through wash, rinse, and drying tanks. The vacuum furnace was replaced in 1988/89 with a heat treat system that used a sodium nitrate quench bath and two rinse tanks. Periodically, the quench tank was cleaned of caked quench residue, which was disposed in a dumpster. Prior to 1997, the cleaning rinse water and water from the rinse tanks were discharged to the sanitary sewer. After 1997, the cleaning rinse water and water from the rinse tanks were pumped to the ground outside the facility.

Following heat treating, the majority of the screwdriver blades were processed in the Grit Blasting area to remove scale and burrs. There were three grit blasters that used steel shot as the blast media. One of the grit blasters was used to blast the tips of screwdriver blades that had been nickel plated at the facility or chrome-plated screwdriver blades that were received from other RBTC facilities. Spent blast media was collected in traps in the back of each machine and periodically emptied into the Scrap Metal Rolloff. Dust generating during the grit blasting operations was processed through a Metals Baghouse located outside the facility. Filters from the Metals Baghouse were initially disposed in the Scrap Metal Rolloff but subsequently were rebuilt and reused.

The screwdrivers and spade bits were next processed in the Screwdriver and Spade Bit Grinding area where the final faces and edges were ground. From 1985 to 1988, dry grinding was performed on screwdriver blades. The dry grinding operations were connected to the Metals Baghouse. Wet face grinding was also performed. Each wet-face grinder had an individual sump to collect grinding swarf and grinding oil. The sumps were cleaned out on a semi-annual basis and the grinding swarf was disposed in the Scrap Metal Rolloff. Sometime in the late 1980s, the wet face grinders were connected to a central coolant system. The grinding swarf was removed from the central coolant system and disposed in the Scrap Metal Rolloff and the grinding oil was recirculated. In 1992, with the addition of the spade bit line, a below-grade Henry Filter was installed in what was previously the wastewater pre-treatment room. At this time, all grinding operations were attached to the Henry Filter. Grinding swarf was removed from the Henry Filter and disposed in the Scrap Metal Rolloff and grinding oils were recirculated. On at least two occasions, all of the grinding oil in the Henry Filter system was pumped out and disposed off site.

After the grinding operations were completed, the screwdriver blade and spade bit surfaces were given their final treatment which included nickel plating, vibratory finishing, black painting, or lacquer coating.

Nickel plating was performed from 1985 to the early 1990s in the Nickel Plating Area. The process included barrel plating and reverse osmosis was used to reclaim the nickel. Wastewater from the plating process flowed to a settling tank then through a filter press. Solids from the filter press were containerized in drums for off site disposal. The resulting wastewater was treated for pH in a wastewater pretreatment system before being discharged to the sanitary sewer. The wastewater pre-treatment system was located in what was subsequently the Henry Filter Area. At the time that nickel plating was discontinued, the wastewater pre-treatment system was removed.

Screwdriver blades and spade bits that required cleaning and polishing prior to plating were processed in the Vibratory Finishing area. Some blades and bits were nickel plated at the facility and others were shipped to other RBTC locations for plating. There were two vibratory bowls that contained a ceramic and plastic media for polishing and a detergent for cleaning. Spent vibratory media was collected in a moat around each vibratory bowl and was periodically shoveled out and disposed off site.

Other screwdriver blades and spade bits were coated in a black paint process in the Oven and Black Painting Line. The blades and bits were dipped in a tank of paint and then cured in a gas-fired oven. The spent paint was removed from the tank and disposed off site twice a year.

The rest of the screwdriver blades and spade bits were coated in a drip spin lacquer process for rust prevention in the Dip Spin Lacquer area. Initially, an oil-based lacquer was used in a two-step process was used where the parts were first dipped in the lacquer, removed from the dip tank, placed in a spinner, spun, and then removed. Later a space age process with a solvent-based lacquer was used where the parts were dipped in the lacquer and then baked in an oven. Eventually, the solvent-based lacquer was replaced with a water-based lacquer. Since 1990, a centrifugal spin process was used similar to the initial lacquer coating process.

Following completion of the finishing process, the screwdriver blades were transported to the Bulk Screwdriver Storage area.

1.2.3 Spade Bits

Raw materials consisted of carbon steel blanks. The spade bits were stamped in the Press Area and then underwent similar heat treating, grit blasting, grinding, and finishing processes as described for the screwdrivers. Following completion of the finishing process, the spade bits were transported to the Packaging Area to be packaged and then shipped.

1.2.4 Assembly

Screwdriver blades, rod handles, and specialty items were assembled in the Screwdriver Assembly area. The assembled screwdrivers and specialty items were transported to the Packaging Area to be packaged and then shipped.

1.3 SITE AND PROPERTY BOUNDARIES

The Site is approximately 22.85 acres in size and consists of a single parcel of land. The Site and adjacent property boundaries are illustrated on **Figure 3**.

1.4 PREVIOUS INVESTIGATIONS AND REPORTS

Several environmental investigations have been conducted by AMEC, formerly MACTEC, at the Site. They include the following, which have previously been provided to the South Carolina Department of Health and Environmental Control (SCDHEC), Bureau of Land and Waste Management.

- Preliminary Site Contamination Assessment, Acetone Release Site, Vermont American Corporation Facility, Fountain Inn, South Carolina (MACTEC Project 30290-6-7856.02), dated December 10, 1996;
- Report of Acetone Tank and Line Testing and Diethyl Phthalate Line Testing, Vermont American Corporation Facility, Fountain Inn, South Carolina (MACTEC Project 30290-7-8046.01), dated August 15, 1997;

- Underground Storage Tank (UST) Assessment Report, Vermont American Corporation, Fountain Inn Division, Fountain Inn, South Carolina, SCDHEC Permit #04235 (MACTEC Project 30200-1-9316-04-917), dated August 21, 2002;

- Report of Phase II Environmental Site Assessment, Vermont American Corporation, Fountain Inn Division, 800 Woodside Avenue, Fountain Inn, South Carolina (MACTEC Project 30200-1-9316-04-917), dated February 4, 2003;

- Second Revised Draft Report of Environmental Services, Vermont American Corporation, Fountain Inn Division, Fountain Inn, South Carolina (MACTEC Project 30200-1-9243-01-917), dated February 5, 2003;

- Results of Field Screening Groundwater Sampling, Robert Bosch Tool Corporation (former Vermont American Corporation), Fountain Inn Division, Fountain Inn, South Carolina (MACTEC Project 3020019316-2, Task 04), dated October 8, 2003;

- Results of Field Screening Sampling, Robert Bosch Tool Corporation (former Vermont American Corporation), Fountain Inn Division, Fountain Inn, South Carolina (MACTEC Project 3020019316-2, Task 05), dated April 18, 2005.

Based on the data collected during these investigations, soils, groundwater, and surface water at the Site have been found to contain concentrations of volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), metals (arsenic, barium, cadmium, chromium, lead, nickel, and selenium), nitrate, nitrite, and total petroleum hydrocarbons-oil & grease (TPH O&G) above the laboratory method detection limits. It should be noted that no samples were collected and analyzed for polychlorinated biphenyls (PCBs) or pesticides as there is no record of PCB or pesticide use during the manufacturing operations at the facility. A detailed account of these previous investigations is provided in Section 3.0 of this Work Plan. Concentration maps for selected constituents (acetone, chromium, and nickel) are included in **Appendix A**. The criteria for selection included five or more detections in surface samples and exceedance of one or more risk-screening levels.

1.5 OBJECTIVES OF THE RI/FS

The primary objectives of the RI/FS are as follows:

- Determine the source, nature, and extent of contaminated media (soil, groundwater, surface water, and sediment) present at the Site.
- Determine if the concentrations of constituents present in Site media present an unacceptable risk to human health or the environment.
- Develop and evaluate alternatives for remedial action to prevent, mitigate, or otherwise respond to the migration or the release or threatened release of hazardous substances, pollutants, or contaminants at or from the Site.

2.0 SITE PHYSICAL SETTING

A consideration of surface and subsurface drainage and geology are of interest since they provide an indication of the direction that contamination, if present on or off the site, could be transported.

2.1 REGIONAL/SITE GEOLOGY

The site is located in the Piedmont Physiographic Province. Based on published literature, the site is underlain at depth by the Inner Piedmont block, which consists of a stack of thrust sheets consisting of a variety of gneisses, schists, and amphibolites. According to the *Geologic Map of the Crystalline Rocks of South Carolina*, the bedrock in the site area is described as biotite schist (MpCs) consisting primarily of gray to black, fine- to coarse-grained, scaly biotite schist and biotite-muscovite-oligoclase schist, with thin layers of biotite gneiss, granitoid gneiss, quartz schist, quartzite, marble, calc-silicate rocks, and hornblende schist. The bedrock is generally overlain by a mantle of residual soil, referred to as “saprolite”, formed by the in-place weathering of the bedrock. Fractures, joints, and the presence of less resistant rock types facilitate weathering. The typical soil profile consists of clayey soils near the ground surface transitioning to sandy silts and silty sands that generally become harder with depth to the top of the parent rock. Bedrock outcrops have not been observed on the site and major geologic features, such as faults, are not documented within the site area in the reviewed published literature.

Information regarding the area soils was obtained from the *Soil Survey of Greenville County, South Carolina*. The soils beneath the site are mapped as the Appling sandy loam (2 to 6% slopes) and the Wehadkee soils. The Appling series consists of gently sloping soils that are well drained. These soils formed in material that weathered from granite, gneiss, and schist. The native vegetation is a mixed hardwood and pine forest that has an understory of vines, briers, and native grasses. In a representative profile, the surface layer is dark grayish-brown sandy loam about eight inches thick. The subsoil is about 36 inches thick. In sequence from the top, the subsoil is light yellowish-brown clay, about six inches thick; yellowish-brown clay, about six inches thick that has that has reddish-yellow mottles; reddish-yellow clay, 12 inches thick, that has strong-brown and red mottles; and mottled brownish-yellow, strong-brown, and red clay 12 inches thick.

The underlying material, extending to a depth of 62 inches, is mottled red, yellowish-brown, and gray, weathered gneiss rock. Permeability is moderate and the available water capacity is medium. The Appling sandy loam is present on broad ridges.

The Wehadkee soils are in poorly drained, elongated areas on the flood plain of creeks and rivers. The elongated areas are generally adjacent to the uplands. Wehadkee silt loam and closely similar, poorly drained to moderately well drained soils characterize this mapping unit.

Subsurface soils at the site consist generally of sandy silts, with some clay and fine to coarse sand. Lithologic cross-sections for the site are provided in **Figure 4**.

2.2 REGIONAL AND SITE HYDROGEOLOGY

In the Piedmont Physiographic Province, groundwater generally occurs under water table conditions and is stored in the overlying mantle of residuum and in the structural features present in the underlying rock (i.e., joints, fractures, and faults). Recharge to the water table is primarily by precipitation infiltrating the upper soils and percolating downward, under the influence of gravity, to the groundwater table. Typically the groundwater is not a level surface, but a subdued replica of the land surface. Also, depth to the water table is variable, being dependant on many factors which include: the amount of rainfall, the permeability of the residuum, the extent of fracturing in the underlying rock, and the amount of groundwater being pumped from the area.

Groundwater generally flows in directions subparallel to the ground surface slopes and under the influence of gravity toward points of discharge such as creeks, swamps, drainage swales, or pumped groundwater wells. Significant surface drainage features as well as subsurface stratigraphy can affect groundwater conditions. Based on review of the USGS 7.5-Minute Series, Topographic Map, Fountain Inn, South Carolina Quadrangle, shallow groundwater flow is expected to flow generally from the northeast to the southwest in the site area, ultimately discharging to Stoddard Creek. There are three groundwater monitoring wells located near the facility building on the site (B-1 through B-3). Depth to groundwater measurements obtained from these three wells in June 2003 during previous environmental activities performed by AMEC indicate that groundwater

flow at the Site should generally be to the southwest. A water table elevation contour map for the June 2003 water levels is presented in **Figure 5**. Monitoring well construction data for the wells is included in **Table 1**.

2.3 LAND USE

The area surrounding the Site is a mix of industrial and commercial properties, residential properties, and undeveloped land. The site is bordered to the northeast by Woodside Avenue, a United States Post Office, and residential properties. The site is bordered to the southeast by McCarter Road (South Carolina Highway 418), residential properties, industrial properties, and undeveloped land. The site is bordered to the northwest by baseball fields (recreation complex), residential properties, and undeveloped land. The site is bordered to the southwest by an industrial property and undeveloped land.

2.4 SURFACE HYDROLOGY

The site is located along the southwest flank of northwest to southeast trending topographic ridge. Two smaller topographic ridges, trending generally northeast to southwest, lie in the northwestern and southeastern portions of the site separated by a small topographic low. According to the USGS 7.5-Minute Series, Topographic Map, Fountain Inn, South Carolina Quadrangle, the elevation of the site ranges from approximately 258 meters (846 feet) above the National Geodetic Vertical Datum (NGVD) of 1929 in the northern portion of the site to approximately 249 meters (817 feet) in the southern portion of the site. Surface water drainage in the undeveloped portions of the site is estimated to be overland sheet flow controlled by surface topography. A series of storm water catch basins are present around the periphery of the facility building that receives drainage from the areas surrounding the building. The storm water catch basins and subgrade piping direct stormwater flow to two outfalls; one located in the southern portion of the site (Outfall 001) and one located in the northern portion of the site (Outfall 002). A map showing the surface water bodies in the site area is presented in **Figure 6**.

3.0 IDENTIFICATION OF ADDITIONAL DATA NEEDS

To assess the need for additional site data, a review of existing site information was conducted. A summary of this review is described below. Based on this information, a review of the areas of concern as defined, and the preliminary Applicable or Relevant and Appropriate Requirements (ARARs), data gaps and additional data needs have been identified.

3.1 SUMMARY OF PREVIOUS REPORTS

The following is a brief summary of the soil, groundwater, and surface water data complied from several environmental investigations that have been conducted at the site. The results of these investigations are described in each section. The soil sampling and groundwater sampling locations associated with previous investigations conducted at the Site are illustrated on **Figure 7**. The proposed soil and groundwater sampling locations contemplated in this Work Plan are illustrated on **Figure 8**. Data tables consisting of laboratory analytical results (detections only) are referenced in each section. The laboratory methods for all samples analyzed are noted on each specific table. Unless otherwise specified in the following text, soil and groundwater samples analyzed by USEPA Method 8260B include Target Compound List (TCL) VOCs and soil and groundwater samples analyzed by USEPA Method 8270C include TCL SVOCs. Soil and groundwater samples analyzed by USEPA Methods 6010B/7471A/7470A include the eight Resource Conservation and Recovery Act (RCRA) metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) and nickel. Previous sampling locations where the laboratory's Practical Quantitation Limit (PQL) exceeds appropriate risk-screening levels for specific constituents is summarized on **Table 2**.

The environmental sampling data and laboratory analytical results presented in this Work Plan represent all of the information collected by AMEC for RBTC at the site. There was a Phase I Environmental Site Assessment (ESA) and a Limited Phase II ESA conducted by URS Corporation (URS) in April and May 2005, respectively. The investigations were conducted for Liberty Property Trust, the prospective purchaser of the property, as part of a due diligence investigation. AMEC has only been provided with the text of each report and efforts to obtain the figures, tables, and laboratory data referenced in the reports has

been unsuccessful to date. At such time as AMEC receives this data, copies will be provided to SCDHEC.

According to the URS Limited Phase II ESA report, 18 direct-push borings were advanced at the site for the purpose of collecting soil and groundwater samples. Twelve soil borings were advanced inside the facility at the Former Vapor Degreasing Area (two borings), the ESCO Manufacturing Area (one boring), the Heat Treat Area (one boring), the Former Nickel Plating Area (one boring), the Henry Filter/Former Waste Water Pre-Treatment Room and Grinding Area Floor Drains (one boring), the Plastic Scrap Regrinding Room (one boring), the Compounding Room (three borings), and the Acetone Room (two borings). Seventeen soil samples were collected from the borings and analyzed for TCL VOCs, TCL SVOCs, and the eight RCRA metals.

No VOCs or SVOCs were reportedly detected by the laboratory in the soil samples collected at the Former Vapor Degreasing Area, ESCO Manufacturing Area, Heat Treat Area, Former Nickel Plating Area, Henry Filter/Former Waste Water Pre-Treatment Room and Grinding Area Floor Drains, and the Plastic Scrap Regrinding Room. DEP was detected in three soil samples collected from two of the borings advanced in the Compounding Room at concentrations ranging from 1,900 micrograms per kilogram (µg/kg) to 63,000 µg/kg. Acetone was detected in one soil sample collected from one of the borings advanced in the Acetone Room at a concentration of 7,200 µg/kg. The metals arsenic, barium, cadmium, chromium, lead, and selenium were detected in one or more of the soil samples collected from the borings. According to URS, the reported concentrations of metals were below their respective USEPA Region IX remediation goals and/or naturally occurring background concentrations observed at the site.

Six soil borings were advanced outside the facility at the Tank Containment Area (two borings) and the sanitary sewer system discharge piping downstream from the Former Oil/Water Separator Area (four borings). The borings were advanced for the purpose of collecting soil samples and installing temporary monitoring wells. Twelve soil samples and six groundwater samples were collected from the borings. The groundwater samples were requested to be held by the laboratory pending the soil sample analytical results. Subsequently, two groundwater samples were analyzed; one from the temporary monitoring well installed at the Tank Containment Area and one from the well installed

along the sanitary sewer system discharge piping. The soil and groundwater samples were analyzed for TCL VOCs, TCL SVOCs, and the eight RCRA metals.

No VOCs or SVOCs were detected by the laboratory in the soil samples or groundwater samples collected from the exterior of the facility. The metals arsenic, barium, chromium, lead, and mercury were detected in one or more soil samples collected from the borings. According to URS, the reported concentrations of metals in soils were below their respective USEPA Region IX remediation goals and/or naturally occurring background concentrations observed at the site. The URS report made no statement with respect to the metals concentrations detected in the groundwater samples, if any.

3.1.1 Review of Existing Soil Data

In response to a suspected release from subgrade piping at an acetone UST in a tank containment area at the site in October 1996, ESE Environmental, Inc. (ESE) collected soil samples from the subgrade piping. The double-walled product piping is above ground from the UST to just outside the containment area and underground to the building where it emerges and enters the building. At two below-ground elbows (one near the containment area and one near the building), which were not double-walled and judged to be likely areas for a release to occur, soil samples were collected to evaluate the suspected release. Six hand auger borings were performed near the subgrade elbows and six soil samples were collected from the borings. The samples were analyzed for acetone by USEPA Method 8260 and for methanol by USEPA Method 8000M. Acetone was detected in five of the borings at concentrations ranging from 2.70 milligrams per kilogram (mg/kg) to 242 mg/kg. In November 1996 MACTEC performed six soil test borings near the tank containment area and along the subgrade piping. Three borings were performed near the tank containment area and three borings were performed along the subgrade piping. One soil sample was collected from each boring. The samples were analyzed for acetone by USEPA Method 8260 and for methanol by USEPA Method 8000M. Acetone was detected in two of the soil samples at concentrations of 0.127 mg/kg and 0.584 mg/kg, respectively. The results of this assessment were documented in MACTEC's *Preliminary Site Contamination Assessment* (1996) referenced in Section 1.3. The laboratory analytical results of the soil sampling performed along the subgrade piping is summarized on **Table 3**. The sample locations are shown on **Figures 7 and 9**.

In June 2001, an area of stressed vegetation was observed by VAC personnel along the northwest side of the manufacturing building. The stressed vegetation was reportedly related to the discharge of fluids from the recent cleaning of a heat treat bath (quench tank) and rinse tanks. The heat treat bath used a salt as the quench medium. The salt was identified as Tempering A Pink W/O, which consisted primarily of potassium nitrate, sodium nitrate, and sodium nitrite. MACTEC performed eight soil test borings (B-1 through B-6, B-1A, and B-3A) and collected soil samples (both surface and subsurface) from each boring for laboratory analysis of nitrate and nitrite, collected 14 surface soil samples (SS-1 through SS-14) for laboratory analysis of nitrate and nitrite, and installed two temporary monitoring wells (TW-1 and TW-2) and collected a groundwater sample from each monitoring well for laboratory analysis of nitrate and nitrite. Following the excavation of the nitrate/nitrite impacted soils, MACTEC collected eight grab soil samples (SS-15 through SS-22) from the bottom of the excavation for laboratory analysis of nitrate and nitrite. The soil samples were analyzed for nitrate using USEPA Method 353.2 and for nitrite using USEPA Method 354.1. Nitrate concentrations in the soil samples collected from the soil test borings ranged from non-detect to 3,100 mg/kg. Nitrite concentrations in the soil samples collected from the soil test borings ranged from non-detect to 1,900 mg/kg. Nitrate concentrations in the surface soil samples ranged from non-detect to 29 mg/kg. Nitrite was not detected in the surface soil samples. Nitrate concentrations from the grab soil samples collected from the bottom of the excavation ranged from non-detect to 280 mg/kg. Nitrate concentrations from the grab soil samples ranged from non-detect to 66 mg/kg. The results of this assessment are documented in MACTEC's *Second Revised Draft Report of Environmental Services* (2003) referenced in Section 1.3. The laboratory analytical results are summarized on **Tables 4 through 6**. The sample locations are shown on **Figures 7 and 10**.

A site reconnaissance was performed by MACTEC and VAC in November 2001 and several environmental conditions were noted. Stained soil was observed near a metals baghouse located along the northwest side of the manufacturing building, stained soil was observed adjacent to a scrap metal rolloff, empty drum storage area, and a blower exhaust system from the compounding room located on the southwest side of the manufacturing building. The presence of an underground oil/water separator was also noted as an environmental concern. Historical reports of stressed vegetation to the south

of the tank containment area and cleaning water discharges to catch basins discharging to stormwater outfalls were also environmental concerns. The results of the assessment described in the following paragraphs were documented in MACTEC's *Report of Phase II Environmental Site Assessment* (2003) referenced in Section 1.3. Sample locations are shown on **Figures 7 and 11 through 13**. The laboratory analytical data is summarized on **Tables 7 through 14**.

MACTEC collected surface soil samples from the metals baghouse (MB-1 through MB-3), scrap metal rolloff (SM-1 through SM-4), empty drum storage area (ED-1 through ED-4), near the blower exhaust from the compounding area (CA-1 and CA-2), south of the tank containment area (TS-1 through TS-4), and from the storm water outfalls (OF-1, OF-1A, OF-2, OF-2A, OF1-2, OF1-3, and OF1-4). As appropriate, based on knowledge of plant processes contributing to the environmental conditions, the surface soil samples were variously analyzed for VOCs by USEPA Method 8260B, SVOCs by USEPA Method 8270C, total petroleum hydrocarbons-oil & grease (TPH O&G) by USEPA Method 9071A, and the eight Resource Conservation and Recovery Act (RCRA) metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) plus nickel by USEPA Methods 6010B and 7471A (mercury). Surface soil samples OF1-2, OF1-3, and OF1-4 were only analyzed for TPH O&G. Subsurface soil samples were collected from the oil/water separator (GT-1 through GT-4) and analyzed for VOCs by USEPA Method 8260B, SVOCs by USEPA Method 8270C, and TPH O&G by USEPA Method 9071A.

Concentrations of barium (25 mg/kg to 31 mg/kg), chromium (68 mg/kg to 180 mg/kg), lead (14 mg/kg to 28 mg/kg), and nickel (30 mg/kg to 73 mg/kg) were detected in the soil samples collected from near the metals baghouse. Arsenic was also detected in sample MB-3 at a concentration of 2.1 mg/kg. Following receipt of the laboratory analytical results, the samples were analyzed for hexavalent chromium by USEPA Method 6010B. Hexavalent chromium was not detected in the soil samples. The soil sample from MB-3 was analyzed for arsenic using the Toxicity Characteristic Leachate Procedure (TCLP) and arsenic was not detected.

Concentrations of acetone from non detect to 27 micrograms per kilogram (µg/kg), diethylphthalate (non detect to 56,000 µg/kg), bis(2-ethylhexyl) phthalate (non detect to 440 µg/kg), barium (27 mg/kg to 44 mg/kg), cadmium (non detect to 0.16 mg/kg),

chromium (21 mg/kg to 140 mg/kg), lead (23 mg/kg to 30 mg/kg), nickel (9.2 mg/kg to 27 mg/kg), and TPH O&G (non detect to 830 mg/kg) were detected in the soil samples collected from near the scrap metal rolloff. Following excavation of the visibly stained soils, four soil samples (SM-1A through SM-2A) were collected from the approximate location of the previous soil samples. Sample SM-1 was analyzed for chromium, which was detected at a concentration of 19 mg/kg. Each sample was analyzed for TPH O&G and the concentrations ranged from non detect to 170 mg/kg.

Concentrations of acetone (non detect to 25 µg/kg), butyl benzyl phthalate (non detect to 620 µg/kg), di-n-butyl phthalate (non detect to 5,100 µg/kg), diethylphthalate (non detect to 16,000 µg/kg), bis(2-ethylhexyl) phthalate (non detect to 1,000 µg/kg), and TPH O&G (370 mg/kg to 5,800 mg/kg) were detected in the soil samples collected from near the empty drum storage area. Following excavation of the visibly stained soils, four soil samples (EB-1A through EB-4A) were collected from the approximate location of the previous soil samples. Each sample was analyzed for TPH O&G and the concentrations ranged from 86 mg/kg to 160 mg/kg.

Concentrations of di-n-butyl phthalate (non detect to 2,800 µg/kg), diethylphthalate (1,600 µg/kg to 1,300,000 µg/kg), dimethylphthalate (non detect to 680 µg/kg), and bis(2-ethylhexyl) phthalate (non detect to 580 µg/kg) were detected in the soil samples collected from near the blower exhaust from the compounding area.

Concentrations of acetone (22 µg/kg to 350 µg/kg) and diethylphthalate (non detect to 630 µg/kg) were detected in the soil samples collected from south of the tank containment area.

Concentrations of arsenic (1.2 mg/kg to 2.7 mg/kg), barium (25 mg/kg to 32 mg/kg), chromium (11 mg/kg to 19 mg/kg), lead (20 mg/kg to 22 mg/kg), nickel (5.4 mg/kg to 12 mg/kg), selenium (1.0 mg/kg to 1.5 mg/kg), and TPH O&G (920 mg/kg to 2,600 mg/kg) were detected in the soil samples from the storm water outfalls. Concentrations of diethylphthalate (820 µg/kg), benzo(a)anthracene (1,100 µg/kg), benzo(a)pyrene (1,200 µg/kg), benzo(b)fluoranthene (1,900 µg/kg), benzo(g,h,i)perylene (610 µg/kg), benzo(k)fluoranthene (760 µg/kg), chrysene (1,600 µg/kg), fluoranthene (2,100 µg/kg), indeno(1,2,3-c,d)pyrene (450 µg/kg), phenanthrene (580 µg/kg), and pyrene (1,700 µg/kg)

were detected in the soil sample collected from Outfall 002 (OF-2A). Concentrations of TPH O&G (non detect to 280 mg/kg) were detected in the soil samples collected downgradient from Outfall 001 (OF1-2, OF1-3, and OF1-4).

Concentrations of acetone (non detect to 290 µg/kg) and TPH O&G (non detect to 190 mg/kg) were detected in the subsurface soil samples collected from near the oil/water separator. Following removal of the oil/water separator and excavation of soils surrounding the oil/water separator, two subsurface soil samples were collected from beneath the inflow end of the separator and the outflow end of the separator. Concentrations of acetone (44 µg/kg to 56 µg/kg), 2-butanone (non detect to 17 µg/kg), 1,1-dichloroethene (non detect to 140 µg/kg), ethylbenzene (non detect to 7.4 µg/kg), toluene (non detect to 13 µg/kg), xylenes (non detect to 100 µg/kg), diethylphthalate (9,600 µg/kg to 89,000 µg/kg), bis(2-ethylhexyl) phthalate (non detect to 800 µg/kg), and TPH O&G (200 mg/kg to 420 mg/kg) were detected in the soils samples.

In May 2002, the acetone UST was closed in place and a closure assessment was performed by MACTEC. Four soil test borings (B-1 through B-4) were performed and soil samples were collected from each boring. The soil samples were analyzed for VOCs by USEPA Method 8260B. Concentrations of acetone were detected in the soil samples from borings B-1 and B-3 at 33 µg/kg and 44 µg/kg, respectively. A concentration of 2-butanone (methyl ethyl ketone, or MEK) was also detected in the soil sample from boring B-1 at 14 µg/kg. The UST closure assessment was documented in MACTEC's *Underground Storage Tank (UST) Assessment Report* (2002) referenced in Section 1.3. The laboratory analytical results are summarized on **Table 15**. The sample locations are shown on **Figures 7 and 9**.

In January 2005, two hand auger borings (GP-16 and GP-17) were performed inside the former hazardous waste accumulation building at the Site. One soil sample was collected from each boring and analyzed for VOCs by USEPA Method 8260B. Tetrachloroethene (perchloroethylene, or PCE) was detected in each sample at concentrations of 1,200 µg/kg and 64 µg/kg, respectively. The results of the soil assessment were documented in MACTEC's *Results of Field Screening Sampling* (2005) referenced in Section 1.3. The sample locations are shown on **Figure 7** and the laboratory analytical results are summarized on **Table 16**.

3.1.2 Review of Existing Groundwater Data

In association with the assessment of the subgrade piping related to the acetone UST at the tank containment area described in Section 3.1.1, MACTEC collected groundwater samples (HP-1 and HP-2) from two of the soil test borings performed during the assessment (STB-1 and STB-6). The groundwater samples were collected using a discrete-interval Hydropunch® sampler and analyzed for acetone by USEPA Method 8260 and methanol by USEPA Method 8000M. Acetone was detected in the groundwater sample (HP-2) collected from boring STB-6 at a concentration of 298 micrograms per liter (µg/L). The groundwater assessment was described in MACTEC's *Preliminary Site Contamination Assessment* (1996) referenced in Section 1.3. Laboratory analytical results are summarized in **Table 17**. Sample locations are shown on **Figure 7**.

In association with the assessment of the quench tank and rinse tank cleaning water release described in Section 3.1.1, MACTEC installed two temporary monitoring wells (TW-1 and TW-2) and collected a groundwater sample from each well. Monitoring well construction data is included on Table 1. The samples were analyzed for total nitrate by USEPA Method 353.2 and total nitrite by USEPA Method 354.1. Concentrations of nitrate were detected in temporary monitoring wells TW-1 and TW-2 at 2.5 milligrams per liter (mg/L) and 2.2 mg/L, respectively. Concentrations of nitrite were detected in TW-1 and TW-2 at 0.066 mg/L and 0.056 mg/L, respectively. The groundwater assessment was documented in MACTEC's *Second Revised Draft Report of Environmental Services* (2003) referenced in Section 1.3. Sample locations are shown on **Figures 7 and 10** and laboratory analytical results are summarized on **Table 18**.

During the assessment performed in 2002, a temporary monitoring well (OWS-TW-1) was installed at the former location of the oil/water separator. Monitoring well construction data is included in **Table 1**. A groundwater sample was collected from the temporary monitoring well and analyzed for VOCs by USEPA Method 8260B, SVOCs by USEPA Method 8270C, and TPH O&G by USEPA Method 9070. Concentrations of acetone (79 µg/L), 2-butanone (15 µg/L), 1,1-dichloroethane (220 µg/L), 1,1-dichloroethene (15 µg/L), ethylbenzene (28 µg/L), naphthalene (6.4 µg/L), toluene (58 µg/L), 1,1,1-trichloroethane

(180 µg/L), xylenes (370 µg/L), di-n-butyl phthalate (26 µg/L), diethylphthalate (320,000 µg/L), and TPH O&G (470 mg/L) were detected in the groundwater sample.

Based on the results of the groundwater sample collected from temporary monitoring well OWS-TW-1, a permanent monitoring well (MW-1) was installed at the former location of the oil/water separator. Monitoring well construction data is included in **Table 1**. The top of the well screen for monitoring well MW-1 is located approximately four feet below the water table surface. A groundwater sample was collected from the permanent monitoring well and analyzed for VOCs by USEPA Method 8260B, SVOCs by USEPA Method 8270C, and TPH O&G by USEPA Method 9070. Concentrations of acetone (350 µg/L), 2-butanone (62 µg/L), chloroethane (35 µg/L), 1,1-dichloroethane (450 µg/L), 1,1-dichloroethene (26 µg/L), ethylbenzene (30 µg/L), naphthalene (10 µg/L), styrene (76 µg/L), toluene (110 µg/L), 1,1,1-trichloroethane (260 µg/L), xylenes (450 µg/L), diethylphthalate (620,000 µg/L), and TPH O&G (690 mg/L) were detected in the groundwater sample. The groundwater assessment was documented in MACTEC's *Report of Phase II Assessment* (2003) referenced in Section 1.3. The laboratory analytical results for OWS-TW-1 are summarized on **Table 19**. The locations of the wells are shown on **Figure 7**.

In June 2003, MACTEC installed 18 temporary monitoring wells (GP-1 through GP-15, GP-1d, GP-4d, and GP-14d) at the site for the purpose of collecting field-screening groundwater samples to assess the groundwater contamination detected at the former oil/water separator, groundwater to the southeast and southwest of the manufacturing building, groundwater southwest of the tank containment area, and groundwater to the southwest of the former hazardous waste accumulation building. Groundwater samples were also collected from monitoring well MW-1 as well as three other monitoring wells installed following construction of the manufacturing building (B-1, B-2, and B-3). The groundwater samples were analyzed for VOCs by USEPA Method 8260B and SVOCs by USEPA Method 8270C. Acetone (150 µg/L), chloroethane (100 µg/L), 1,1-dichloroethane (210 µg/L), 1,1-dichloroethene (9.3 µg/L), cis-1,2-dichloroethene (32 µg/L), ethylbenzene (26 µg/L), naphthalene (9.3 µg/L), toluene (110 µg/L), 1,1,1-trichloroethane (75 µg/L), xylenes (400 µg/L), and diethylphthalate (210,000 µg/L) were detected in the groundwater sample from monitoring well MW-1. PCE and trichloroethene (TCE) were detected in the groundwater sample from temporary monitoring well GP-14 at concentrations of 11,000

µg/L and 16 µg/L, respectively. PCE was detected in the groundwater sample from temporary monitoring well GP-14d at a concentration of 53 µg/L and temporary monitoring well GP-15 at a concentration of 57 µg/L. The results of the field-screening groundwater assessment are documented in MACTEC's *Results of Field-Screening Groundwater Sampling* (2003) referenced in Section 1.3. The temporary monitoring well locations are shown on **Figure 7**. Laboratory analytical results are summarized on **Tables 20 and 21**.

Based on the field-screening groundwater results in temporary monitoring wells GP-14, GP-14d, and GP-15, MACTEC performed additional field screening activities in January and February 2005 downgradient from the former hazardous waste accumulation building, both on the Site and on the adjacent former Sherwin-Williams property. The assessment consisted of performing 15 Geoprobe® groundwater sample borings, collecting groundwater samples and analyzing the samples for VOCs by USEPA Method 8260B. PCE was detected in the groundwater samples at concentrations ranging from non detect to 460 µg/L. The results of the field-screening groundwater assessment are documented in MACTEC's *Results of Field-Screening Sampling* (2005) referenced in Section 1.3. Laboratory analytical results are summarized on **Table 22**. The field-screening groundwater locations are shown on **Figure 7**.

3.1.3 Review of Existing Surface Water Data

In February 2005, MACTEC collected four surface water samples (SW-01, SW-06, SW-09, and SW-011) from an unnamed tributary to Stoddard Creek located on both the Site and the adjacent former Sherwin-Williams property. The surface water samples were analyzed for VOCs by USEPA Method 8260B. Concentrations of PCE (non detect to 99 µg/L) were detected in the surface water samples. The results of the field-screening surface water assessment are documented in MACTEC's *Results of Field-Screening Sampling* (2005) referenced in Section 1.3. The surface water sampling locations are shown on **Figure 7**. Laboratory analytical results are summarized on **Table 23**.

3.1.4 Review of Existing Sediment Data

No sediment samples have been collected at the Site or from adjacent properties.

3.2 AREAS OF CONCERN

As a result of the review of the available Site data and historical reports, nine Areas of Concern (AOCs) have been identified at the Site. They are illustrated on **Figure 14** and are identified as follows.

3.2.1 AOC #1 Tank Containment and Underground Piping Area

The Tank Containment and Underground Piping Area consists of a bermed and covered concrete containment structure and both aboveground and subgrade product piping lines located to the southwest and southeast of the facility building. The concrete containment structure contains two 6,000-gallon ASTs that formerly contained DEP and overlies a 6,000-gallon acetone UST. Product from the acetone UST was pumped from the UST into the facility via an underground pipeline and was used for polishing screwdriver handles. Product from the diethylphthalate ASTs was pumped from the ASTs into the facility via an aboveground pipeline and was used in compounding plastic materials for extrusion for screwdriver handles, mallet heads, and other plastic parts. Potential contaminants are acetone and diethylphthalate. A release from the underground piping was documented in 1996 and assigned Site ID #04235 by the SCDHEC, Bureau of Underground Storage Tank Management.

Twelve soil samples and two groundwater samples were collected from the areas of suspected release. Acetone was detected in six of the soil samples at concentrations above the laboratory's PQL. Acetone was detected in one of the groundwater samples. MACTEC submitted the assessment data to the SCDHEC Bureau of UST Management and negotiated a revised groundwater Health Advisory for acetone with the SCDHEC. The SCDHEC issued a Conditional No Further Action (CNFA) letter for the acetone release on June 24, 1997. A copy of the CNFA letter is included in **Appendix A**.

Tank tightness testing was later performed on the acetone UST and the acetone UST underground piping (two tests) and the diethylphthalate aboveground piping. One of the tests of the acetone underground piping included the 90º elbows and the second test did not include the 90º elbows. The acetone UST, acetone UST underground piping (without the 90º elbows), and the aboveground piping from the diethylphthalate ASTs passed the

tightness testing. The test of the acetone UST piping with the 90º elbows failed the test and it was concluded that the acetone release was a result of a failure at the 90º elbows. The results of the tightness testing were documented in MACTEC's *Report of Acetone Tank and Line Testing and Diethylphthalate Line Testing* (1997) referenced in Section 1.4. The underground piping from the acetone UST was subsequently closed in place and replaced with aboveground piping from the UST into the facility building.

Four soil samples were collected from an area of reported historical stressed vegetation downgradient from the stormwater drain valve in the tank containment area. Concentrations of acetone and diethylphthalate were detected above the laboratory's PQL in one or more soil samples collected from this area.

The acetone UST was closed in place during 2002 and four soil samples were collected in conjunction with the closure assessment. Acetone was detected above the laboratory's PQL in two of the soil samples and MEK was detected above the laboratory's PQL in one of the soil samples.

A field-screening groundwater sample was collected downgradient of the acetone UST. VOCs or SVOCs were not detected above the laboratory's PQL.

Soil sampling results are summarized in **Tables 3, 11, and 15**. Groundwater sampling results are summarized in **Tables 17 and 21** (GP-9). No concentrations of acetone in soil exceeded the USEPA Residential Regional Screening Level (RSL); however the Soil Screening Levels (SSLs) were exceeded in five samples. No concentrations of acetone in groundwater exceeded the USEPA Tap Water RSL. Based on the previous environmental assessments performed at this AOC and a comparison of soil and groundwater concentrations to available published risk-based screening levels, sufficient data exist to determine the potential risk associated with this AOC to human health and the environment.

3.2.2 AOC #2 Heat Treat Cleaning Water Disposal Area

The Heat Treat Cleaning Water Disposal Area is located to the northwest of the facility building. The heat treat process consisted of a quench tanks (300.7 cubic feet) and two rinse tanks (153.7 cubic feet each). The quench tank held about 2,200 gallons of quench salt identified as Temper A Pink W/O that contained potassium nitrate, sodium nitrate, and sodium nitrite. Each rinse tank held about 1,150 gallons of water. Periodically, the quench tank and rinse tanks were cleaned and waste cleaning water and rinse water were discharged to the ground outside the heat treat area to the northwest of the facility building. Potential contaminants are nitrate and nitrite.

Fourteen surface samples (SS-1 through SS-14 on Figure 10) were collected to assess the horizontal extent of nitrate and nitrite contamination and seven soil test borings (B-1 through B-5, B-1A, and B-2A on Figures 7 and 10) were performed to assess the vertical extent of nitrate and nitrite contamination. One soil test boring (B-6) was drilled as a control boring located on the opposite side of the facility from the Heat Treat Cleaning Water Disposal Area. Nitrate was detected in five of the 14 surface soil samples but nitrite was not detected in any of the surface soil samples. Both nitrate and nitrite were detected in the subsurface soil samples. Two temporary monitoring wells (TW-1 and TW-2 on **Figure 7** and **Figure 10**) were installed (see **Table 1** for well construction data) and groundwater samples were collected to assess the nitrate and nitrite in Site groundwater. Both nitrate and nitrite were detected in the groundwater samples.

An approximate 70 by 150 foot area with the highest concentrations of nitrate on the surface was excavated down to a depth of approximately one foot below ground surface (see limits of excavation on Figure 10) and to five feet below ground surface at the location of one soil test boring that had elevated nitrate concentrations at five feet below ground surface (see limits of excavation on Figure 10). Following completion of the excavation activities, eight grab soil samples were collected from the base of the excavation. Nitrate was detected in four of the eight samples and nitrite was detected in three of the samples. The excavation was subsequently backfilled with clean soil.

Soil sampling results are summarized in **Tables 4 through 6** and groundwater sampling results are summarized in **Table 18**. The concentrations of nitrate and nitrite in soil do not

exceed their respective USEPA Residential RSL and there is no established SSL. The concentrations of nitrate and nitrite in groundwater do not exceed their respective Maximum Contaminant Levels (MCLs). Based on the previous environmental assessments performed at this AOC and a comparison of soil and groundwater concentrations to available published risk-based screening levels, sufficient data exist to determine the potential risk associated with this AOC to human health and the environment. Additionally, the SCDHEC Bureau of Water issued a letter indicating no investigation was required for this area on March 22, 2002 and assigned Site ID # 01817. A copy of the SCDHEC letter is included in **Appendix A**.

3.2.3 AOC #3 Former Metals Baghouse

The Metals Baghouse is located along the northwest side of the facility building. The Metals Baghouse was used to collect dust from grinding and grit blasting operations for screwdriver blades, spade bits, and nut drivers. Steel stock included American Iron and Steel Institute (AISI) 6150 (chromium-vanadium steel), AISI S2 (tool steel), AISI 1060 (carbon steel), AISI 4130 (chromium-molybdenum steel), AISI 4037 (molybdenum steel), and AISI 10B53 (boron steel). Stained soil was documented during previous environmental investigations and three soil samples were collected from the areas of stained soil. Potential contaminants are metals. Concentrations of arsenic, barium, chromium, lead, and nickel were detected above the laboratory's PQL in one or more soil samples collected from this area.

Soil sampling results are summarized in **Table 7**. Other than arsenic, the detected metals concentrations did not exceed the USEPA Residential RSLs. One sample had a concentration of arsenic that exceeded the Residential RSL, but was within the range of naturally occurring arsenic in South Carolina soils. This sample also exceeded the SSL for arsenic. All three samples had concentrations of nickel that exceeded the SSL. Based on the previous environmental assessments performed at this AOC and a comparison of soil and groundwater concentrations to available published risk-based screening levels, additional data is necessary to determine the potential risk associated with this AOC to human health and the environment.

3.2.4 AOC #4 Former Scrap Metal Rolloff

The Scrap Metal Rolloff is located adjacent to an asphalt driveway located to the southwest of the facility building. The Scrap Metal Rolloff received scrap steel from hydraulic press operations, metal swarf from grinding operations, and spent media from grit blast operations (see Section 3.2.3 for steel types). Stained soil and asphalt were documented in previous environmental investigations and four soil samples were collected from the areas of stained soil. Potential contaminants are VOCs, SVOCs, metals, and oil and grease. Concentrations of acetone, DEP, bis(2-ethylhexyl)phthalate, barium, cadmium chromium, lead, nickel, and TPH O&G were detected above the laboratory's PQL in one or more samples collected from this area. An approximate 1.5 foot by 60 foot "L" shaped excavation was conducted to a depth of approximately one foot to remove the visibly stained soils (Figure 15). Four soil samples were collected from the base of the excavation at the approximate location of the previous soil samples. Concentrations of chromium and TPH O&G were detected above the laboratory's PQL in one or more of the soil samples collected following the excavation. The excavation was subsequently backfilled with clean soil.

Soil sample results are summarized in **Table 8**. Detected VOCs and SVOCs were below their respective USEPA Residential RSLs. One sample had concentrations of DEP and bis(2-ethylhexyl)phthalate that exceeded the SSL. Detected metal concentrations were below their respective Residential RSLs and SSLs. Based on the previous environmental assessments performed at this AOC and a comparison of soil and groundwater concentrations to available published risk-based screening levels, additional data is necessary to determine the potential risk associated with this AOC to human health and the environment.

3.2.5 AOC #5 Former Empty Drum Storage Pad

The Empty Drum Storage Pad is located at the western corner of the facility building. Empty drums of chemicals used in the manufacturing process were stored in this area including various oils (compressor, hydraulic, stamping, lubrication, rust preventative, machining, and heat transfer), solvents (Freon 113, mineral spirits, and chlorinated degreasing solvents), aqueous coolants, various paints, inks, and thinners, and

plasticizers (phthalate compounds). Stained soil was documented in previous environmental investigations and four soil samples were collected from the areas of stained soil. Potential contaminants are VOCs, SVOCs, and oil and grease. Concentrations of acetone, butyl benzyl phthalate, di-n-butyl phthalate, DEP, bis(2-ethylhexyl)phthalate, and TPH O&G were detected above the laboratory's PQL in one or more samples collected from this area. An approximate 1.5 foot by 45 foot "L" shaped excavation was conducted to a depth of approximately one foot to remove the visibly stained soils (**Figure 16**). Four soil samples were collected from the base of the excavation at the approximate location of the previous soil samples. Concentrations of TPH O&G were detected above the laboratory's PQL in each of the soil samples collected following the excavation. The excavation was subsequently backfilled with clean soil.

Field-screening groundwater samples collected downgradient from the Empty Drum Storage Area did not detect concentrations of VOCs or SVOCs above the laboratory's PQL.

Soil sample results are summarized in **Table 9** and groundwater sample results are summarized on **Table 21** (GP-12 and GP-13). Detected VOCs and SVOCs were below their respective USEPA Residential RSLs. Three samples had concentrations of DEP that exceeded the SSL and one of those samples also contained concentrations of bis(2-ethylhexyl)phthalate and di-n-butyl phthalate exceeding their SSLs. No VOCs or SVOCs were detected in the groundwater samples collected downgradient from this AOC. Based on the previous environmental assessments performed at this AOC and a comparison of soil concentrations to available published risk-based screening levels, sufficient data exist to determine the potential risk associated with this AOC to human health and the environment.

3.2.6 AOC #6 Compounding Room Blower Exhaust

The Compounding Room Blower Exhaust is located along the southwest side of the facility building. Exhaust vapors from the Compounding Room were observed to condense on the piping near the exhaust vents and drip on the ground. Two surface soil samples were collected from the areas where condensate dripped on the soil. Potential contaminants are plasticizer compounds (SVOCs). Concentrations of di-n-butyl phthalate,

DEP, dimethylphthalate, and bis(2-ethylhexyl) phthalate were detected above the laboratory's PQL in the soil samples collected in this area. Field-screening groundwater samples collected downgradient from this AOC did not detect concentrations of VOCs or SVOCs above the laboratory's PQL.

Soil sample results are summarized on **Table 10** and groundwater sample results are summarized on **Table 21** (GP-11). Detected SVOCs were below their respective USEPA Residential RSLs. Two samples had concentrations of DEP that exceeded the SSL and one of those samples also had concentrations of bis(2-ethylhexyl)phthalate and di-n-butyl phthalate exceeding their SSLs. No VOCs or SVOCs were detected in the groundwater sample collected downgradient from this AOC. Based on the previous environmental assessments performed at this AOC and a comparison of soil and groundwater concentrations to available published risk-based screening levels, additional data is necessary to determine the potential risk associated with this AOC to human health and the environment.

3.2.7 AOC #7 Storm Water Outfalls

The Storm Water Outfalls are located in the southern portion of the property (Outfall 001) and the northern portion of the property (Outfall 002). Water generated from cleaning operations (i.e., mopping) inside the plant were reported to be historically discharged into a storm water catch basin to the west of the facility building that has the potential to discharge to either, or both, of the outfalls. Potential contaminants are VOCs, SVOCs, metals, and oil and grease (degreasers, plasticizers, metals, oils, etc.). Soil samples collected from near Outfall 001 detected concentrations of diethylphthalate, arsenic, barium, chromium, lead, nickel, selenium, and TPH O&G above the laboratory's PQL. Soil samples collected from near Outfall 002 detected concentrations of benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, fluoranthene, indeno(1,2,3-c,d)pyrene, phenanthrene, pyrene, arsenic, barium, chromium, lead, nickel, selenium, and TPH O&G above the laboratory's PQL.

Soil sampling results are summarized in **Table 12**. No VOCs were detected in the soil samples. Four SVOCs (benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, and

indeno(1,2,3-c,d)pyrene) were detected at concentrations that exceed their respective USEPA Residential RSLs and the SSLs in one sample. Two SVOCs (benzo(k)fluoranthene and chrysene) exceeded the SSL but were below the USEPA Residential RSLs. Other detected SVOCs were below the Residential RSL and SSL. It should be noted that Outfall 002 is located near and receives runoff from a street and parking area for a baseball field, which is the likely source of SVOCs observed in the soil sample collected at Outfall 002. Arsenic was detected in both samples that exceeded the USEPA Residential RSL and the SSL. Selenium was detected in two samples at concentrations less than the USEPA Residential RSL but above the SSL. Based on the previous environmental assessments performed at this AOC and a comparison of soil and groundwater concentrations to available published risk-based screening levels, additional data is necessary to determine the potential risk associated with this AOC to human health and the environment.

3.2.8 AOC #8 Former Oil/Water Separator

The former Oil/Water Separator was located below grade on the southeast side of the facility building. The separator was connected to the sanitary sewer discharge line from facility and received wastewater from floor drains inside the building (**Figure 17**). The subsurface soils adjacent to the separator were assessed during previous environmental investigations by collecting a soil sample on each side of the separator from the approximate depth of the bottom of the separator. Potential contaminants are VOCs, SVOCs, metals, and oil and grease (degreasers, plasticizers, metals, and oils, etc.). Concentrations of acetone and TPH O&G were detected above the laboratory's PQL in one or more of the soil samples collected from around the separator. The separator was subsequently removed and contaminated soils were excavated around the separator to the apparent depth of groundwater in the area (**Figure 18**). The excavation was approximately 15 feet by 15 feet in surface area and approximately 19 feet in depth. Two soil samples collected from the bottom of the excavation detected concentrations of acetone, MEK, 1,1-dichloroethane, ethylbenzene, toluene, xylenes (total), diethylphthalate, bis(2-ethylhexyl)phthalate, and TPH O&G above the laboratory's PQL. The excavation was subsequently backfilled with clean soil.

The groundwater sample from temporary monitoring well OWS-TW-1 detected concentrations of acetone, 2-butanone, 1,1-dichloroethane, 1,1-dichloroethene, ethylbenzene, naphthalene, toluene, 1,1,1-trichloroethane, xylenes, di-n-butyl phthalate, diethylphthalate, and TPH O&G above the laboratory's PQL. The groundwater sample collected from permanent monitoring well MW-1 detected concentrations of acetone, 2-butanone, chloroethane, 1,1-dichloroethane, 1,1-dichloroethene, ethylbenzene, naphthalene, styrene, toluene, 1,1,1-trichloroethane, xylenes, diethylphthalate, and TPH O&G above the laboratory's PWL. Neither VOCs nor SVOCs were detected in the field-screening groundwater samples collected near this AOC.

Soil sample results are summarized in **Tables 13 and 14**. Groundwater sample results are summarized in **Tables 19 and 20**. Detected concentrations of VOCs and SVOCs were below the USEPA Residential RSLs. Detected concentrations of 1,1-dichloroethene, ethylbenzene, and bis(2-ethylhexyl)phthalate, were above the SSL in one sample. Detected concentrations of DEP in two samples were greater than the SSL. Concentrations of 1,1-dichloroethene were detected above the MCL. Concentrations of 1,1-dichloroethane, cis-1,2-dichloroethene, ethylbenzene, naphthalene, xylenes, and DEP were above their respective Tap Water RSLs. Based on the previous environmental assessments performed at this AOC and a comparison of soil and groundwater concentrations to available published risk-based screening levels, additional data is necessary to determine the potential risk associated with this AOC to human health and the environment.

3.2.9 AOC #9 Former Hazardous Waste Accumulation Building

The Former Hazardous Waste Storage Accumulation Building is located to the southwest of the facility building near the southwest property boundary. Various hazardous and non-hazardous wastes were accumulated in the building prior to being picked up for disposal including solvents (Freon 113, mineral spirits, and chlorinated degreasing solvents), various paints, inks, and thinners, and plasticizers (phthalate compounds). Potential contaminants are VOCs and SVOCs. Two soil samples were collected from beneath the concrete floor of the building. Concentrations of PCE were detected above the laboratory's PQL in both soil samples. No other VOCs were detected above the laboratory's PQL.

PCE and TCE were detected above the laboratory's PQL in the field-screening groundwater samples collected immediately downgradient from this AOC. PCE was also detected above the laboratory's PQL in field-screening groundwater samples collected from the adjacent former Sherwin-Williams property, which is downgradient from the former Hazardous Waste Accumulation Building. Concentrations of PCE and TCE exceed the SCDHEC MCLs. It should be noted that the Sherwin-Williams property is not believed to be a source of VOC contamination and the Sherwin-Williams property is only impacted by contaminated groundwater from the release at the former Hazardous Waste Accumulation Building.

Surface water samples collected from an unnamed tributary to Stoddard Creek, both on the site and on the adjacent former Sherwin-Williams property indicate elevated concentrations of PCE. Concentrations of PCE exceed the SCDHEC WQS.

Soil sample results are summarized in **Table 16**. Groundwater sample results are summarized in **Table 21** (GP-14, GP-14d, and GP-15) and **Table 22**. Surface water sample results are summarized in **Table 23**. Concentrations of PCE above the SSL, but less than the Residential RSLs, were detected in two soil samples. Concentrations of PCE and TCE were detected above their MCLs and Residential RSLs. SVOCs were not detected in the groundwater samples collected downgradient from this AOC. Concentrations of PCE were detected in two surface water samples above its South Carolina Water Quality Criteria (WQC). Based on the previous environmental assessments performed at this AOC and a comparison of soil, groundwater, and surface water concentrations to available published risk-based screening levels, additional data is necessary to determine the potential risk associated with this AOC to human health and the environment.

3.3 DEVELOPMENT OF PRELIMINARY ARARS

One of the site objectives specified in the scoping phase of the Scope of Work for the RI/FS is to identify the potential Federal and State ARARs for the project. The ARARs are used in the FS to develop Remedial Action Objectives and appropriate Remedial Action Alternatives. ARARs may be categorized as chemical-specific requirements, location-specific requirements, and action-specific requirements. Early identification of potential ARARs aids in planning field activities. In accordance with USEPA "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" (October, 1998), a list of potential ARARs for the Site is presented below for SCDHEC consideration.

3.3.1 Chemical-Specific Requirements

Chemical specific ARARs are usually health or risk-based numerical values or methodologies which, when applied to site-specific conditions, result in the establishment of numerical values. In general, chemical-specific requirements are set for a single chemical or a closely-related group of chemicals. The chemical-specific requirements that will be considered for development of the ARARs for the Site are as follows:

- National Primary Drinking Water standards based on the Safe Drinking Water Act (SDWA) MCLs.
- SDWA MCL Goals (MCLGs).
- SCDHEC Water Quality Criteria (WQC).
- Regional Screening Levels (RSLs) and Soil Screening Level (SSLs) published by USEPA.
- Site-Specific Action Levels for Acetone, Vermont American Corporation, SCDHEC Bureau of Underground Storage Tank Management, Site ID #04235, June 24, 1997.

3.3.2 Location-Specific Requirements

A site's location is a fundamental determinant of its impact on human health and the environment. Location-specific ARARs are restrictions placed on the concentration of

hazardous substances or the conduct of activities due to location. Some examples of special locations include floodplains, wetlands, historic places and sensitive ecosystems or habitats. The location-specific requirements that will be considered for development of the ARARs for the Site are as follows:

- National Historic Preservation Act of 1966 (NHPA).
- Endangered Species Act.
- Wilderness Act.
- Fish and Wildlife Coordination Act.
- Wild and Scenic Rivers Act.
- Clean Water Act.
- 40 CFR Part 6 Appendix A (Floodplain Management and Protection of Wetlands).
- Water Classification and Standards (Regulation 61-68) and Classified Waters (Regulation 61-69), State of South Carolina.

3.3.3 Action-Specific Requirements

Action-specific ARARs are usually technology or activity-based requirements or limitations on actions taken with respect to hazardous waste. These requirements are triggered by a particular remedial activity that is selected to accomplish site remedy. Potential action-specific ARARs will begin to be identified for the Site during the FS when Remedial Action Alternatives are developed and evaluated.

The refinement of ARARs will continue as the conditions and the characterization of contaminants at the site and potential Remedial Action Alternatives are better defined.

3.4 IDENTIFICATION OF DATA GAPS

To properly assess the nature and extent of impacted media at the Site the following additional data will need to be obtained at the following AOCs.

3.4.1 AOC #2 Heat Treat Cleaning Water Disposal Area

One shallow monitoring well will be installed downgradient from previous soil sample location SS-6 and a groundwater sample will be collected to confirm that, other than nitrates and nitrates, no other chemical constituents were discharged with the Heat Treat cleaning water in this AOC.

The data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis are included in the Field Sampling and Analysis Plan (see Section 4.3.1).

3.4.2 AOC #3 Former Metals Baghouse

Shallow monitoring wells will be installed near the Former Metals Baghouse and groundwater samples will be collected to evaluate the vertical extent of contamination at the AOC.

The data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis are included in the Field Sampling and Analysis Plan (see Section 4.3.2).

3.4.3 AOC #4 Former Scrap Metal Rolloff

Soil borings will be performed and subsurface soil samples will be collected to evaluate the vertical extent of contamination at the AOC. Shallow monitoring wells will be installed and groundwater samples collected to evaluate the vertical extent of contamination at the AOC.

The data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis are included in the Field Sampling and Analysis Plan (see Sections 4.2.1 and 4.3.3).

3.4.4 AOC #6 Compounding Room Blower Exhaust

Soil borings will be performed and subsurface soil samples will be collected to evaluate the vertical extent of contamination at the AOC.

The data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis are included in the Field Sampling and Analysis Plan (see Section 4.2.2).

3.4.5 AOC #7 Storm Water Outfalls

Surface soil samples will be collected to evaluate the potential source of PAHs detected in previous environmental investigations at the AOC.

The data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis are included in the Field Sampling and Analysis Plan (see Section 4.1).

3.4.6 AOC #8 Former Oil/Water Separator (Grease Trap) Area

Soil borings will be performed and subsurface soil samples will be collected to evaluate the horizontal and vertical extent of contamination at the AOC. An additional monitoring well will be installed to the top of the bedrock surface at the source area and shallow monitoring wells will be installed downgradient of the source area to evaluate the vertical and horizontal extent of contamination at the AOC. Hydraulic conductivity (slug) testing will be performed in selected monitoring wells.

The data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis are included in the Field Sampling and Analysis Plan (see Section 4.2.3 and 4.3.4).

3.4.7 AOC #9 Former Hazardous Waste Accumulation Building

Soil borings will be performed in the area of the former Hazardous Waste Accumulation Building to evaluate the vertical and horizontal extent of contamination beneath the building. Monitoring wells, both shallow and to the top of the bedrock surface, will be installed in the AOC to evaluate the vertical and horizontal extent of contamination and to allow for groundwater monitoring at the AOC. Hydraulic conductivity (slug) testing will be performed in selected monitoring wells. Pore water samples will be collected from the bank of the unnamed tributary to Stoddard Creek to evaluate the groundwater/surface water interface at the AOC. Surface water and stream sediment samples will be collected to evaluate the impact of groundwater contamination to the unnamed tributary to Stoddard Creek.

The data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis are included in the Field Sampling and Analysis Plan (see Section 4.2.4 and 4.3.5).

3.4.8 Determination of Constituents of Concern

Based on a review of the analytical results from previous Site investigations, organic constituents are currently considered the primary constituents of concern (COCs) at the Site. Several VOCs and SVOCs have been detected in media (groundwater, surface water, etc.) collected from the Site. Therefore, the investigation COCs will include the organic compounds that comprise the TCL list by USEPA Method 8260B and the TCL list by USEPA Method 8270D. Additionally, the investigation will include Target Analyte List (TAL) metals by USEPA Methods 6010C/7470A/7471B. The specific compounds that comprise these lists are provided on Tables A-3 through A-5 of the Quality Assurance Project Plan (see Section 4.2.1).

3.4.9 Areas Of Concern

The physical boundaries for each AOC will need to be further defined and mapped.

3.4.10 Determination of Potential Risk

Currently there is insufficient data to determine if elevated levels of VOCs and SVOCs observed in samples collected at the site pose a risk to human health or the environment. Based on information obtained during the investigation at the site, an assessment of the risk the site poses to human health and the environment will be conducted.

4.0 REMEDIAL INVESTIGATION

The proposed RI for the Site will be conducted in one step and will focus on collecting characterization samples from the identified AOCs.

4.1 REMEDIAL INVESTIGATION SCOPE OF WORK

The objectives for the RI were discussed in Section 1.5. The objectives the RI include:

- Determine the source, nature, and extent of contaminated media (soil, groundwater, surface water, and sediment) present at the Site.
- Determine if the concentrations of constituents present in Site media present an unacceptable risk to human health or the environment.
- Develop and evaluate alternatives for remedial action to prevent, mitigate, or otherwise respond to the migration or the release or threatened release of hazardous substances, pollutants, or contaminants at or from the Site.

To meet these objectives, the RI will include completion of the following items:

- A review of existing information pertaining to the Site (prior to conducting investigation work).
- A Site visit (to locate sample locations and identify background locations).
- Completion of a well survey within a one mile radius of the Site (including location and determination of water uses).
- Collecting samples to further characterize the concentration of constituents in soils, groundwater, surface water, and sediment at the applicable AOCs.
- The development of a water table elevation contour map of the saprolite aquifer at the Site from existing and newly-installed monitoring wells.
- Preparation of isoconcentration maps of affected media illustrating distribution of constituents, as appropriate.
- Development of a Site Conceptual Hydrogeologic Model.

4.2 SAMPLING AND ANALYSIS PLAN

Site sampling and analysis will be conducted in accordance with protocols and procedures outlined in the Quality Assurance Project Plan and the Field Sampling and Analysis plan. Additionally, site sampling and analysis will be conducted under a site-specific Health and Safety Plan.

4.2.1 Quality Assurance Project Plan

A Quality Assurance Project Plan (QAPP) that describes the project objectives and organization, functional activities, and the quality assurance and quality control (QA/QC) protocols that will be used to achieve the desired data quality objective for the project has been prepared and is provided in Appendix B. The QAPP has been prepared in accordance with SCDHEC "Guidance Document for Preparing Quality Assurance Project Plans (QAPPs) for Environmental Monitoring Projects/Studies" (SCDHEC, October 2007).

4.2.2 Field Sampling and Analysis Plan

The Field Sampling and Analysis Plan (FSAP) that describes the data gathering methods, sampling objectives, sample locations and frequency, sampling equipment and procedures, and sampling and analysis has been prepared and is provided in Appendix C. In general, the FSAP includes:

- A review of existing information pertaining to the Site (prior to conducting investigation work).
- A Site visit (to locate sample locations and identify background locations).
- Completion of a well survey within a one mile radius of the Site (including location and determination of water uses). Hydraulic conductivity (slug) tests will be performed in selected monitoring wells.
- Installation of permanent monitoring wells for the collection of groundwater samples and future monitoring activities.
- Collection of a limited number of samples from various media at applicable AOCs to create a defensible data set to be compared to published risk-based screening levels.

- The development of a water table elevation contour map of shallow groundwater from existing monitoring wells.

4.2.3 Health and Safety Plan

A Site Health and Safety Plan (HASP) has been developed specific to the Site activities and is included in Appendix D. The HASP applies to AMEC employees and AMEC subcontractors, only. Each field team will have a copy of the HASP during field activities. Personnel working at the Site will be required to read, understand, and conform to the requirements of the HASP. As site activities progress and if new information arises, the HASP will be updated, as necessary, to ensure compliance with the Occupational Safety and Health Act (OSHA) and safe working conditions.

4.3 REMEDIAL INVESTIGATION REPORTING

At the conclusion of the RI, a Draft RI Report will be prepared and submitted that will:

- Summarize the characterization activities.
- Identify the sources of contamination.
- Delineate the nature and extent of contamination.
- Address the fate and transport of potential contaminants identified.

5.0 HUMAN HEALTH RISK ASSESSMENT

As part of the RI/FS, a screening assessment of potential human health risk associated with soil, groundwater, and surface water releases at the Vermont Bosch facility will be performed. The area is commercial/industrial in nature and this land use is not expected to change in the future.

The risk assessment will include an exposure assessment based on potential current and future site exposures, including a residential future use of the site, to soil and comparison of groundwater and surface water concentrations to risk-based screening levels protective of human health. The objective of this risk assessment is to identify constituents of potential concern (COPCs) in soil, groundwater, and surface water through comparison to available residential and industrial risk-based action levels (i.e., USEPA Regional Screening Levels (RSLs) for residential and industrial soil and for tap water, USEPA MCLs, SCDHEC WQCs). In accordance with USEPA Region IV risk assessment guidance, the USEPA RSLs are recommended for risk-based screening and the identification of COPCs. Background concentrations, both site-specific and regional, may also be used to screen site data and identify COPCs. A brief risk assessment will be prepared and included in the RI/FS submittal to the SCDHEC that summarizes the results of the constituent screening process and the exposure assessment.

6.0 FEASIBILITY STUDY

The FS will develop an appropriate range of management options for materials posing unacceptable risks in a manner consistent with the VCC, the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), the "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" (USEPA, 1988b) (the RI/FS Guidance), and other applicable USEPA guidance. Remedial action alternatives will be developed by assembling combinations of technologies (including innovative ones that offer the potential for superior treatment performance or lower cost for performance similar to that of demonstrated technologies) and the media to which they will be applied into overall alternatives that address contamination at an individual AOC, or if appropriate, several similar AOCs. Alternatives for each AOC where remediation is necessary will then be assembled to form a comprehensive, protective remedy for the Site.

6.1 DEVELOPMENT/SCREENING OF REMEDIAL ACTION ALTERNATIVES

The development and screening of Remedial Action Alternatives is performed to select an appropriate range of waste management options to be evaluated. This range of options will include, at a minimum, alternatives in which treatment is used to reduce the toxicity, mobility, or volume of the waste, but varying in the types of treatment, the amount treated, and the manner in which long-term residuals or untreated wastes are managed; alternatives that involve containment and treatment components; alternatives that involve containment with little or no treatment; and a no-action alternative. The following activities will be performed as a function of the development and screening of Remedial Action Alternatives.

6.1.1 Development and Screening of Remedial Action Alternatives

Remedial Action Alternatives (RAAs) will be developed and evaluated concurrent with the RI. A range of appropriate waste management options that, at a minimum, ensure protection of human health and the environment and comply with all ARARs will be identified.

6.1.2 Refine and Document Remedial Action Objectives

The Remedial Action Objectives (RAOs) will specify the contaminants and media of interest, exposure pathways and receptors, and an acceptable contaminant level or range of levels (at particular locations for each exposure route).

6.1.3 Develop General Response Actions

General response actions will be developed for each medium of interest defining containment, treatment, excavation, pumping, or other actions, singly or in combination, to satisfy the remedial action objectives.

6.1.4 Identify Areas and Volumes of Media

The areas and volumes of media to which general response actions may apply will be identified taking into account requirements for protectiveness as identified in the RAOs. The chemical and physical characterization of the Site, the risk screening, and remediation goals will also be taken into account.

6.1.5 Identify, Screen, and Document Remedial Technologies

Remedial technologies applicable to each general response action to eliminate those that cannot be implemented at the Site will be identified. General response actions will be refined to specify remedial technology types. Technology process options for each of the technology types will be identified either concurrent with the identification of technology types or following the screening of the considered technology types. Process options will be evaluated on the basis of effectiveness, implementability, and cost factors to select and retain one or, if necessary, more representative processes for each technology type.

6.1.6 Assemble and Document Alternatives

Selected representative technologies will be assembled into alternatives for each affected medium or operable unit. Together, all of the alternatives will represent a range of

treatment and containment combinations that will address either the Site or the operable unit as a whole.

6.1.7 Refine Alternatives

The Remedial Action Alternatives will be refined to identify contaminant volumes to be addressed by the proposed process and sizing of critical unit operations as necessary. Sufficient information will be collected for an adequate comparison of alternatives. Remedial action objectives for each medium will also be refined as necessary to incorporate risk screening. Additionally, action-specific ARARs will be updated as the RAAs are refined.

6.1.8 Conduct and Document Screening Evaluation of Each Alternative

A final screening process may be performed based on short and long term aspects of effectiveness, implementability, and relative cost. Note that the evaluation of effectiveness involves evaluating the long-term and short-term risks, among other factors, associated with a remedial alternative. Generally, this screening process is only necessary when there are many feasible alternatives available for detailed analysis. If necessary, the screening of alternatives will be conducted to assure that only the alternatives with the most favorable composite evaluation of all factors are retained for further analysis.

As appropriate, the screening will preserve the range of treatment and containment alternatives that was initially developed. The range of remaining alternatives will include options that use treatment technologies and permanent solutions to the maximum extent practicable.

6.2 DETAILED ANALYSIS OF REMEDIAL ACTION ALTERNATIVES

If remedial action is deemed appropriate for the Site, a detailed analysis will be conducted to provide the SCDHEC with the information needed to allow for the selection of a remedy for the Site. This analysis will consist of an assessment of each option against a set of

nine evaluation criteria and a comparative review of all options using the same nine evaluation criteria as a basis for comparison.

The nine evaluation criteria will be applied to the assembled RAAs to ensure that the selected RAA will be protective of human health and the environment; will be in compliance with, or include a waiver of, ARARs; will be cost-effective; will utilize permanent solutions and alternative treatment technologies, or resource recovery technologies, to the maximum extent practicable; and will address the statutory preference for treatment as a principal element. The evaluation criteria include:

1. Overall protection of human health and the environment;
2. Compliance with ARARs;
3. Long-term effectiveness and permanence;
4. Reduction of toxicity, mobility, or volume;
5. Short-term effectiveness;
6. Implementability;
7. Cost;
8. State acceptance; and
9. Community acceptance.

Criteria 8 and 9 are considered after the RI/FS Report has been released to the general public. For each alternative, the following will be provided: (1) a description of the alternative that outlines the waste management strategy involved and identifies the key ARARs associated with each alternative; and (2) a discussion of the individual criterion assessment. The community acceptance (9) will be addressed by the SCDHEC after completion of the Draft FS Report.

A comparative analysis among the RAAs will be performed. That is, each alternative will be compared against the others using the nine evaluation criteria as a basis of comparison. No one alternative will be identified as the preferred alternative in the FS. The preferred alternative will be identified and selected by the SCDHEC.

6.3 FEASIBILITY STUDY DELIVERABLES

The following deliverables will be prepared as part of developing, screening, and performing detailed analysis of the RAAs:

- A Draft FS Report will be prepared and submitted for SCDHEC review and comment. This report, as ultimately adopted or amended by the SCDHEC, provides a basis for remedy selection by the SCDHEC and documents the development and analysis of RAAs. The FS report will be prepared in accordance with the RI/FS Guidance (USEPA, October 1988).

- A Final FS Report will be prepared and submitted that satisfactorily addresses the SCDHEC's comments. Once the SCDHEC's comments have been addressed to the SCDHEC's satisfaction and SCDHEC approval has been obtained or an amendment has been furnished by the SCDHEC, the Final FS Report may be bound with the Final RI Report.

The draft and final FS will include the following four sections:

- Introduction including the purpose and organization of the report and background information summarized from the RI Report;

- Identification and screening of technologies including remedial action objectives, general response actions, and identification and screening of technology types and process options;

- Development and screening of alternatives; and

- Detailed analysis of alternatives.

7.0 SCHEDULE

The proposed Project Schedule for the overall RI/FS is presented below. The Project Schedule will be updated when the schedule changes by showing the original due date and revisions of the due date. A copy of the schedule will be contained in each major deliverable of the RI/FS.

All days are calendar (not business) days. The schedule below assumes a 30-day SCDHEC review time for each milestone listed. If the SCDHEC review times exceeds 30 days, the subsequent due dates will be adjusted.

Activity	**Schedule Date (Days)**
Effective Date of VCC	08/29/05
RI/FS Work Plan Submitted to SCDHEC	10/29/05
RI/FS Work Plan Revision 1.0 Submitted to SCDHEC	5/17/07
RI/FS Work Plan Revision 2.0 Submitted to SCDHEC	9/25/07
RI/FS Work Plan Revision 3.0 Submitted to SCDHEC	11/24/08
RI/FS Work Plan Revision 4.0 Submitted to SCDHEC	5/31/12
Initiate Fieldwork – RI	Within 60 days after receipt of SCDHEC approval of Final RI/FS Work Plan
Draft RI Submitted	Within 75 days after receipt of validated data from RI
Final RI Submitted	90 days after receipt of SCDHEC comments
Draft FS Report	180 days after SCDHEC approval of RI Report
Final FS Report	60 days after receipt of SCDHEC comments on Draft FS Report

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TABLES

TABLE 1

Summary of Permanent and Temporary Monitoring Well Construction Data
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Monitoring Well ID	Date Installed	Date Abandoned	Elevation Ground	TOC	Total Depth	Elevation Total Depth	Screen Interval	Elevation Screen Interval	Depth to Water	Date Measured	Elevation of Water Table
B-1	4/23/85	-	102.81	103.23	21	82.23	10 - 20	93.23 - 83.23	9.66	6/5/03	93.57
B-2	4/23/85	-	100.27	100.82	26	74.82	15 - 25	85.82 - 75.82	16.95	6/5/03	83.87
B-3	4/23/85	-	94.16	94.78	21	73.78	10 - 20	84.78 - 74.78	5.88	6/5/03	88.90
TW-1	2/5/02	4/10/02	NS	NS	32.5	-	22.5 - 32.5	- - -	20.77	2/8/02	-
TW-2	2/5/02	4/10/02	NS	NS	31.7	-	21.7 - 31.7	- - -	20.57	2/8/02	-
OWS-TW-1	6/7/02	6/10/02	NS	NS	24	-	14 - 24	- - -	13.5	6/7/02	-
MW-1	8/30/02	-	NS	NS	24	-	14 - 24	- - -	9.70	6/5/03	-

Notes:
NS = not surveyed.
B-1, B-2, and B-3 surveyed on 3/18/97 by C.O. Riddle Surveying Co., Inc.
TOC = top of casing.

TABLE 2

Summary of Practical Quantitation Limits That Exceed Screening Levels
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Media	Constituent	Type	Screening Level	Sample Identification
Soil	Benzene	VOC	SSL	SM-1, SM-2, SM-3, SM-4, SM-1A, SM-2 SM-3A, SM-4A, ED-1, ED-2, ED-3, ED-4, ED-1A, ED-2A, ED-3A, ED-4A, TS-1, TS-2, TS-3, TS-4, OF-1A, OF-2A, GT-1, GT-2, GT-3, GT-4, OWS-1, OWS-2, B-1, B-2, B-3, B-4, GP-16, GP-17
Soil	Carbon tetrachloride	VOC	SSL	
Soil	1,2-Dibromo-3-chloropropane	VOC	SSL	
Soil	1,2-Dibromomethane (EDB)	VOC	SSL	
Soil	1,1-Dichloroethane	VOC	SSL	
Soil	1,2-Dichloroethane	VOC	SSL	
Soil	1,1-Dichloroethene	VOC	SSL	
Soil	1,2-Dichloropropene	VOC	SSL	
Soil	Ethylbenzene	VOC	SSL	
Soil	Methanol	VOC	SSL	
Soil	Methylene chloride	VOC	SSL	
Soil	Tetrachloroethene	VOC	SSL	
Soil	1,1,2-Trichloroethane	VOC	SSL	
Soil	Trichloroethene	VOC	SSL	
Soil	Vinyl chloride	VOC	SSL	
Soil	Benzo(a)anthracene	SVOC	RRSL/SSL	SM-1, SM-2, SM-3, SM-4, SM-1A, SM-2 SM-3A, SM-4A, ED-1, ED-2, ED-3, ED-4, ED-1A, ED-2A, ED-3A, ED-4A, CA-1, CA-2, TS-1, TS-2, TS-3, TS-4, OF-1A, OF-2A, GT-1, GT-2, GT-3, GT-4, OWS-1, OWS-2
Soil	Benzo(a)pyrene	SVOC	RRSL/SSL	
Soil	Benzo(b)fluoranthene	SVOC	RRSL/SSL	
Soil	Benzo(k)fluoranthene	SVOC	SSL	
Soil	Bis(2-chloroethoxy)ether	SVOC	RRSL	
Soil	bis(2-ethylhexyl)phthalate	SVOC	SSL	
Soil	Butylbenzyl phthalate	SVOC	SSL	
Soil	Dibenz(a,h)anthracene	SVOC	RRSL	
Soil	1,4-Dichlorobenzene	SVOC	SSL	
Soil	Diethylphthalate	SVOC	SSL	
Soil	Hexachlorobenzene	SVOC	RRSL/SSL	
Soil	Hexachloropentadiene	SVOC	SSL	
Soil	Indeno(1,2,3-c,d)pyrene	SVOC	RRSL/SSL	
Soil	N-nitroso-n-propylamine	SVOC	RRSL	
Soil	Pentachlorophenol	SVOC	SSL	
Soil	1,2,4-Trichlorobenzene	SVOC	SSL	
Soil	Arsenic	Metals	SSL	MB-1, MB-2, MB-3
Soil	Chromium (hexavalent)	Metals	RRSL/SSL	
Soil	Thallium	Metals	SSL	No samples analyzed for thallium
Water	Chloromethane	VOC	TWRSL	B-1, B-2, B-3, MW-1, OWS-TW-1, GP-1, GP-1d, GP-2, GP-3, GP-4, GP-4d, GP-5, GP-6, GP-7, GP-8, GP-9, GP-10, GP-11, GP-12, GP-13, GP-14, GP-14d, GP-15, GP-18D, GP-19, GP-20, GP-21, GP-22, GP-23, GP-24, GP-25, GP-26, GP-27, GP-28, GP-29, GP-30, GP-31, GP-32, GP-33, HP-1, HP-2, SW-2, SW-06, SW-09, SW-11
Water	1,2-Dibromo-3-chloropropane	VOC	MCL	
Water	1,1-Dichloroethane	VOC	TWRSL	
Water	1,1-Dichloroethene	VOC	TWRSL	
Water	1,3-Dichloropropene	VOC	TWRSL	
Water	Ethylbenzene	VOC	TWRSL	
Water	Methanol	VOC	TWRSL	
Water	Naphthalene	VOC	TWRSL	
Water	1,1,2,2-Tetrachloroethane	VOC	TWRSL	
Water	Tetrachloroethene	VOC	SCWQC	
Water	Trichloroethene	VOC	TWRSL/SCWQC	
Water	Benzo(a)anthracene	SVOC	TWRSL	B-1, B-2, B-3, MW-1, OWS-TW-1, GP-1, GP-1d, GP-2, GP-3, GP-4, GP-4d, GP-5, GP-6, GP-7, GP-8, GP-9, GP-10, GP-11, GP-12, GP-13, GP-14, GP-14d, GP-15
Water	Benzo(a)pyrene	SVOC	MCL	
Water	Benzo(b)fluoranthene	SVOC	TWRSL	
Water	Benzo(k)fluoranthene	SVOC	TWRSL	
Water	4-Chloroaniline	SVOC	TWRSL	
Water	Bis(2-chloroethyl)ether	SVOC	TWRSL	
Water	Bis(2-chloroisopropyl)ether	SVOC	TWRSL	
Water	Chrysene	SVOC	TWRSL	
Water	Dibenz(a,h)anthracene	SVOC	TWRSL	
Water	Di-n-butylphthalate	SVOC	TWRSL	
Water	3,3'-Dichlorobenzidine	SVOC	TWRSL	
Water	4,6-Dinitro-2-methylphenol	SVOC	TWRSL	
Water	Hexachlorobenzene	SVOC	MCL	
Water	Hexachlorobutadiene	SVOC	TWRSL	
Water	Hexachloroethane	SVOC	TWRSL	
Water	Indeno(1,2,3-c,d)pyrene	SVOC	TWRSL	
Water	N-nitroso-n-propylamine	SVOC	TWRSL	
Water	3-Nitroaniline	SVOC	TWRSL	
Water	4-Nitroaniline	SVOC	TWRSL	
Water	Nitrobenzene	SVOC	TWRSL	
Water	2,4,6-Trichlorphenol	SVOC	TWRSL	
Water	Cobalt	Metals	TWRSL	No samples analyzed for cobalt

Notes:
VOC = volatile organic compound
SVOC = semi-volatile organic compound
SSL = Soil Screening Level
RRSL = Residential Regional Screening Level
TWRSL = Tap Water Regional Screening Level
MCL = Maximum Contaminant Level
SCWQC = South Carolina Water Quality Criteria

TABLE 3

Soil Sampling Results From Acetone UST Pipeline Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Sample Number	Sample Depth	Sample Date	EPA Method		Units	Acetone	Methanol
			Actone	Methanol			
HA-1	10	10/23/96	8240B	8015	mg/kg	**5.25**	< 10.0
HA-1	12.75	10/23/96	8240B		mg/kg	**242**	NT
HA-2	12.5	10/23/96	8240B		mg/kg	**20.2**	NT
HA-3	12	10/23/96	8240B		mg/kg	**2.7**	NT
HA-5	3.5	10/24/96	8240B	8015	mg/kg	**204**	< 10.0
HA-6	3.5	10/24/96	8240B		mg/kg	< 1.00	NT
STB-1	12 - 14	11/5/96	8260	8000M	mg/kg	0.127	< 2.00
STB-2	8 - 10	11/5/96	8260		mg/kg	< 0.025	NT
STB-3	8 - 10	11/5/96	8260		mg/kg	< 0.025	NT
STB-4	10 - 12	11/5/96	8260		mg/kg	< 0.025	NT
STB-5	10 - 12	11/6/96	8260		mg/kg	< 0.025	NT
STB-6	12 - 14	11/9/96	8260	8000M	mg/kg	0.584	< 2.00
EPA Residential RSL					mg/kg	61,000	31,000
EPA Industrial RSL					mg/kg	630,000	310,000
EPA SSL					mg/kg	2.4	1.6

Notes:
Samples HA-1 through HA-6 collected by ESE Environmental, Inc.
mg/kg = milligrams per kilogram.
ug/kg = micrograms per kilogram.
EPA = United States Environmental Protection Agency.
HA samples analyzed by Specialized Assays, Inc.
STB samples analyzed by Environmental Testing & Consulting, Inc.
Samples only analyzed for acetone and methanol.
RSL = Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NT = not tested.
NE = not established.
Bold values exceed SSL.

TABLE 4

Soil Test Boring Soil Sampling Results
From Heat Treat Cleaning Water Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Sample ID	Depth	Date	Nitrate	Nitrite	Units
B-1	Surface	7/12/01	29	190	mg/kg
	1 foot	7/12/01	1.3	10	mg/kg
	5 feet	7/12/01	2.2	3.6	mg/kg
	10 feet	7/12/01	2.2	1.3	mg/kg
B-1A	12 feet	8/23/01	1.3	<0.27	mg/kg
	14 feet	8/23/01	2.0	<0.26	mg/kg
	16 feet	8/23/01	1.8	<0.28	mg/kg
	18 feet	8/23/01	2.9	<0.28	mg/kg
	20 feet	8/23/01	1.4	<0.27	mg/kg
B-2	Surface	7/12/01	3,100	1,300	mg/kg
	1 foot	7/12/01	0.54	12	mg/kg
	5 feet	7/12/01	1.2	2.3	mg/kg
	10 feet	7/12/01	2.4	0.41	mg/kg
B-3	Surface	7/12/01	2,400	1,200	mg/kg
	1 foot	7/12/01	35	19	mg/kg
	5 feet	7/12/01	0.52	0.72	mg/kg
	10 feet	7/12/01	1.8	1.6	mg/kg
B-3A	12 feet	8/23/01	<0.24	<0.24	mg/kg
	14 feet	8/23/01	<0.25	<0.25	mg/kg
	16 feet	8/23/01	0.60	0.58	mg/kg
	18 feet	8/23/01	<0.27	<0.27	mg/kg
	20 feet	8/23/01	<0.26	<0.26	mg/kg
B-4	Surface	7/12/01	0.4	1,900	mg/kg
	1 foot	7/12/01	74	33	mg/kg
	5 feet	7/12/01	< 0.24	< 0.24	mg/kg
	10 feet	7/12/01	< 0.24	0.33	mg/kg
B-5	Surface	7/12/01	260	350	mg/kg
	1 foot	7/12/01	2.7	< 0.26	mg/kg
	5 feet	7/12/01	120	52	mg/kg
	10 feet	7/12/01	0.87	0.67	mg/kg
B-6	Surface	7/12/01	< 0.22	3.1	mg/kg
	1 foot	7/12/01	< 0.24	< 0.24	mg/kg
	5 feet	7/12/01	< 0.30	< 0.30	mg/kg
	10 feet	7/12/01	< 0.29	< 0.29	mg/kg
EPA Residential RSL			130,000	7,800	mg/kg
EPA Industrial RSL			1,600,000	100,000	mg/kg
EPA SSL			NE	NE	mg/kg

Notes:
Boring B-6 was drilled as a control boring.
Depths reported in feet below ground surface.
Samples only analyzed for nitrate and nitrite.
Nitrate analyzed by EPA Method 353.2.
Nitrite analyzed by EPA Method 354.1.
mg/kg = milligram per kilogram
Sample B-6 collected as a background soil sample.
Samples analyzed by Shealy Environmental Services, Inc.
EPA = United States Environmental Protection Agency.
RSL = Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.

TABLE 5

Surface Soil Sampling Results From
Heat Treat Cleaning Water Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Sample ID	Sample Date	Sample Depth	Nitrate	Nitrite	Units
SS-1	8/22/01	0 - 0.5	<0.21	<0.21	mg/kg
SS-2	8/22/01	0 - 0.5	29	<0.26	mg/kg
SS-3	8/22/01	0 - 0.5	2.1	<0.22	mg/kg
SS-4	8/22/01	0 - 0.5	<0.21	<0.21	mg/kg
SS-5	8/22/01	0 - 0.5	1.9	<0.22	mg/kg
SS-6	8/22/01	0 - 0.5	<0.21	<0.21	mg/kg
SS-7	8/22/01	0 - 0.5	0.58	<0.20	mg/kg
SS-8	8/22/01	0 - 0.5	<0.21	<0.21	mg/kg
SS-9	8/22/01	0 - 0.5	<0.21	<0.21	mg/kg
SS-10	8/22/01	0 - 0.5	<0.20	<0.20	mg/kg
SS-11	8/22/01	0 - 0.5	<0.20	<0.20	mg/kg
SS-12	8/22/01	0 - 0.5	0.52	<0.32	mg/kg
SS-13	8/22/01	0 - 0.5	<0.20	<0.20	mg/kg
SS-14	8/22/01	0 - 0.5	<0.21	<0.21	mg/kg
EPA Residential RSL			130,000	7,800	mg/kg
EPA Industrial RSL			1,600,000	100,000	mg/kg
EPA SSL			NE	NE	mg/kg

Notes:
Samples are composite soil samples from 0 to 0.5 feet below ground surface.
Samples only analyzed for nitrate and nitrite.
Nitrate analyzed by EPA Method 353.2
Nitrite analyzed by EPA Method 354.1
mg/kg = milligram per kilogram
Samples analyzed by Shealy Environmental Services, Inc.
EPA = United States Environmental Protection Agency.
RSL = Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.

TABLE 6

Confirmation Soil Sampling Results From
Heat Treat Cleaning Water Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Sample ID	Sample Date	Sample Depth	Nitrate	Nitrite	Units
SS-15	4/11/02	1 - 1.5	99	< 0.25	mg/kg
SS-16	4/11/02	1 - 1.5	< 0.25	< 0.25	mg/kg
SS-17	4/11/02	1 - 1.5	< 0.26	0.28	mg/kg
SS-18	4/11/02	1 - 1.5	< 0.26	< 0.26	mg/kg
SS-19	4/11/02	1 - 1.5	< 0.25	< 0.25	mg/kg
SS-20	4/11/02	1 - 1.5	1.2	< 0.26	mg/kg
SS-21	4/11/02	5 - 5.5	18	0.73	mg/kg
SS-22	4/11/02	1 - 1.5	280	66	mg/kg
EPA Residential RSL			130,000	7,800	mg/kg
EPA Industrial RSL			1,600,000	100,000	mg/kg
EPA SSL			NE	NE	mg/kg

Notes:
Sample depths reported in feet below ground surface (0 to 0.5 feet below base of excavation).
Samples only analyzed for nitrate and nitrate.
Nitrate analyzed by EPA Method 353.2
Nitrite analyzed by EPA Method 354.1
mg/kg = milligram per kilogram
Samples analyzed by Shealy Environmental Services, Inc.
EPA = United States Environmental Protection Agency.
RSL = Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.

TABLE 7

Soil Sampling Results From Metals Baghouse
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	EPA RRSL	EPA IRSL	EPA SSL	SC Range	EPA TCLP	Units	Sample Identification MB-1	MB-2	MB-3
Arsenic	6010B	0.39	1.6	0.0013	ND - 210	-	mg/kg	< 0.32	< 0.31	**2.1**
Arsenic (TCLP)	6010B	-	-	-	-	5.0	mg/l	NA	NA	< 0.050
Barium	6010B	15,000	190,000	120	ND - 370	-	mg/kg	31	27	25
Chromium (total)	6010B	NE	NE	NE	ND - 140	-	mg/kg	68	180	170
Chromium (hexavalent)	6010B	0.29	5.6	0.00059	-	-	mg/kg	< 1.3	< 1.2	< 1.2
Lead	6010B	400	800	NE	ND - 200	-	mg/kg	28	14	15
Nickel	6010B	1,500	20,000	20	ND - 47	-	mg/kg	**30**	**46**	**73**
Sample Interval							ft bgs	0-0.5	0-0.5	0-0.5
Sample Date								3/13/02	3/13/02	3/13/02

Notes:
Samples only analyzed for eight RCRA metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver) and nickel
Samples also analyzed for hexavalent chromium.
TCLP = Toxicity Characteristic Leachate Procedure
EPA = United States Environmental Protection Agency
mg/kg = milligrams per kilogram
mg/l = milligrams per liter
ft bgs = feet below ground surface
NA = not analyzed
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
SC Range from Table 2 in "Elements in South Carolina Inferred Background Soil and Stream Sediment Samples."
NE = Not Established.
ND = Not Detected.
Bold values exceed RRSL or SSL.

TABLE 8

Soil Sampling Results From Scrap Metal Rolloff
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA RRSL	EPA IRSL	EPA SSL	SC Range	Sample Identification SM-1	SM-1A	SM-2	SM-2A	SM-3	SM-3A	SM-4	SM-4A
Acetone	8260B	ug/kg	61,000,000	630,000,000	2,400	-	< 16	NA	< 18	NA	27	NA	< 21	NA
Diethylphthalate	8270C	ug/kg	49,000,000	490,000,000	4.7	-	**56,000**	NA	< 420	NA	**3,300**	NA	< 380	NA
Bis(2-ethylhexyl)phthalate	8270C	ug/kg	35,000	120,000	17	-	**440**	NA	< 420	NA	< 400	NA	< 380	NA
Barium	6010B	mg/kg	15,000	190,000	120	ND - 370	35	NA	32	NA	27	NA	44	NA
Cadmium	6010B	mg/kg	70	800	0.52	ND - 17	< 0.12	NA	< 0.13	NA	0.16	NA	< 0.11	NA
Chromium	6010B	mg/kg	NE	NE	NE	ND - 140	140	19	32	NA	36	NA	21	NA
Lead	6010B	mg/kg	400	800	NE	ND - 200	30	NA	28	NA	23	NA	25	NA
Nickel	6010B	mg/kg	1,500	20,000	20	ND - 47	**27**	NA	**23**	NA	17	NA	9.2	NA
Oil and Grease	9071A	mg/kg	NE	NE	NE	-	830	170	< 63	160	130	< 61	140	98
Sample Interval		ft bgs					0-0.5	1-1.5	0-0.5	1-1.5	0-0.5	1-1.5	0-0.5	1-1.5
Sample Date							3/13/02	4/11/02	3/13/02	4/11/02	3/13/02	4/11/02	3/13/02	4/11/02

Notes:
Samples SM-1 through SM-4 analyzed for TCL VOCs, TCL SVOCs, Oil & Grease, and the eight RCRA metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver) and nickel.
Samples SM-1A, SM-2A, SM-3A, and SM-4A were collected post excavation.
Sample SM-1A was only analyzed for chromium. Samples SM-1A through SM-4A were analyzed for Oil & Grease.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regiona Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.
SC Range from Table 2 in "Elements in South Carolina Inferred Background Soil and Stream Sediment Samples."
ND = Not Detected.
Bold values exceed RRSL or SSL.

TABLE 9

Soil Sampling Results From Empty Drum Storage Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA RRSL	EPA IRSL	EPA SSL	Sample Identification ED-1	ED-1A	ED-2	ED-2A	ED-3	ED-3A	ED-4	ED-4A
Acetone	8260B	ug/kg	61,000,000	630,000,000	2,400	< 21	NA	< 20	NA	25	NA	< 21	NA
Butyl benzyl phthalate	8270C	ug/kg	260,000	910,000	200	620	NA	< 350	NA	< 360	NA	< 360	NA
Di-n-butyl phthalate	8270C	ug/kg	6,100,000	62,000,000	1,700	**5,100**	NA	< 350	NA	< 360	NA	< 360	NA
Diethylphthalate	8270C	ug/kg	49,000,000	490,000,000	4.7	**16,000**	NA	**530**	NA	**420**	NA	< 360	NA
Bis(2-ethylhexyl)phthalate	8270C	ug/kg	35,000	120,000	17	**1,000**	NA	< 350	NA	< 360	NA	< 360	NA
Oil and Grease	9071A	mg/kg	NE	NE	NE	1,700	100	780	86	5,800	160	370	130
Sample Interval		ft bgs				0-0.5	1-1.5	0-0.5	1-1.5	0-0.5	1-1.5	0-0.5	1-1.5
Sample Date						3/13/02	4/11/02	3/13/02	4/11/02	3/13/02	4/11/02	3/13/02	4/11/02

Notes:
Samples ED-1 through ED-4 analyzed TCL VOCs, TCL SVOCs, and Oil & Grease.
Samples ED-1A, ED-2A, ED-3A, and ED-4A were collected post excavation and only analyzed for Oil & Grease.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.
Bold values exceed RRSL or SSL

TABLE 10

Soil Sampling Results From Compounding Exhaust Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA			Sample ID	
			RRSL	IRSL	SSL	CA-1	CA-2
Di-n-butyl phthalate	8270C	ug/kg	6,100,000	62,000,000	1,700	< 400	**2800**
Diethylphthalate	8270C	ug/kg	49,000,000	490,000,000	5	**1,600**	**1,300,000**
Dimethylphthalate	8270C	ug/kg	NE	NE	NE	< 400	680
Bis(2-ethylhexyl)phthalate	8270C	ug/kg	35,000	120,000	17	< 400	**580**
Sample Interval		ft bgs				0-0.5	0-0.5

Notes:
Samples only analyzed for TCL SVOCs
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.
Bold values exceed RRSL or SSL.

TABLE 11

Soil Sampling Results From Tank Storage Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA			Sample Identification			
			RRSL	IRSL	SSL	TS-1	TS-2	TS-3	TS-4
Acetone	8260B	ug/kg	61,000,000	630,000,000	2,400	83	22	67	350
Diethylphthalate	8270C	ug/kg	49,000,000	490,000,000	5	< 380	**630**	< 400	< 420
Sample Interval		ft bgs				0-0.5	0-0.5	0-0.5	0-0.5

Notes:
Samples analyzed for TCL VOCs and TCL SVOCs.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.

TABLE 12

Soil Sampling Results From Storm Water Outfalls
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA RRSL	EPA IRSL	EPA SSL	SC Range	Sample Identification OF-1	OF-1A	OF1-2	OF1-3	OF1-4	OF-2	OF-2A
Diethylphthalate	8270C	ug/kg	49,000,000	490,000,000	4.7	-	NA	**820**	NA	NA	NA	NA	< 390
Benzo(a)anthracene	8270C	ug/kg	150	2,100	10	-	NA	< 440	NA	NA	NA	NA	**1,100**
Benzo(a)pyrene	8270C	ug/kg	15	210	3.5	-	NA	< 440	NA	NA	NA	NA	**1,200**
Benzo(b)fluoranthene	8270C	ug/kg	150	2,100	35	-	NA	< 440	NA	NA	NA	NA	**1,900**
Benzo(g,h,i)perylene	8270C	ug/kg	NE	NE	NE	-	NA	< 440	NA	NA	NA	NA	610
Benzo(k)fluoranthene	8270C	ug/kg	1,500	21,000	350	-	NA	< 440	NA	NA	NA	NA	**760**
Chrysene	8270C	ug/kg	15,000	210,000	1,100	-	NA	< 440	NA	NA	NA	NA	**1,600**
Fluoranthene	8270C	ug/kg	2,300,000	22,000,000	70,000	-	NA	< 440	NA	NA	NA	NA	2,100
Indeno(1,2,3-c,d)pyrene	8270C	ug/kg	150	2,100	120	-	NA	< 440	NA	NA	NA	NA	**450**
Phenanthrene	8270C	ug/kg	NE	NE	NE	-	NA	< 440	NA	NA	NA	NA	580
Pyrene	8270C	ug/kg	1,700,000	17,000,000	9,500	-	NA	< 440	NA	NA	NA	NA	1,700
Arsenic	6010B	mg/kg	0.39	1.6	0.0013	ND - 210	**1.2**	NA	NA	NA	NA	**2.7**	NA
Barium	6010B	mg/kg	15,000	190,000	120	ND - 370	25	NA	NA	NA	NA	32	NA
Chromium	6010B	mg/kg	NE	NE	NE	ND - 140	19	NA	NA	NA	NA	11	NA
Lead	6010B	mg/kg	400	800	NE	ND - 200	20	NA	NA	NA	NA	22	NA
Nickel	6010B	mg/kg	1,500	20,000	20	ND - 47	12	NA	NA	NA	NA	5.4	NA
Selenium	6010B	mg/kg	390	5,100	0.40	ND - 2.4	**1.5**	NA	NA	NA	NA	**1.0**	NA
Oil and Grease	9071A	mg/kg	NE	NE	NE	-	NA	2,600	< 62	280	< 62	NA	920
Sample Depth		ft/bgs					0 - 0.5	0 - 0.5	0 - 0.5	0 - 0.5	0 - 0.5	0 - 0.5	0 - 0.5
Sample Date							4/19/02	4/24/02	8/28/02	8/28/02	8/28/02	4/19/02	4/24/02

Notes:
Samples OF-1 and OF-2 analyzed for the eight RCRA metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver) and nickel.
Samples OF-1A and OF-2A analyzed for TCL VOCs, TCL SVOCs, and Oil & Grease.
Samples OF1-2, OF1-3, and OF1-4 only analyzed for Oil & Grease.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.
SC Range from Table 2 in "Elements in South Carolina Inferred Background Soil and Stream Sediment Samples."
ND = Not Detected.
Bold values exceed RRSL or SSL.

TABLE 13

Soil Sampling Results From Oil/Water Separator
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA			Sample Identification			
			RRSL	IRSL	SSL	GT-1	GT-2	GT-3	GT-4
Acetone	8260B	ug/kg	61,000,000	630,000,000	2,400	86	< 22	280	290
Oil and Grease	9071A	mg/kg	NE	NE	NE	< 61	190	86	< 67
Sample Interval		ft bgs				8-10	8-10	8-10	8-10
Sample Date						3/18/02	3/18/02	3/18/02	3/18/02

Notes:
Samples analyzed for TCL VOCs, TCL SVOCs, and Oil & Grease.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.

TABLE 14

Soil Sampling Results Following Removal
of Oil/Water Separator
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA			Sample ID	
			RPRG	IPRG	SSL	OWS-1	OWS-2
Acetone	8260B	ug/kg	61,000,000	630,000,000	2,400	56	44
2-Butanone (MEK)	8260B	ug/kg	28,000,000	200,000,000	1,000	17	< 11
1,1-Dichloroethane	8260B	ug/kg	3,300	17,000	0.68	**140**	< 5.4
Ethylbenzene	8260B	ug/kg	5,400	27,000	1.5	**7.4**	< 5.4
Toluene	8260B	ug/kg	5,000,000	45,000,000	590	13	< 5.4
Xylenes (total)	8260B	ug/kg	630,000	2,700,000	190	100	< 5.4
Diethylphthalate	8270C	ug/kg	49,000,000	490,000,000	4.7	**89,000**	**9,600**
Bis(2-Ethylhexyl)phthalate	8270C	ug/kg	35,000	120,000	17	< 520	**800**
Oil and Grease	9071A	mg/kg	NE	NE	NE	420	200
Sample Interval		ft bgs				18.5-19	18.5-19
Sample Date						4/11/02	4/11/02

Notes:
Samples analyzed for TCL VOCs, TCL SVOCs, and Oil & Grease.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RPRG = Residential Preliminary Remediation Goal.
IPRG = Industrial Preliminary Remediation Goal.
SSL = Soil Screening Level (Riak-based).
NE = Not Established.
Bold values exceed RRSL or SSL.

TABLE 15

Soil Sampling Results From Acetone UST Closure
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA			Sample Identification			
			RRSL	IRSL	SSL	B-1	B-2	B-3	B-4
Acetone	8260B	ug/kg	61,000,000	630,000,000	2,400	33	< 22	44	< 22
2-Butanone (MEK)	8260B	ug/kg	28,000,000	200,000,000	1,000	14	< 11	< 10	< 11
Sample Interval		ft bgs				9-11	9-11	9-11	9-11
Sample Date						5/20/02	5/20/02	5/20/02	5/20/02

Notes:
Samples only analyzed for TCL VOCs.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
NE = Not Established.

TABLE 16

Soil Sampling Results From
Former Hazardous Waste Accumulation Building
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA			Sample ID	
			RRSL	IRSL	SSL	GP-16	GP-17
Tetrachloroethene	8260B	ug/kg	22,000	110,000	4.4	**1,200**	**64**
Sample Interval		ft bgs				5	3
Sample Date						1/27/05	1/27/05

Notes:
Samples only analyzed for TCL VOCs.
EPA = United States Environmental Protection Agency
ug/kg = micrograms per kilogram
ft bgs = feet below ground surface
Samples analyzed by Shealy Environmental Services, Inc.
RRSL = Residential Regional Screening Level.
IRSL = Industrial Regional Screening Level.
SSL = Soil Screening Level (Risk-based).
Bold value exceeds RRSL or SSL.

TABLE 17

Ground-Water Sampling Results From
Acetone UST Pipeline Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	EPA Tap Water RSL	Sample ID	
				HP-1	HP-2
Acetone	8260	µg/l	12,000	< 25.0	298
Methanol	8000M	mg/l	7.8	< 10.0	< 10.0
Boring Number				STB-1	STB-2
Sample Depth (Hydropunch screen)		ft/bgs		20 - 21	20.5 - 21.5
Sample Date				11/5/96	11/6/96

Notes:
Samples only analyzed for acetone and methanol
EPA = United States Environmental Protection Agency
RSL = Regional Screening Level
µg/l = micrograms per liter
mg/l = milligrams per liter
ft/bgs = feet below ground surface
Samples only analyzed for acetone and methanol
No other VOCs analyzed
HP-1 collected from boring STB-1
HP-2 collected from boring STB-6

TABLE 18

Ground-Water Sampling Results From
Heat Treat Cleaning Water Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	MCL	Sample ID	
				TW-1	TW-2
Nitrate	353.2	mg/l	10	2.5	2.2
Nitrite	354.1	mg/l	1	0.066	0.056
Sample Interval (well screen)		ft/bgs		22.5-32.5	21.7-31.8
Date Sampled				2/11/02	2/11/02

Notes:
Sample only analyzed for nitrate and nitrite.
EPA = United States Environmental Protection Agency
mg/l = milligrams per liter
ft/bgs = feet below ground surface
MCL = Maximum Contaminant Level promulgated by the EPA
Samples analyzed by Shealy Environmental Services, Inc.

TABLE 19

Ground-Water Sampling Results From
Oil/Water Separator Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	MCL	EPA Tap Water RSL	Sample ID	
					OWS-TW-1	MW-1
Acetone	8260B	µg/l	NE	12,000	79	150
2-Butanone (MEK)	8260B	µg/l	NE	4,900	15	< 10
Chloroethane	8260B	µg/l	NE	21,000	< 5.0	100
1,1-Dichloroethane	8260B	µg/l	NE	2.4	**220**	**210**
1,1-Dichloroethene	8260B	µg/l	7	260	**15**	**9.3**
cis-1,2-Dichloroethene	8260B	µg/l	70	28	< 5.0	**32**
Ethylbenzene	8260B	µg/l	700	1.3	**28**	**26**
Naphthalene	8260B	µg/l	25	0.14	**6.4**	**9.3**
Styrene	8260B	µg/l	100	1,100	< 5.0	< 5.0
Toluene	8260B	µg/l	1,000	860	58	110
1,1,1-Trichloroethane	8260B	µg/l	200	7,500	180	75
Xylenes (total)	8260B	µg/l	10,000	190	**370**	**400**
Di-n-butyl phthalate	8270C	µg/l	NE	670	26	< 5,300
Diethylphthalate	8270C	µg/l	NE	11,000	**320,000**	**210,000**
Oil and Grease	9071A	mg/l	NE	NE	470	NA
Sample Interval (well screen)		ft/bgs			14 - 24	14 - 24
Sample Date					6/7/02	6/5/03

Notes:
OWS-TW-1 analyzed for TCL VOCs, TCL SVOCs, and Oil & Grease
MW-1 analyzed for TCL VOCs and TCL SVOCs
EPA = United States Environmental Protection Agency
µg/l = micrograms per liter
mg/l = milligrams per liter
ft/bgs = feet below ground surface
MCL = Maximum Contaminant Level promulgated by the EPA
NE = not established
NA = not analyzed
RSL = Regional Screening Level.
Samples analyzed by Shealy Environmental Services, Inc.
Bold values exceed MCL or RSL.

TABLE 20

Field-Screening Ground-Water Sampling Results From
Oil/Water Separator Area
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	MCL	EPA Tap Water RSL	Sample Identification								
					MW-1	GP-1	GP-1d	GP-2	GP-3	GP-4	GP-4d	GP-5	GP-6
Acetone	8260B	µg/l	NE	12,000	150	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20
Chloroethane	8260B	µg/l	NE	21,000	100	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
1,1-Dichloroethane	8260B	µg/l	NE	2.4	**210**	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
1,1-Dichloroethene	8260B	µg/l	7	260	**9.3**	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
cis-1,2-Dichloroethene	8260B	µg/l	70	28	**32**	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Ethylbenzene	8260B	µg/l	700	1.3	**26**	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Naphthalene	8260B	µg/l	25	0.14	**9.3**	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Tetrachloroethene	8260B	µg/l	5	9.7	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Toluene	8260B	µg/l	1,000	860	110	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
1,1,1-Trichloroethane	8260B	µg/l	200	7,500	75	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Trichloroethene	8260B	µg/l	5	0.44	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Xylenes	8260B	µg/l	10,000	190	**400**	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Diethylphthalate	8270C	µg/l	NE	11,000	**210,000**	< 5.2	< 5.2	< 5.4	< 5.2	NA	< 5.2	< 5.2	< 5.2
Sample Interval (well screen)		ft/bgs			14 - 24	10 - 15	30 - 35	10 - 15	10 - 15	9 - 19	37 - 42	15 - 25	10 - 15
Sample Date					6/5/03	6/2/03	6/4/03	6/2/03	6/2/03	6/2/03	6/4/03	6/5/03	6/2/03

Notes:
Samples analyzed for TCL VOCs and TCL SVOCs
EPA = United States Environmental Protection Agency
µg/l = micrograms per liter
MCL = Maximum Contaminant Level promulgated by the EPA
Samples analyzed by Shealy Environmental Services, Inc.
NE = not established
ft/bgs = feet below ground surface
Bold values exceed MCL or RSL.
RSL = Regional Screening Level.

TABLE 21

Field-Screening Ground-Water Sampling Results From
General Plant and Former Hazardous Waste Accumulation Areas
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	EPA Method	Units	MCL	EPA Tap Water RSL	Sample Identification												
					GP-7	GP-8	GP-9	GP-10	GP-11	GP-12	GP-13	GP-14	GP-14d	GP-15	B-1	B-2	B-3
Acetone	8260B	µg/l	NE	12,000	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20
Chloroethane	8260B	µg/l	NE	21,000	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
1,1-Dichloroethane	8260B	µg/l	NE	2.4	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
1,1-Dichloroethene	8260B	µg/l	7	260	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
cis-1,2-Dichloroethene	8260B	µg/l	70	28	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Ethylbenzene	8260B	µg/l	700	1.3	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Naphthalene	8260B	µg/l	25	0.14	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Tetrachloroethene	8260B	µg/l	5	9.7	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	**11,000**	**53**	**57**	< 5.0	< 5.0	< 5.0
Toluene	8260B	µg/l	1,000	860	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
1,1,1-Trichloroethane	8260B	µg/l	200	7,500	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Trichloroethene	8260B	µg/l	5	0.44	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	**16**	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Xylenes	8260B	µg/l	10,000	190	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Diethylphthalate	8270C	µg/l	NE	11,000	< 6.2	< 5.2	< 5.3	< 5.2	< 5.2	< 5.2	< 6.1	< 5.1	< 5.1	< 5.1	< 5.2	< 5.1	< 5.2
Sample Interval (well screen)		ft/bgs			15 - 25	10 - 20	12 - 22	20 - 30	20 - 30	14 - 24	16 - 26	16 - 26	38 - 43	16 - 26	10 - 20	15 - 25	10 - 20
Sample Date					6/5/03	6/4/03	6/4/03	6/4/03	6/4/03	6/4/03	6/4/03	6/5/03	6/5/03	6/5/03	6/5/03	6/5/03	6/5/03

Notes:
Samples analyzed for TCL VOCs and TCL SVOCs
EPA = United States Environmental Protection Agency
µg/l = micrograms per liter
ft/bgs = feet below ground surface
MCL = Maximum Contaminant Level promulgated by the EPA
Samples analyzed by Shealy Environmental Services, Inc. in Cayce, South Carolina
NE = not established
RSL = Regional Screening Level.
Bold values exceed MCL or RSL.

TABLE 22

Field-Screening Ground-Water Sampling Results From
Former Hazardous Waste Accumulation Building
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Sample ID	Sample Date	Depth (feet bgs)	EPA Method	Units	Constituents	
					PCE	TCE
GP-18D	1/27/05	50-54	8260B	µg/l	<5.0	<5.0
GP-19	2/1/05	18-22	8260B	µg/l	**350**	<5.0
GP-20	2/1/05	25-29	8260B	µg/l	<5.0	<5.0
GP-21	2/1/05	21-25	8260B	µg/l	**26**	<5.0
GP-22	2/1/05	21-25	8260B	µg/l	<5.0	<5.0
GP-23	2/1/05	10-15	8260B	µg/l	<5.0	<5.0
GP-24	2/1/05	11-14	8260B	µg/l	**460**	<5.0
	2/1/05	41-45	8260B	µg/l	**7.2**	<5.0
GP-25	2/1/05	11-15	8260B	µg/l	**5.1**	<5.0
GP-26	2/1/05	11-15	8260B	µg/l	**32**	<5.0
GP-27	2/1/05	11-15	8260B	µg/l	<5.0	<5.0
GP-28	2/2/05	45-49	8260B	µg/l	<5.0	<5.0
GP-29	2/2/05	16-20	8260B	µg/l	<5.0	<5.0
GP-30	2/2/05	16-20	8260B	µg/l	<5.0	<5.0
GP-31	2/2/05	16-20	8260B	µg/l	**32**	<5.0
GP-32	2/2/05	16-20	8260B	µg/l	<5.0	<5.0
GP-33	2/2/05	16-20	8260B	µg/l	<5.0	<5.0
EPA Drinking Water MCL					5	5

Notes:
Samples only analyzed for TCL VOCs
bgs = below ground surface.
µg/l = micrograms per liter.
PCE = tetrachloroethene (perchloroethylene)
TCE = trichloroethene.
EPA = United States Environmental Protection Agency.
MCL = Maximum Contaminant Level.
Bold values exceed MCL.

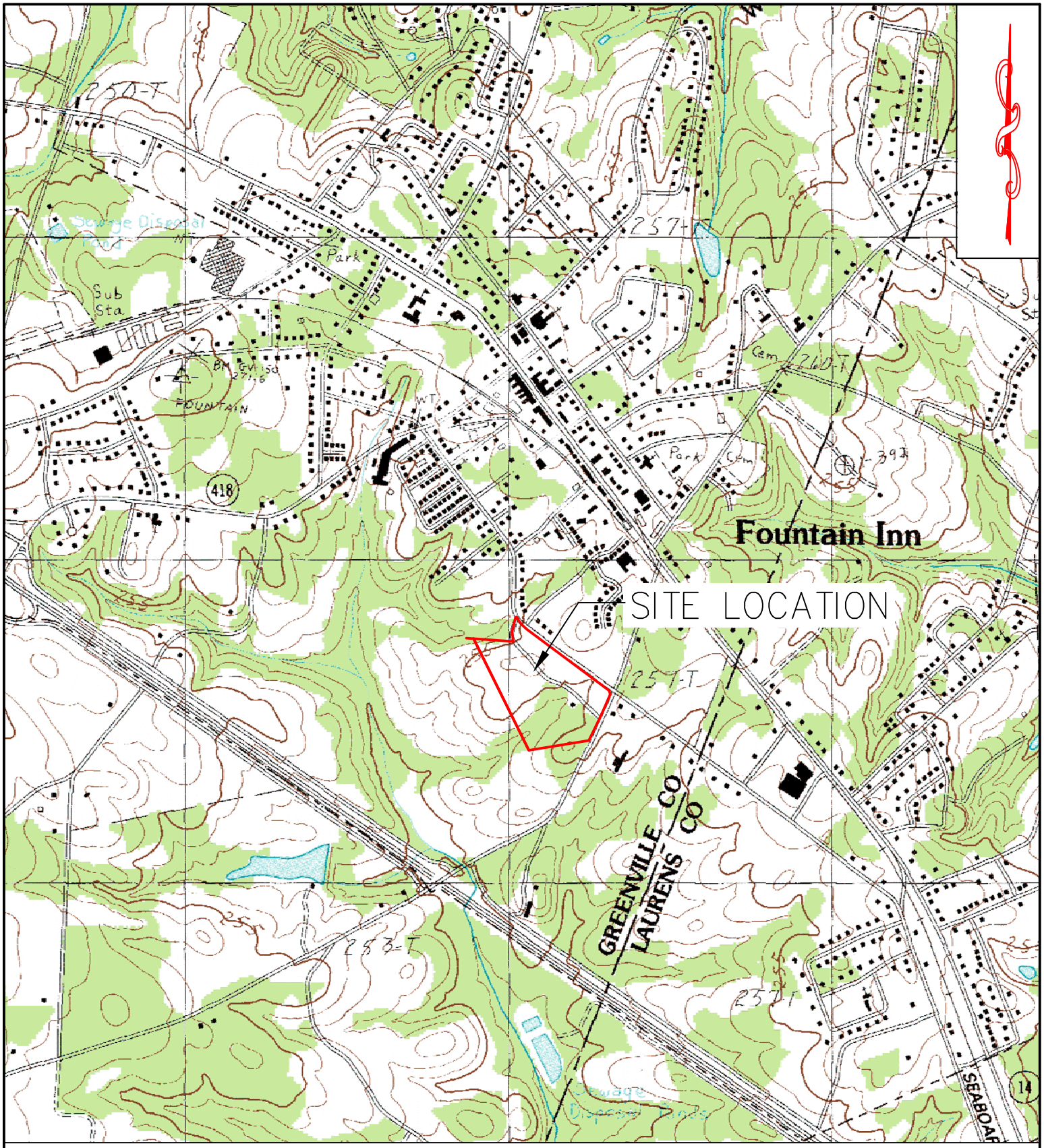
TABLE 23

Field-Screening Surface-Water Sampling Results From
Former Hazardous Waste Accumulation Building
Remedial Investigation/Feasibility Study Work Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Sample ID	Sample Date	EPA Method	Units	Constituents	
				PCE	TCE
SW-01	2/2/05	8260B	µg/l	**99**	< 5.0
SW-06	2/11/05	8260B	µg/l	< 5.0	< 5.0
SW-09	2/11/05	8260B	µg/l	**9.7**	< 5.0
SW-11	2/11/05	8260B	µg/l	< 5.0	< 5.0
South Carolina WQC				0.69	2.5

Notes:
Samples only analyzed for TCL VOCs.
µg/l = micrograms per liter.
PCE = tetrachloroethene (perchloroethylene)
TCE = trichloroethene.
EPA = United States Environmental Protection Agency.
WQC = Water Quality Criteria.
Bold values exceed WQC.

FIGURES



REFERENCE:
2001 DELORME STREET ATLAS USA

0.25 0.125 0 0.25 0.5

APPROXIMATE SCALE IN MILES

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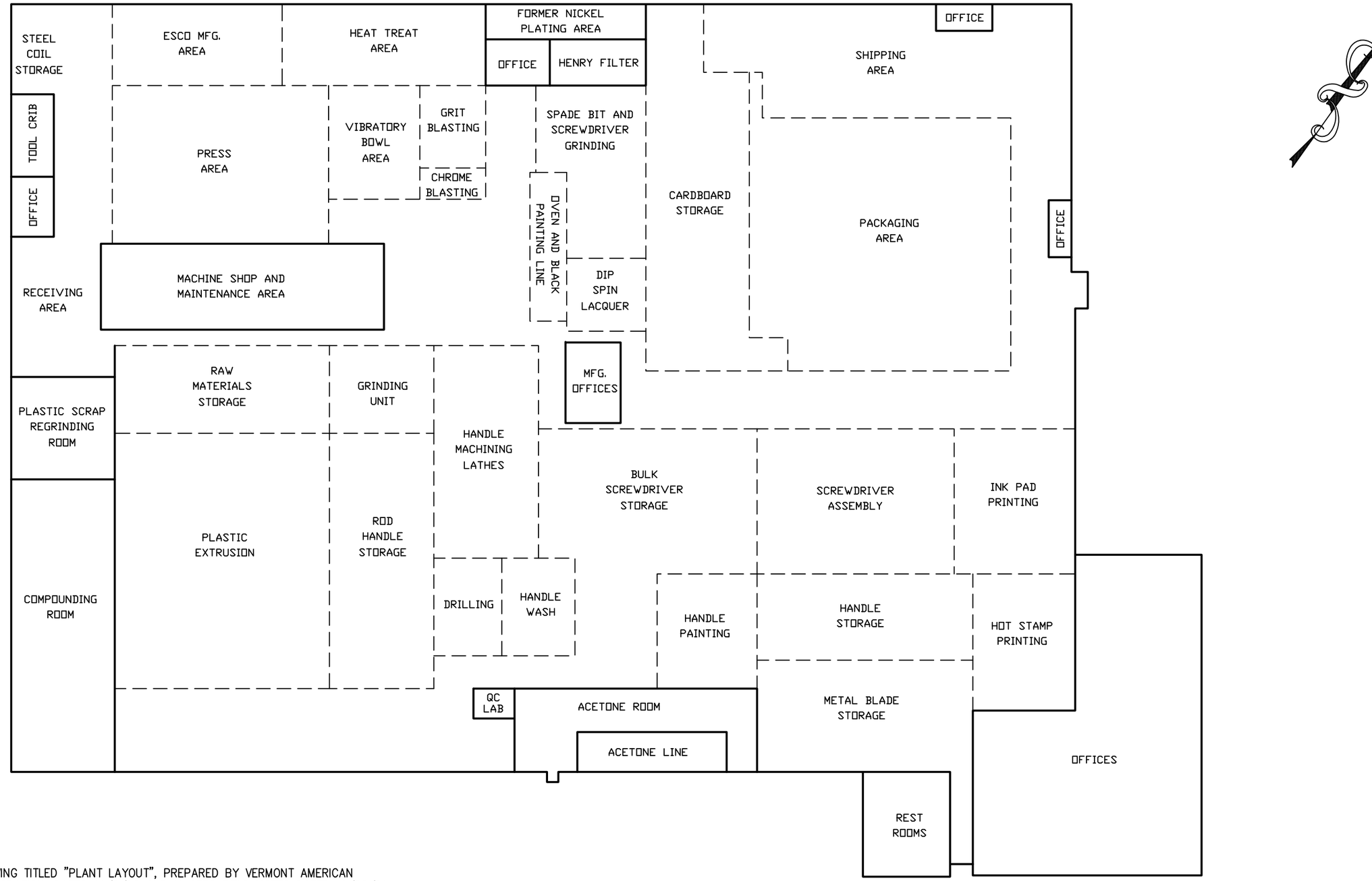
SITE LOCATION MAP
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
1

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 1.DWG	CHB	PSJ	PSJ	5/14/12	6251121007.01.01



REFERENCE: DRAWING TITLED "PLANT LAYOUT", PREPARED BY VERMONT AMERICAN CORPORATION, DATED 11/5/98 AND MACTEC FIELD NOTES DATED 11/28/01.

NOT TO SCALE

DRAWN CHB	DATE 5/14/12	REVISIONS		
CHECKED GWW	FILE FIGURE 2.DWG	No.	DESCRIPTION	BY
APPROVED PSJ	JOB NO: 6251121007.01.01			

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INTERIOR PLANT LAYOUT AS OF 2001
FORMER BOSCH/VERMONT AMERICAN CORPORATION FACILITY
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 2

BOUNDARY AND AS-BUILT SURVEY FOR

VERMONT AMERICAN CORPORATION

FOUNTAIN INN DIVISION

TOWN OF FOUNTAIN INN
GREENVILLE COUNTY
SOUTH CAROLINA
AUGUST 24, 2001

SCALE: 1" = 50'

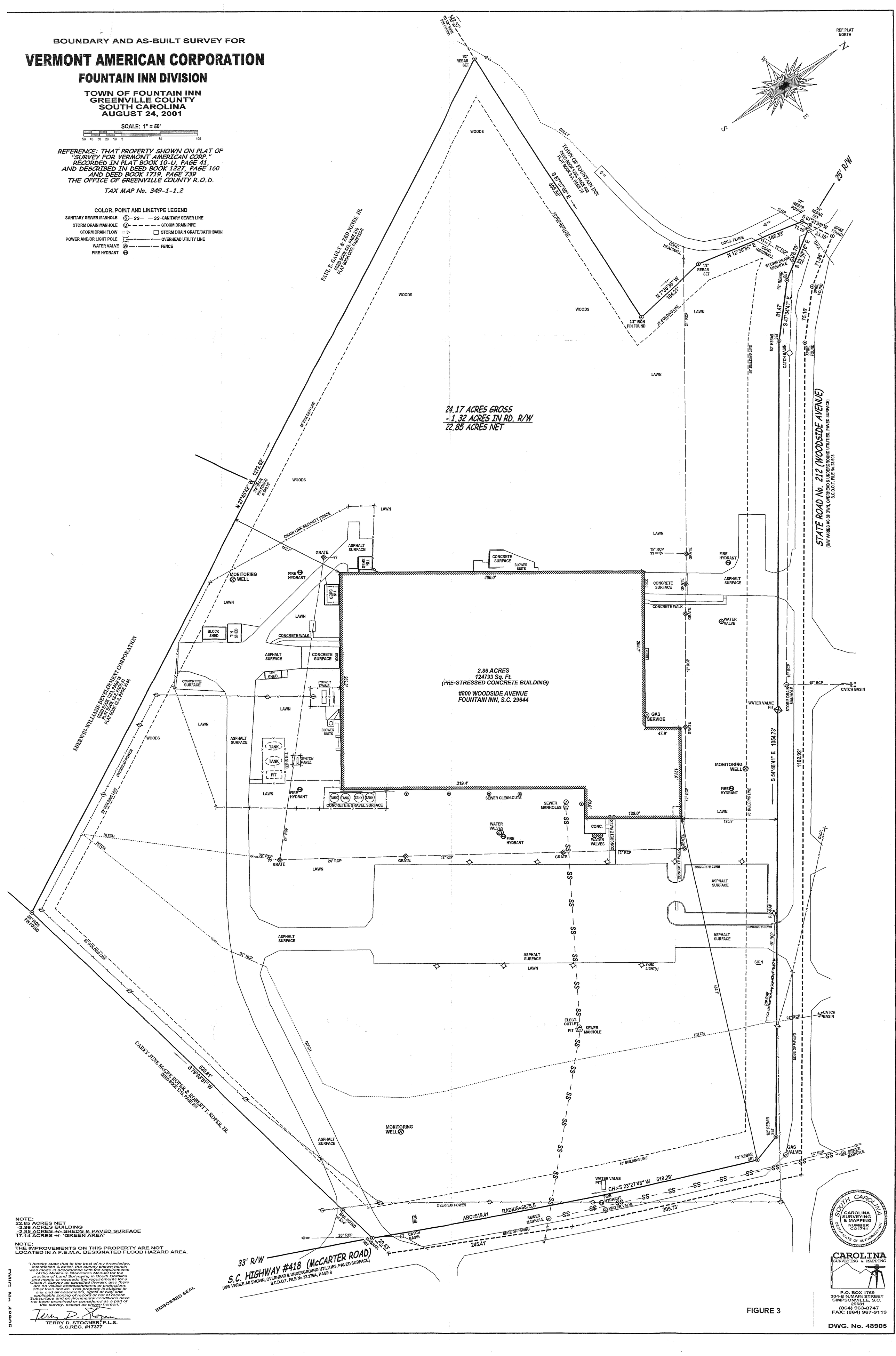
50 40 30 20 10 0 50 100

REFERENCE: THAT PROPERTY SHOWN ON PLAT OF "SURVEY FOR VERMONT AMERICAN CORP." RECORDED IN PLAT BOOK 10-U, PAGE 41, AND DESCRIBED IN DEED BOOK 1227, PAGE 160 AND DEED BOOK 1719, PAGE 739 THE OFFICE OF GREENVILLE COUNTY R.O.D.

TAX MAP No. 349-1-1.2

COLOR, POINT AND LINETYPE LEGEND

- SANITARY SEWER MANHOLE — SANITARY SEWER LINE
- STORM DRAIN MANHOLE — STORM DRAIN PIPE
- STORM DRAIN FLOW — STORM DRAIN GRATE/CATCHBASIN
- POWER AND/OR LIGHT POLE — OVERHEAD UTILITY LINE
- WATER VALVE — FENCE
- FIRE HYDRANT



NOTE:
22.85 ACRES NET
-2.86 ACRES BUILDING
-2.85 ACRES +/- SHEDS & PAVED SURFACE
17.14 ACRES +/- 'GREEN AREA'

NOTE:
THE IMPROVEMENTS ON THIS PROPERTY ARE NOT LOCATED IN A F.E.M.A. DESIGNATED FLOOD HAZARD AREA.

"I hereby state that to the best of my knowledge, information & belief, the survey shown hereon was made in accordance with the requirements of the Minimum Standards Manual for the practice of Land Surveying in South Carolina and meets or exceeds the requirements for a Class A Survey as specified therein; also there are no visible encroachments or projections other than shown. This property is subject to any and all easements, rights of way and applicable zoning of record or not of record. Subsurface and environmental conditions have not been examined or considered as a part of this survey, except as shown hereon."

TERRY D. STOGNER, P.L.S.
S.C.REG. #17377

EMBOSSED SEAL

FIGURE 3

SOUTH CAROLINA
CAROLINA SURVEYING & MAPPING
NUMBER C01744
CERTIFICATE OF AUTHORIZATION

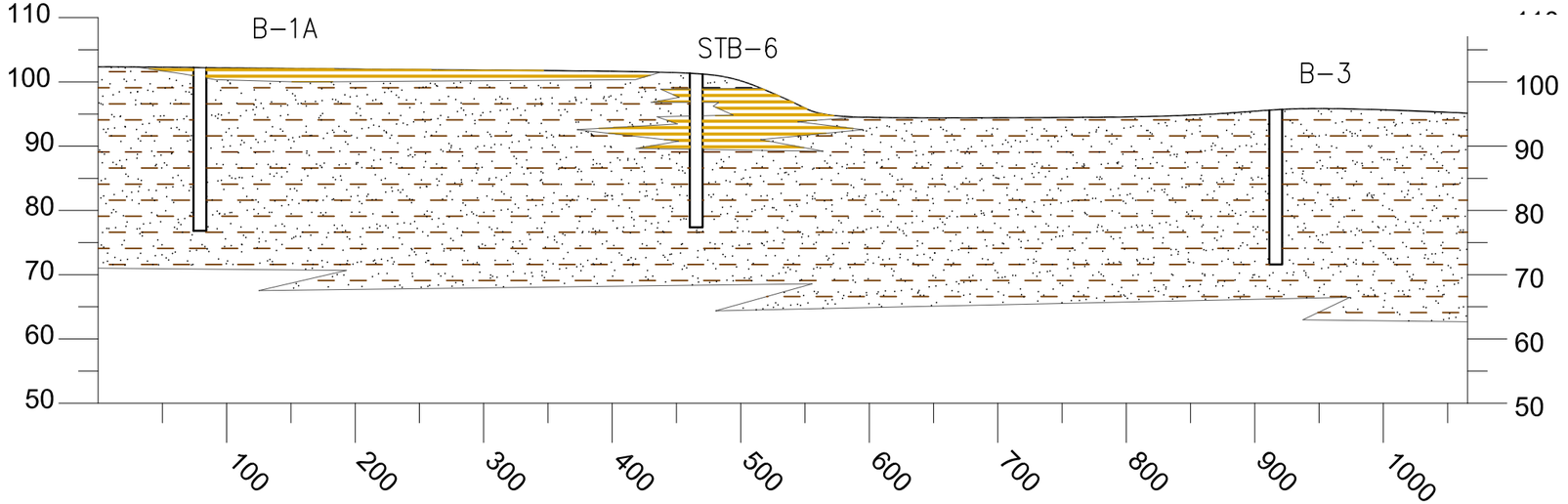
CAROLINA SURVEYING & MAPPING

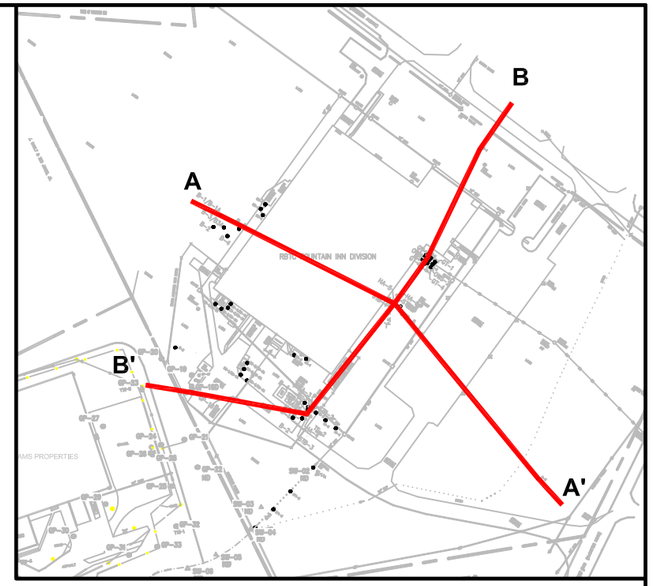
P.O. BOX 1769
304-B N.MAIN STREET
SIMPSONVILLE, S.C.
29681
(864) 963-8747
FAX: (864) 967-9119

DWG. No. 48905

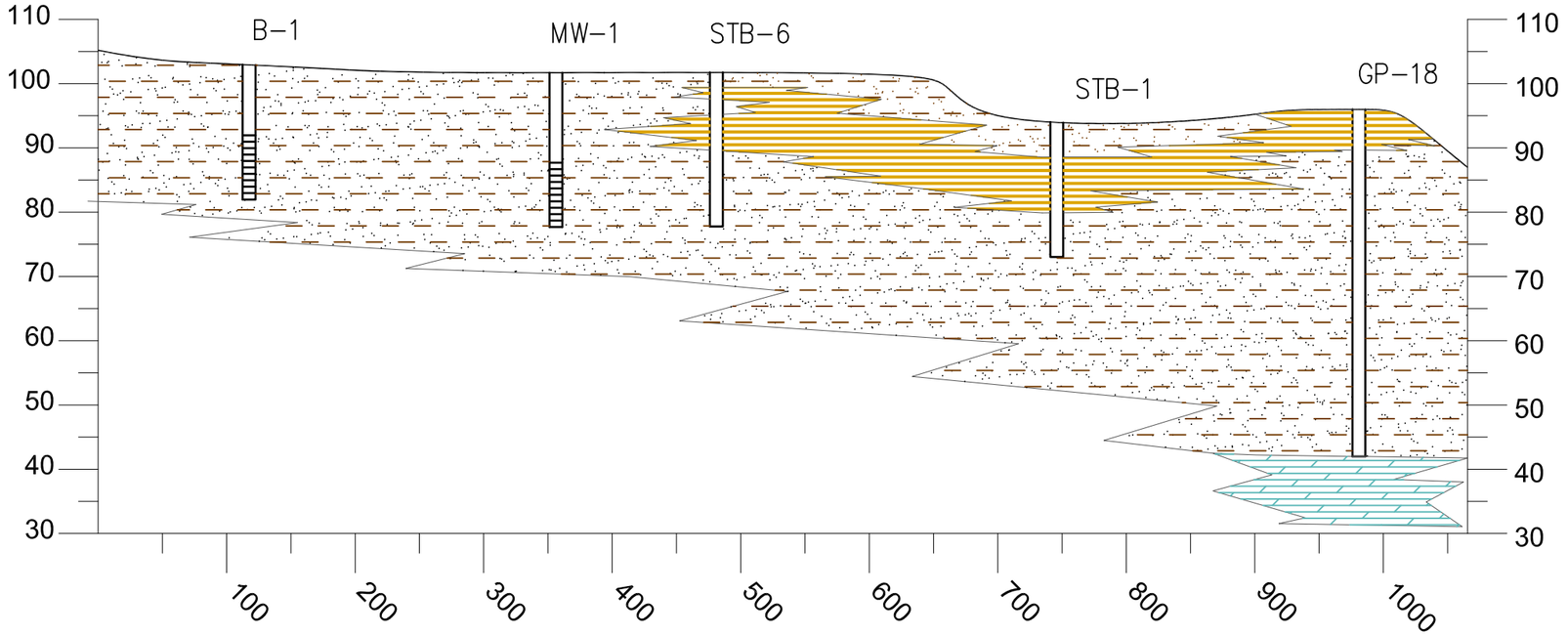
DWG No. 48905

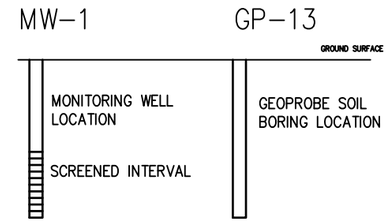
NORTHWEST **A** — **SOUTHEAST** **A'**





NORTHEAST **B** — **SOUTHWEST** **B'**





CLAY UNIT
(silty clay to a sandy clay)

SAND AND SILT UNIT
(silty sand to a clayey sand)

BEDROCK
(based on Geoprobe Refusal)

NOTE: LITHOLOGIC CONTACTS ARE ESTIMATED AND BASED ON FIELD OBSERVATIONS AND REGIONAL GEOLOGY.

ELEVATIONS BASED ON A BUILDING FINISHED FLOOR ELEVATION OF 102.39.

DRAWN	CHB	DATE	5/14/12
CHECKED	GWW	FILE	FIGURE 4.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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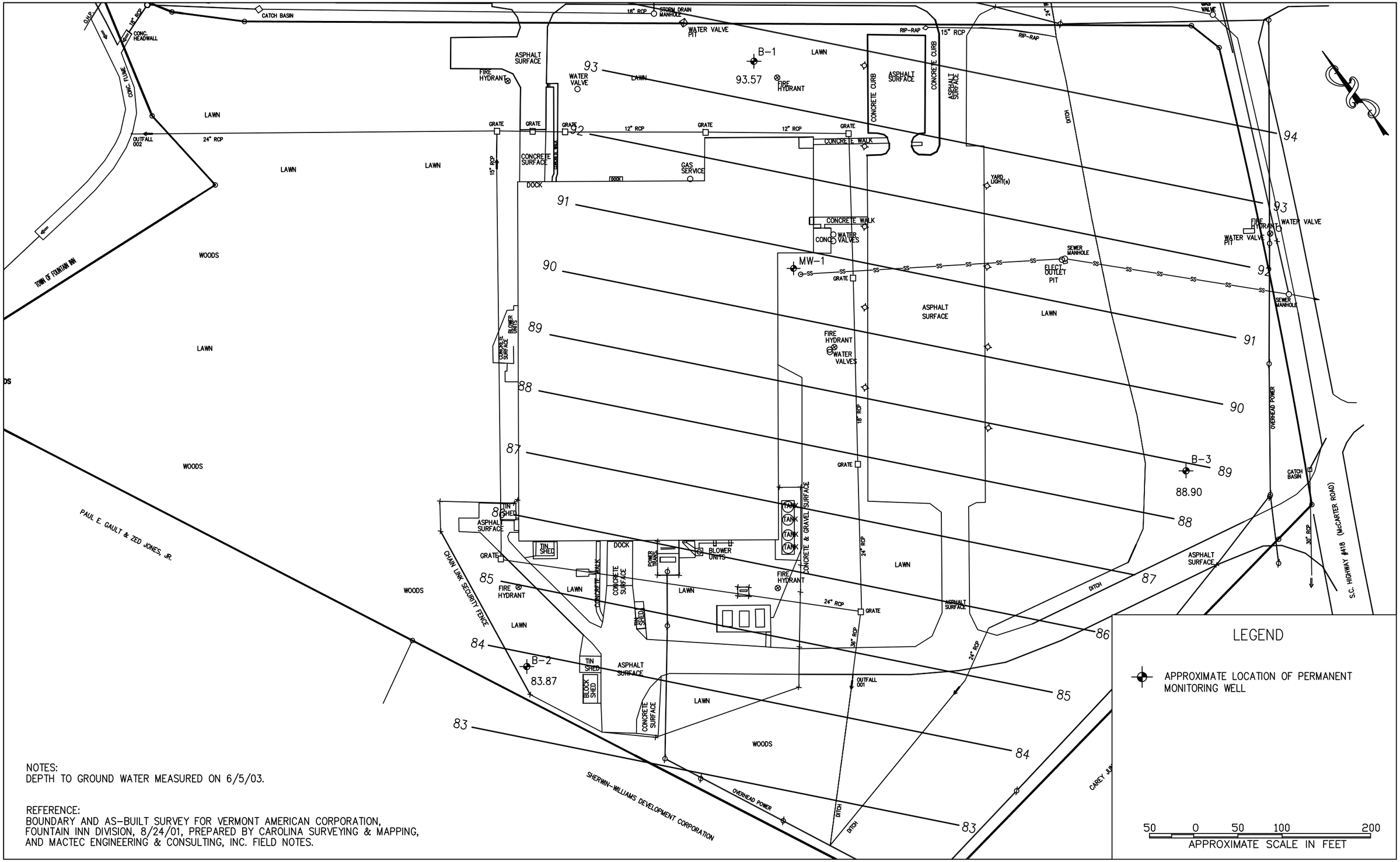
Fax: (864) 552-9699

LITHOLOGIC CROSS SECTIONS
A-A' AND B-B'
RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
4



NOTES:
DEPTH TO GROUND WATER MEASURED ON 6/5/03.

REFERENCE:
BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND MACTEC ENGINEERING & CONSULTING, INC. FIELD NOTES.

LEGEND

APPROXIMATE LOCATION OF PERMANENT MONITORING WELL

50 0 50 100 200
APPROXIMATE SCALE IN FEET

DRAWN CHB	DATE 5/14/12	REVISIONS No. / DESCRIPTION / BY	WATER TABLE ELEVATION CONTOUR MAP RBTC FOUNTAIN INN DIVISION FOUNTAIN INN, SOUTH CAROLINA
CHECKED GWW	FILE FIGURE 5.DWG		FIGURE 5
APPROVED PSJ	JOB NO: 6251121007.01.01		

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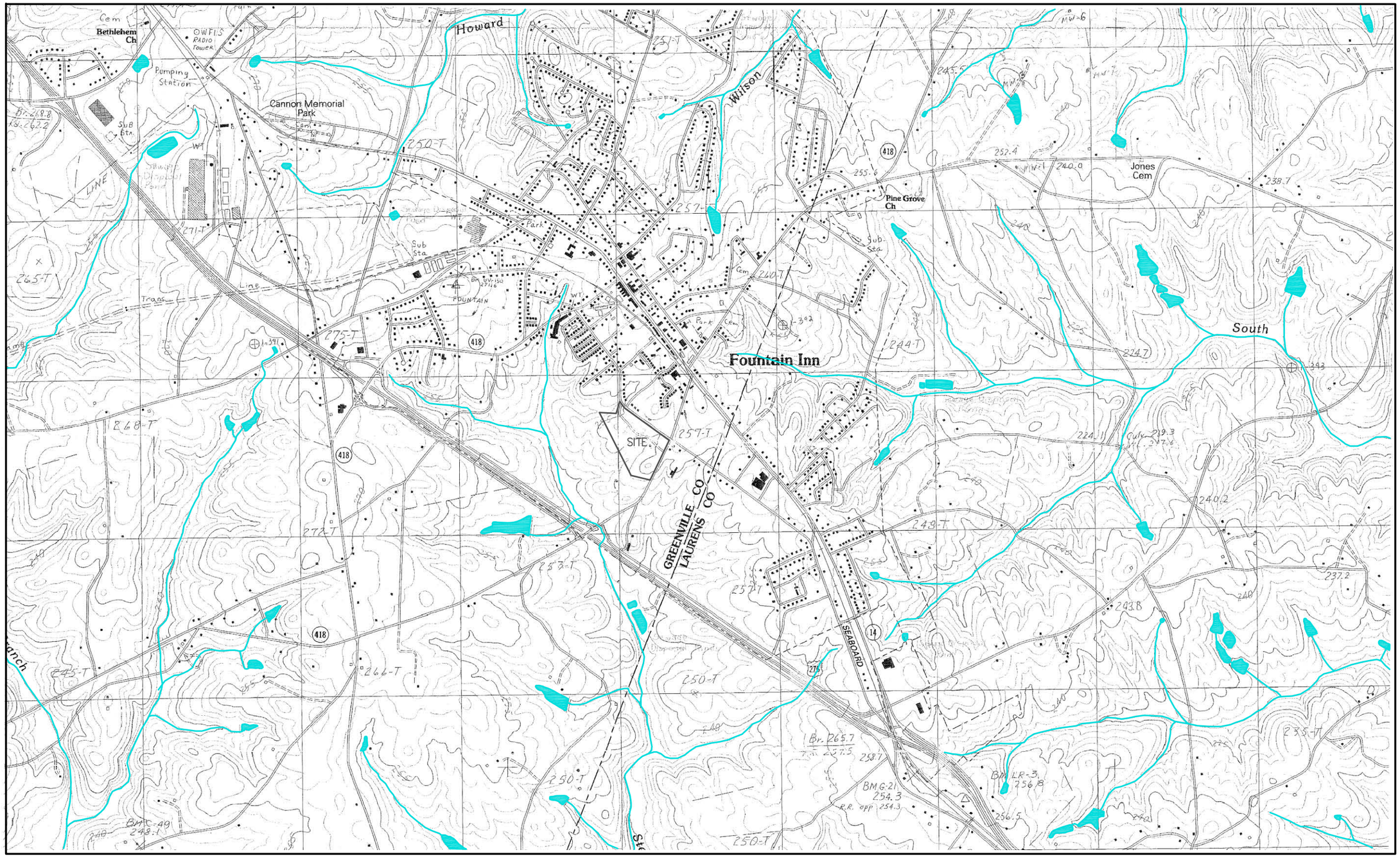

REFERENCE:
USGS 7.5-MINUTE SERIES TOPOGRAPHIC MAP,
FOUNTAIN INN, SC QUADRANGLE, PROV. ED. 1983.

2000 1000 0 2000 4000
APPROXIMATE SCALE IN FEET

DRAWN	CHB	DATE	5/14/12
CHECKED	GWW	FILE	FIGURE 6.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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Suite 202
GREENVILLE, S.C. 29607
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AREA SURFACE WATER MAP
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
6

RBTC FOUNTAIN INN DIVISION

FORMER SHERWIN WILLIAMS PROPERTIES

FORMER METALS BAGHOUSE (AOC #3)

HEAT TREAT CLEANING WATER DISPOSAL AREA (AOC #2)

FORMER EMPTY DRUM STORAGE PAD (AOC #5)

FORMER SCRAP METAL ROLLOFF (AOC #4)

FORMER HAZARDOUS WASTE ACCUMULATION BUILDING (AOC #9)

FORMER OIL/WATER SEPARATOR (AOC #8)

TANK CONTAINMENT AND UNDERGROUND PIPING AREA (AOC #1)

COMPOUNDING ROOM BLOWER EXHAUST (AOC #6)

STORMWATER OUTFALLS (AOC#7)

OF-2/2A

OF-1/1A

REFERENCES:

1. BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND MACTEC ENGINEERING & CONSULTING, INC. FIELD NOTES.

2. SAMPLE LOCATION MAP, SHERMAN WILLIAMS GRAPHIC ARTS DIVISION, FOUNTAIN INN, SOUTH CAROLINA, DRAWING NUMBER 63102-F2, SHEET 1, REVISION A, 2/25/02, PREPARED BY THE FLETCHER GROUP.

LEGEND

MW-1	APPROXIMATE PERMANENT MONITORING WELL LOCATION
TW-3	APPROXIMATE TEMPORARY MONITORING WELL LOCATION
TW-5	APPROXIMATE TEMPORARY MONITORING WELL LOCATION (INSTALLED BY OTHERS)
OWS-1	APPROXIMATE LOCATION OF SURFACE AND/OR SUBSURFACE SOIL SAMPLE
B-3	APPROXIMATE SOIL BORING LOCATION
GP-22	APPROXIMATE GEOPROBE SOIL OR GROUND-WATER SAMPLE LOCATION
GP-14D	APPROXIMATE DEEP GEOPROBE SOIL OR GROUND-WATER SAMPLE LOCATION
SS-13	APPROXIMATE SURFACE SOIL SAMPLE LOCATION
HA-4	APPROXIMATE HAND AUGER BORING LOCATION
SW-06	APPROXIMATE SURFACE WATER SAMPLE LOCATION

50 25 0 50 100
SCALE 1" = 50'

DRAWN	CHB	DATE	5/8/12
CHECKED	GWW	FILE	FIGURE 7.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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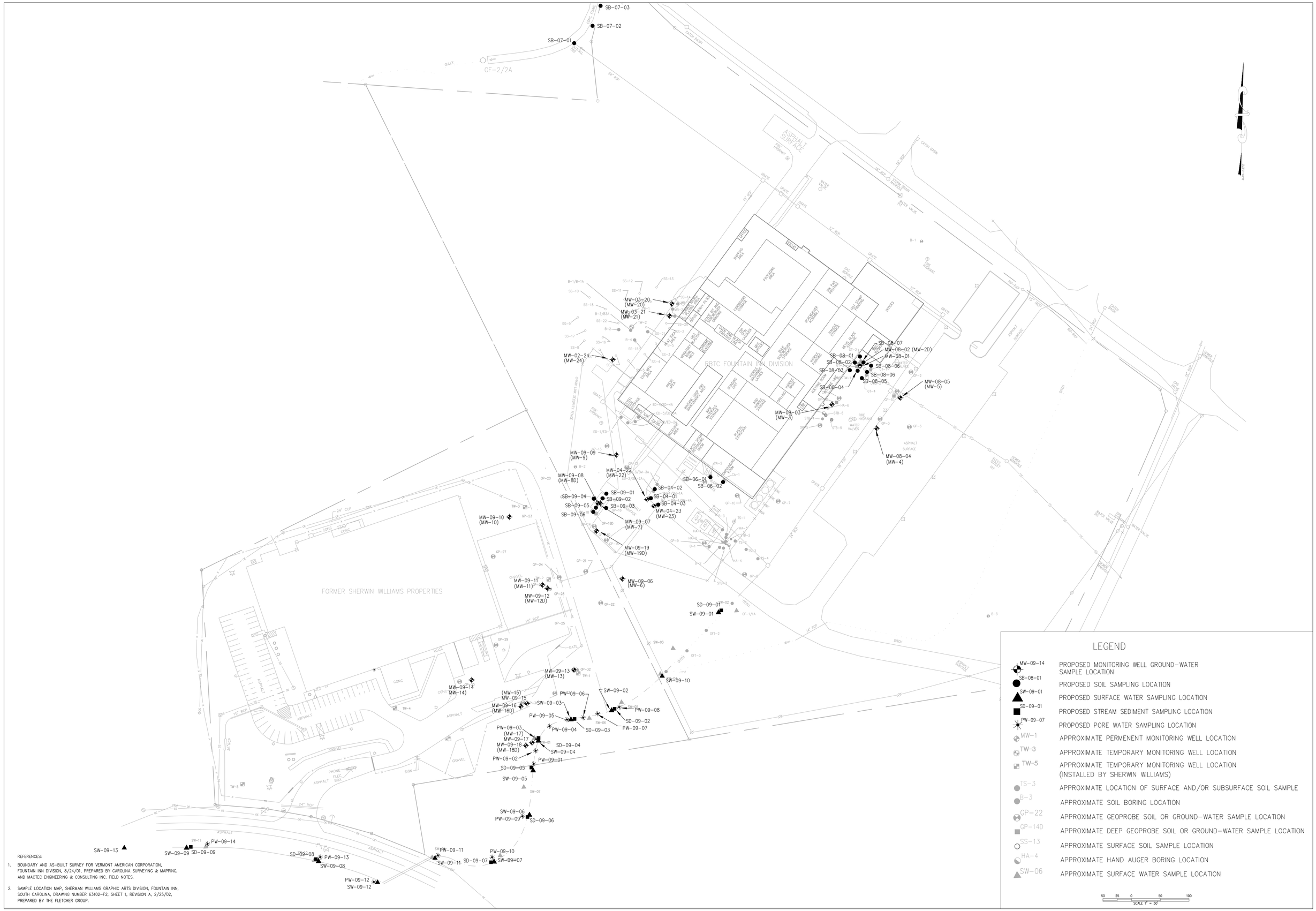
Fax: (864) 552-9699

PREVIOUS SOIL SAMPLING AND GROUND-WATER SAMPLING MAP
RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 7



REFERENCES:

1. BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND MACTEC ENGINEERING & CONSULTING INC. FIELD NOTES.
2. SAMPLE LOCATION MAP, SHERMAN WILLIAMS GRAPHIC ARTS DIVISION, FOUNTAIN INN, SOUTH CAROLINA, DRAWING NUMBER 63102-F2, SHEET 1, REVISION A, 2/25/02, PREPARED BY THE FLETCHER GROUP.

LEGEND

MW-09-14	PROPOSED MONITORING WELL GROUND-WATER SAMPLE LOCATION
SB-08-01	PROPOSED SOIL SAMPLING LOCATION
SW-09-01	PROPOSED SURFACE WATER SAMPLING LOCATION
SD-09-01	PROPOSED STREAM SEDIMENT SAMPLING LOCATION
PW-09-07	PROPOSED PORE WATER SAMPLING LOCATION
MW-1	APPROXIMATE PERMENENT MONITORING WELL LOCATION
TW-3	APPROXIMATE TEMPORARY MONITORING WELL LOCATION
TW-5	APPROXIMATE TEMPORARY MONITORING WELL LOCATION (INSTALLED BY SHERWIN WILLIAMS)
TS-3	APPROXIMATE LOCATION OF SURFACE AND/OR SUBSURFACE SOIL SAMPLE
B-3	APPROXIMATE SOIL BORING LOCATION
GP-22	APPROXIMATE GEOPROBE SOIL OR GROUND-WATER SAMPLE LOCATION
GP-14D	APPROXIMATE DEEP GEOPROBE SOIL OR GROUND-WATER SAMPLE LOCATION
SS-13	APPROXIMATE SURFACE SOIL SAMPLE LOCATION
HA-4	APPROXIMATE HAND AUGER BORING LOCATION
SW-06	APPROXIMATE SURFACE WATER SAMPLE LOCATION

50 25 0 50 100
SCALE 1" = 50'

DRAWN	CHB	DATE	5/8/12
CHECKED	GWW	FILE	FIGURE 7.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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Suite 202
GREENVILLE, S.C. 29607
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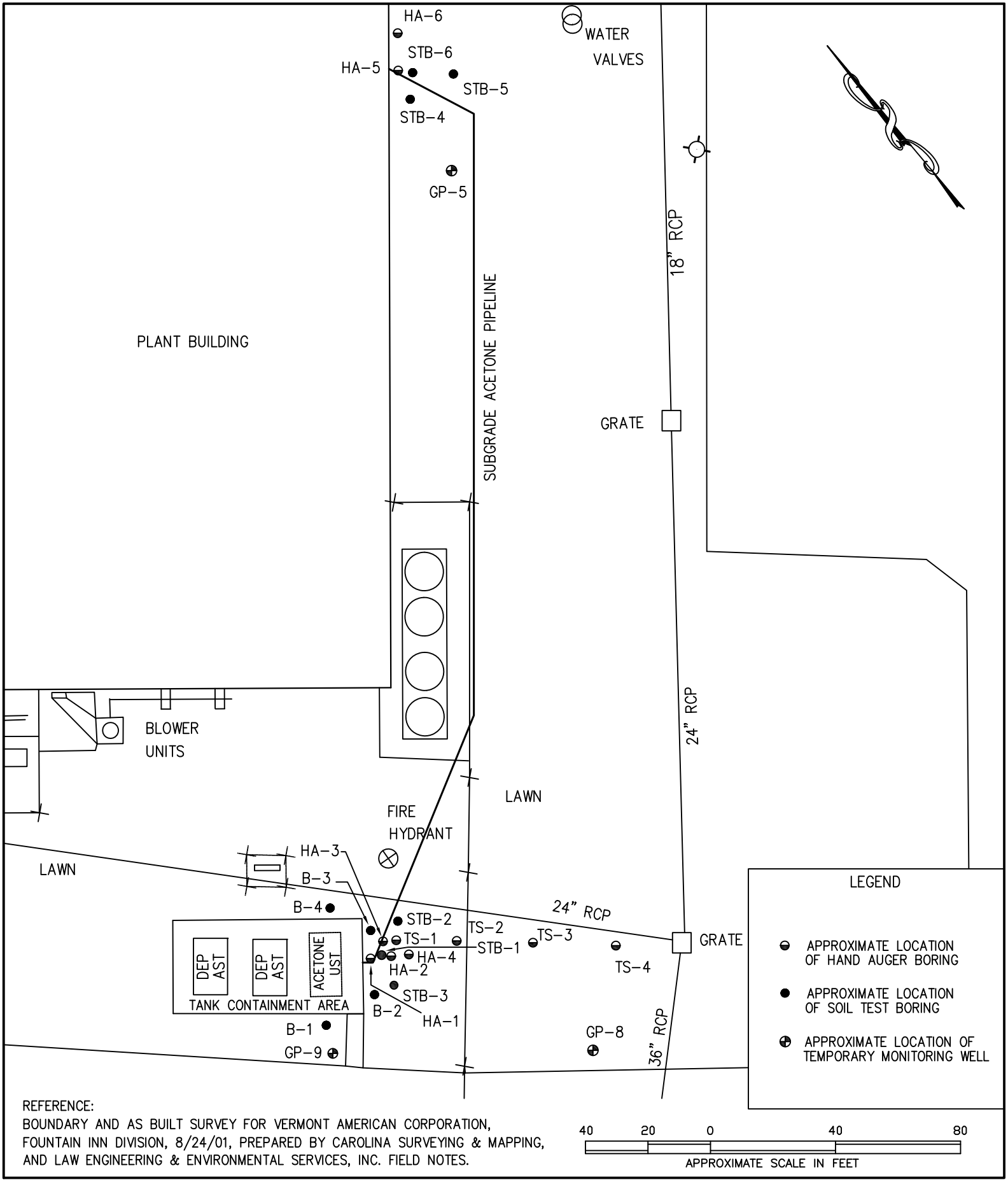
Fax: (864) 552-9699

PROPOSED SAMPLE LOCATION MAP

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 8



REFERENCE:
BOUNDARY AND AS BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND LAW ENGINEERING & ENVIRONMENTAL SERVICES, INC. FIELD NOTES.

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555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
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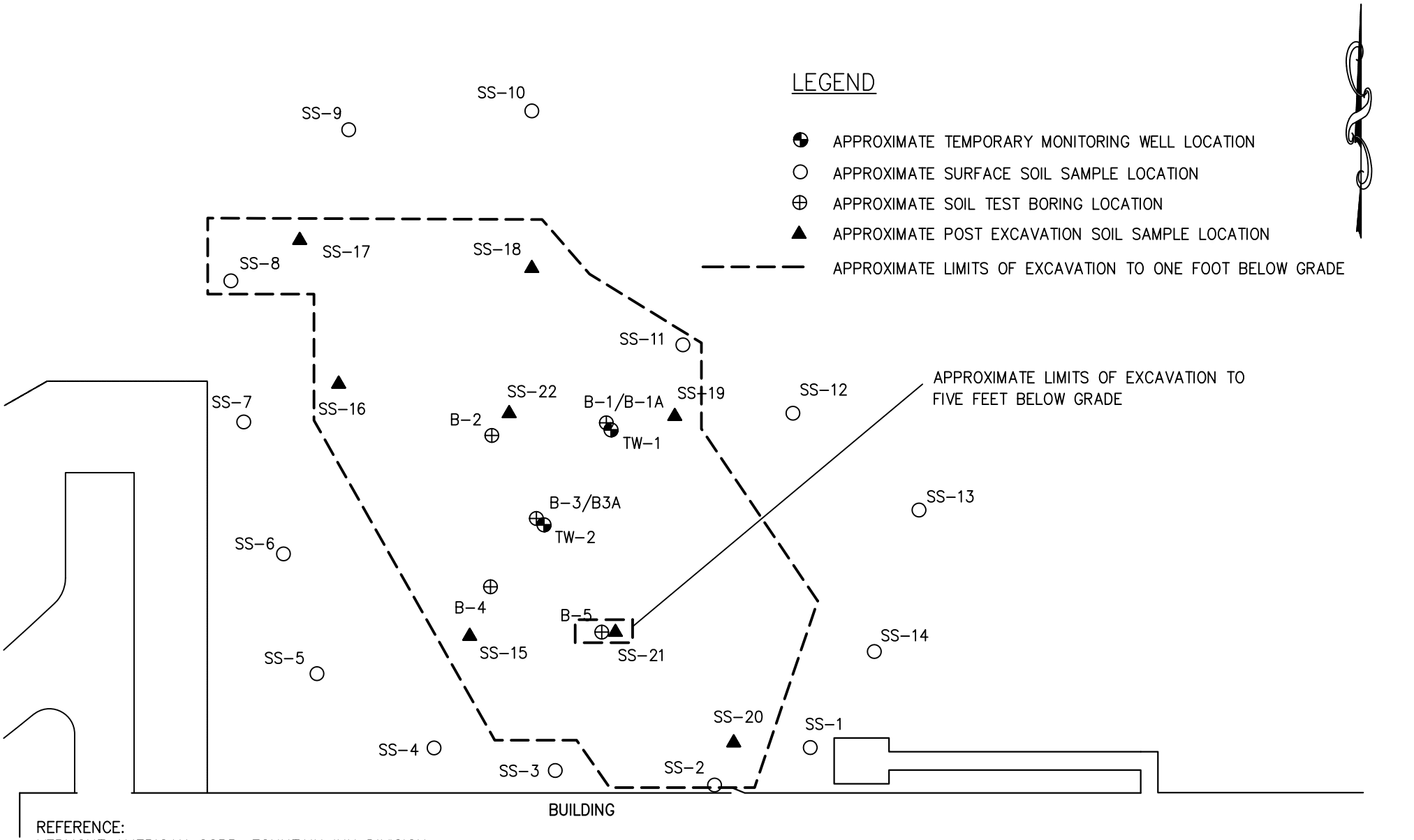
Fax: (864) 552-9699

TANK CONTAINMENT AREA SAMPLING LOCATION MAP
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 9

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 9.DWG	CHB	GWW	PSJ	5/14/12	6251121007.01.01



REFERENCE:
VERMONT AMERICAN CORP. FOUNTAIN INN DIVISION
PLANT LAYOUT, DATED NOVEMBER 30, 2000, AND
MACTEC ENGINEERING & CONSULTING, INC. FIELD NOTES.

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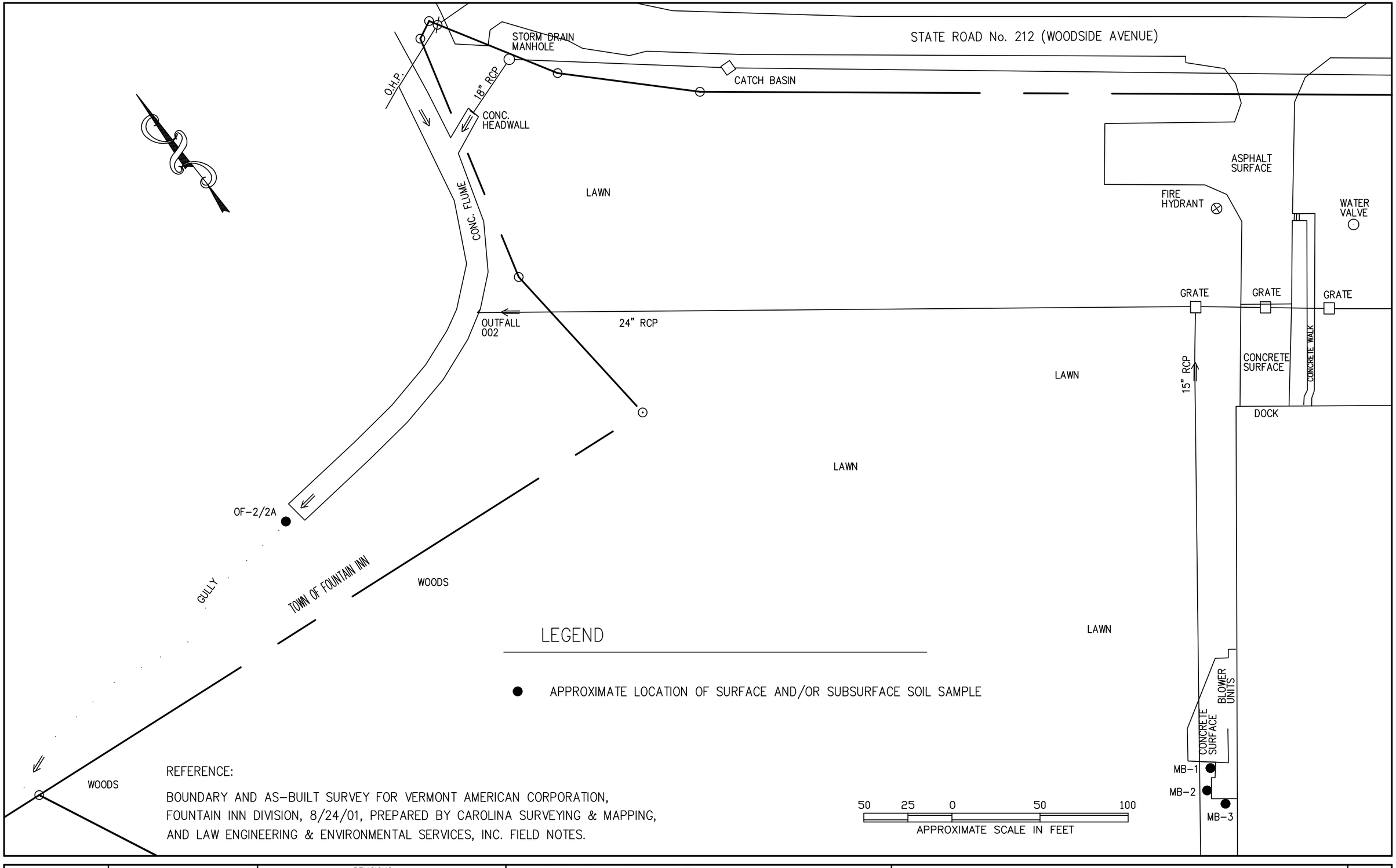
Fax: (864) 552-9699

HEAT TREATING CLEANING WATER DISPOSAL AREA
SAMPLING LOCATION PLAN
FORMER VERMONT AMERICAN FACILITY
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 10

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 10.DWG	CHB	GWW	PSJ	5/14/12	6251121007.01.01



REFERENCE:

BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND LAW ENGINEERING & ENVIRONMENTAL SERVICES, INC. FIELD NOTES.

DRAWN	CHB	DATE	5/14/12
CHECKED	GWW	FILE	FIGURE 11.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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Fax: (864) 552-9699

SAMPLE LOCATION PLAN – NORTHERN PORTION
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
11

TIN SHED
ASPHALT SURFACE
GRATE
CHAIN LINK SECURITY FENCE
TIN SHED
ED-4/EB-4A
ED-3/EB-3A
ED-2/EB-2A
ED-1/EB-1A
DOCK
CONCRETE SURFACE
POWER TRANS.
CA-2
CA-1
BLOWER UNITS
TANK
TANK
TANK
TANK
CONCRETE & GRAVEL SURFACE
24" RCP
LAWN
FIRE HYDRANT
WOODS
LAWN
CONCRETE WALK
SWITCH PANEL
FIRE HYDRANT
LAWN
DITCH
SM-3/SM-3A
SM-2/SM-2A
SM-4/SM-4A
TIN SHED
AST
AST
UST
TS-1
TS-2
TS-3
TS-4
24" RCP
GRATE
ASPHALT SURFACE
LAWN
36" RCP
SM-1/SM-1A
ASPHALT SURFACE
24" RCP
B-2
TIN SHED
ASPHALT SURFACE
BLOCK SHED
OUTFALL 001
OF-1/1A
CONCRETE SURFACE
LAWN
WOODS
OF1-2
DITCH
DITCH
OF1-3
OVERHEAD POWER
SHERWIN-WILLIAMS DEVELOPMENT CORPORATION
OF1-4
CAREY JUNE McGEE ROPER & ROBERT T. ROPER, JR.

LEGEND

- APPROXIMATE LOCATION OF PERMANENT MONITORING WELL
- APPROXIMATE LOCATION OF SURFACE SOIL SAMPLE

REFERENCE:

BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND LAW ENGINEERING & ENVIRONMENTAL SERVICES, INC. FIELD NOTES.

50 25 0 50 100
APPROXIMATE SCALE IN FEET

DRAWN	CHB	DATE	5/14/12
CHECKED	GWW	FILE	FIGURE 12.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

REVISIONS		
No.	DESCRIPTION	BY

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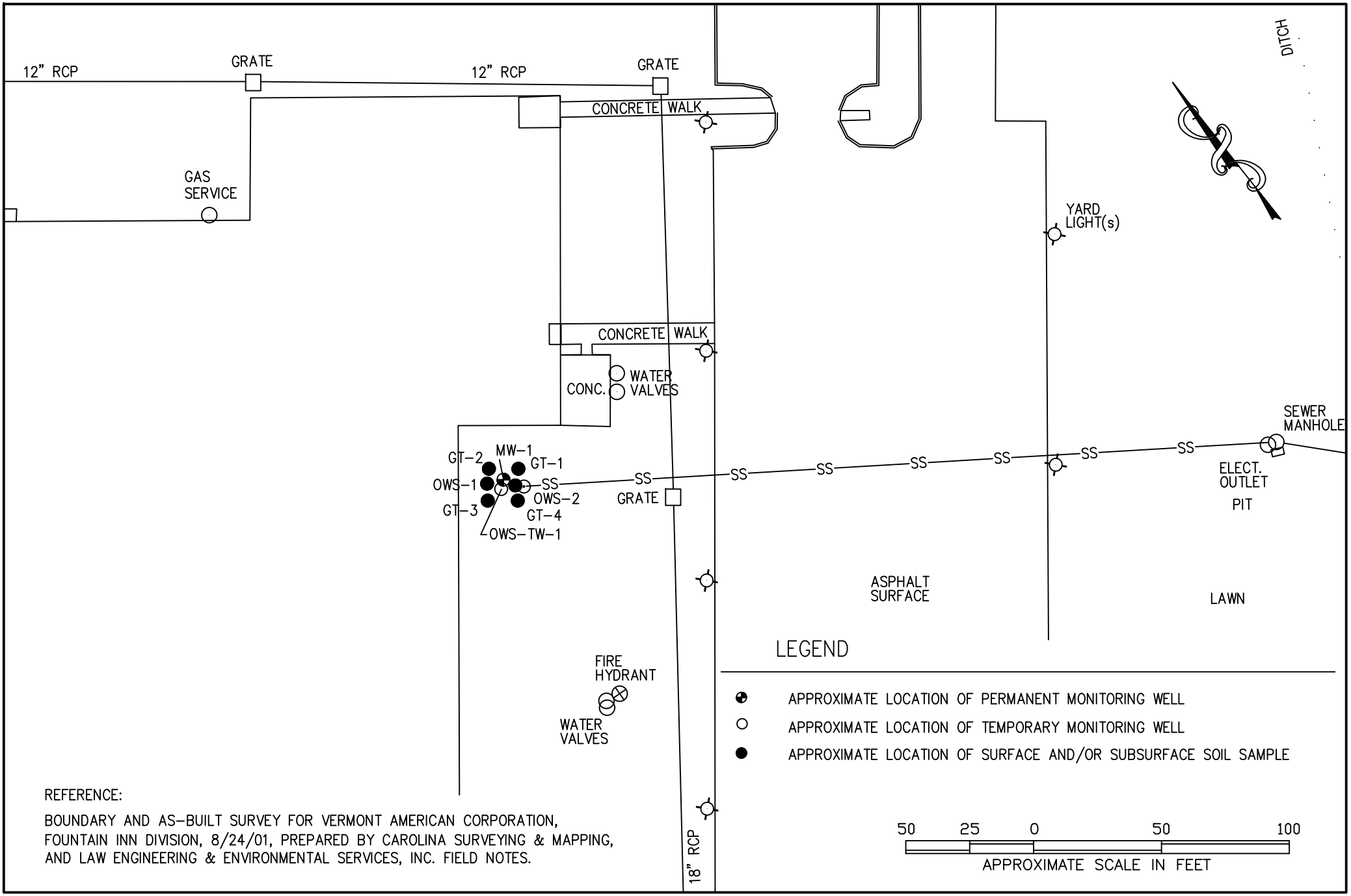
Fax: (864) 552-9699

SAMPLE LOCATION PLAN – WESTERN PORTION
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
12



REFERENCE:

BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND LAW ENGINEERING & ENVIRONMENTAL SERVICES, INC. FIELD NOTES.

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GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

SAMPLE LOCATION PLAN – SOUTHERN PORTION
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 13

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 13.DWG	CHB	GWW	PSJ	5/14/12	6251121007.01.01

LEGEND

AREA OF CONCERN (AOC # 1)

200 100 0 200 400

SCALE 1" = 200'

DRAWN	CHB	DATE	5/14/12	REVISIONS No.	DESCRIPTION	BY
CHECKED	GWW	FILE	FIGURE 14.DWG			
APPROVED	PSJ	JOB NO:	6251121007.01.01			

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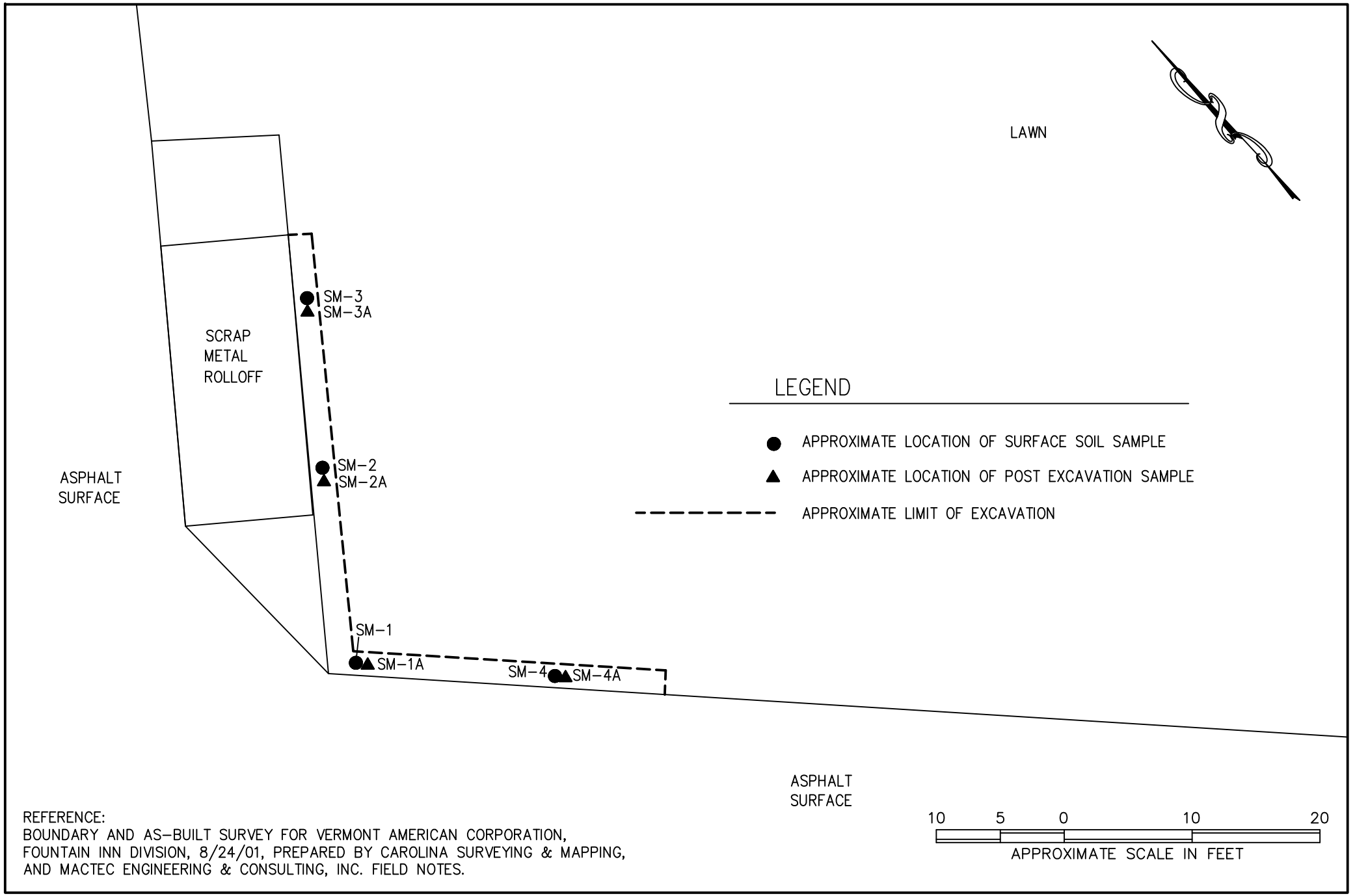
Fax: (864) 552-9699

AREAS OF CONCERN
RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 14



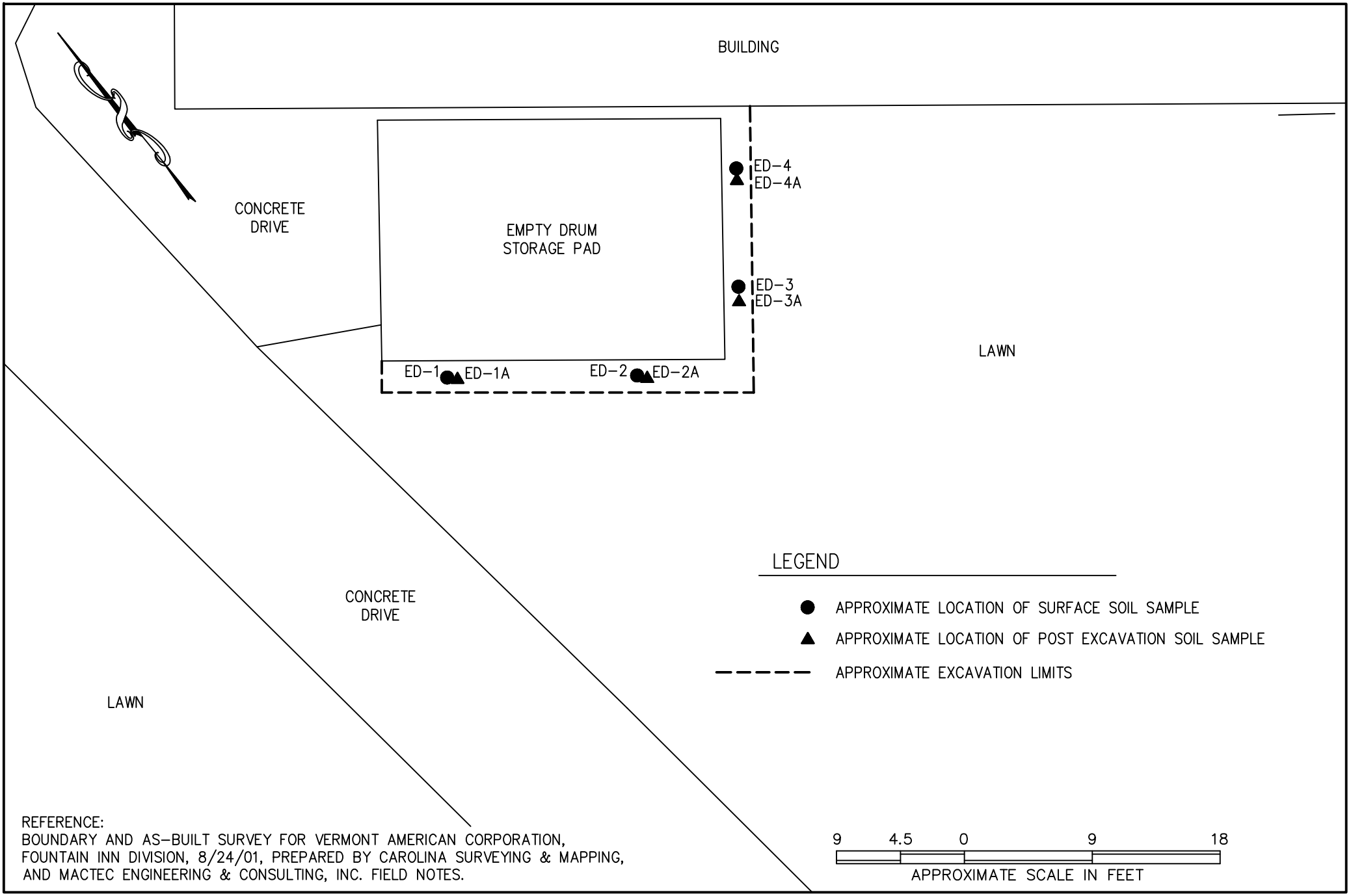
REFERENCE:
BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND MACTEC ENGINEERING & CONSULTING, INC. FIELD NOTES.

amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

SCRAP METAL ROLLOFF AREA
FORMER VERMONT AMERICAN FACILITY
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 15

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 15.DWG	CHB	GWW	PSJ	5/14/12	6251121007.01.01



REFERENCE:
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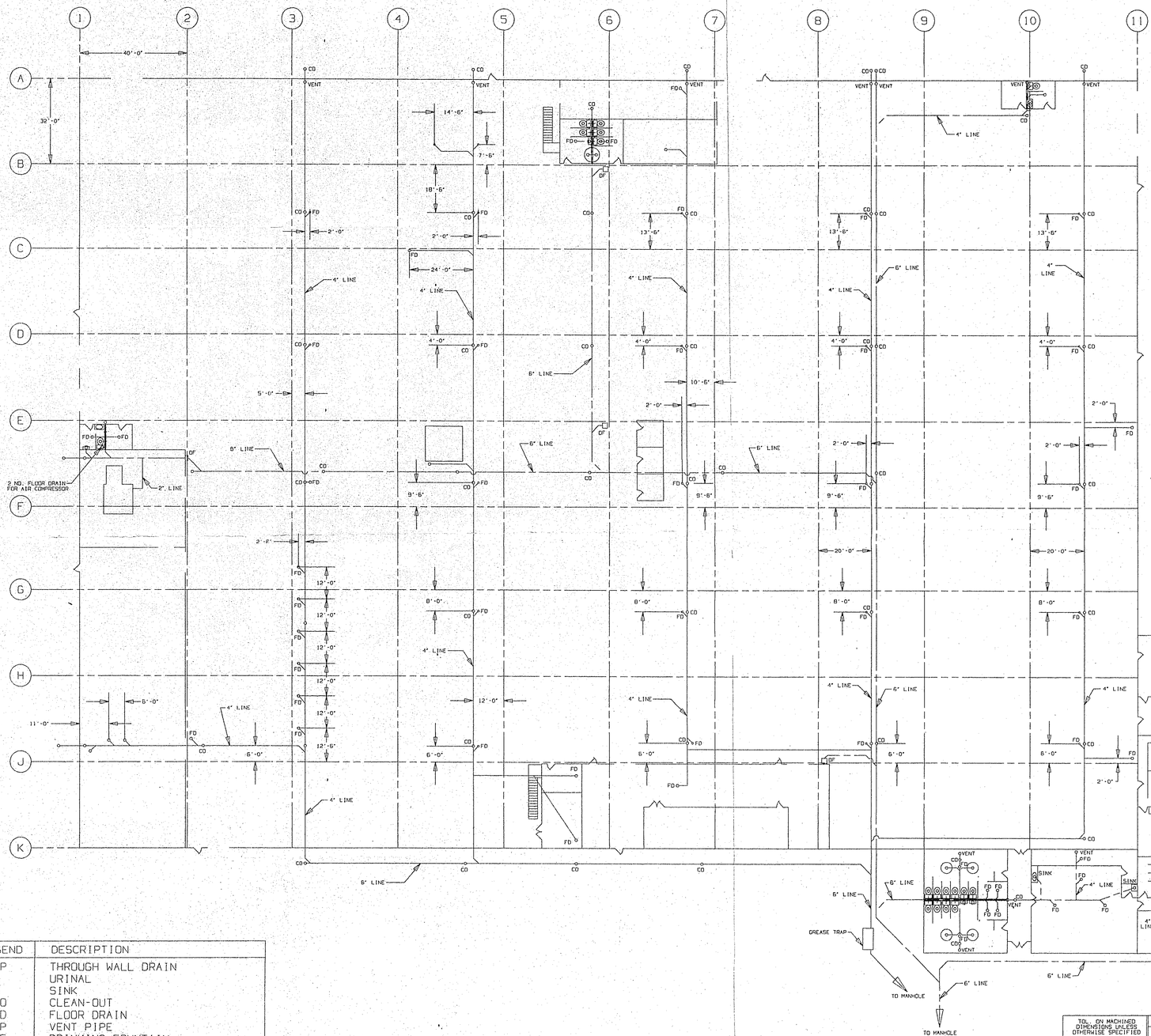
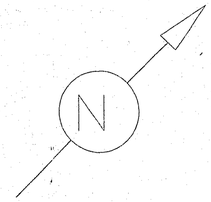
EMPTY DRUM STORAGE AREA
FORMER VERMONT AMERICAN FACILITY
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 16

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 16.DWG	CHB	GWW	PSJ	5/14/12	6251121007.01.01





NO. REQ'D.	LEGEND	DESCRIPTION
2	SP	THROUGH WALL DRAIN
4	U	URINAL
3	S	SINK
61	CO	CLEAN-OUT
56	FD	FLOOR DRAIN
17	VP	VENT PIPE
5	DF	DRINKING FOUNTAIN
2	HDF	HANDICAPPED DRINKING FOUNTAIN
16	WC	WATER CLOSET
6	HWC	HANDICAPPED WATER CLOSET
10	L	LAVITORY
4	BWB	BRADLEY WASH BASIN

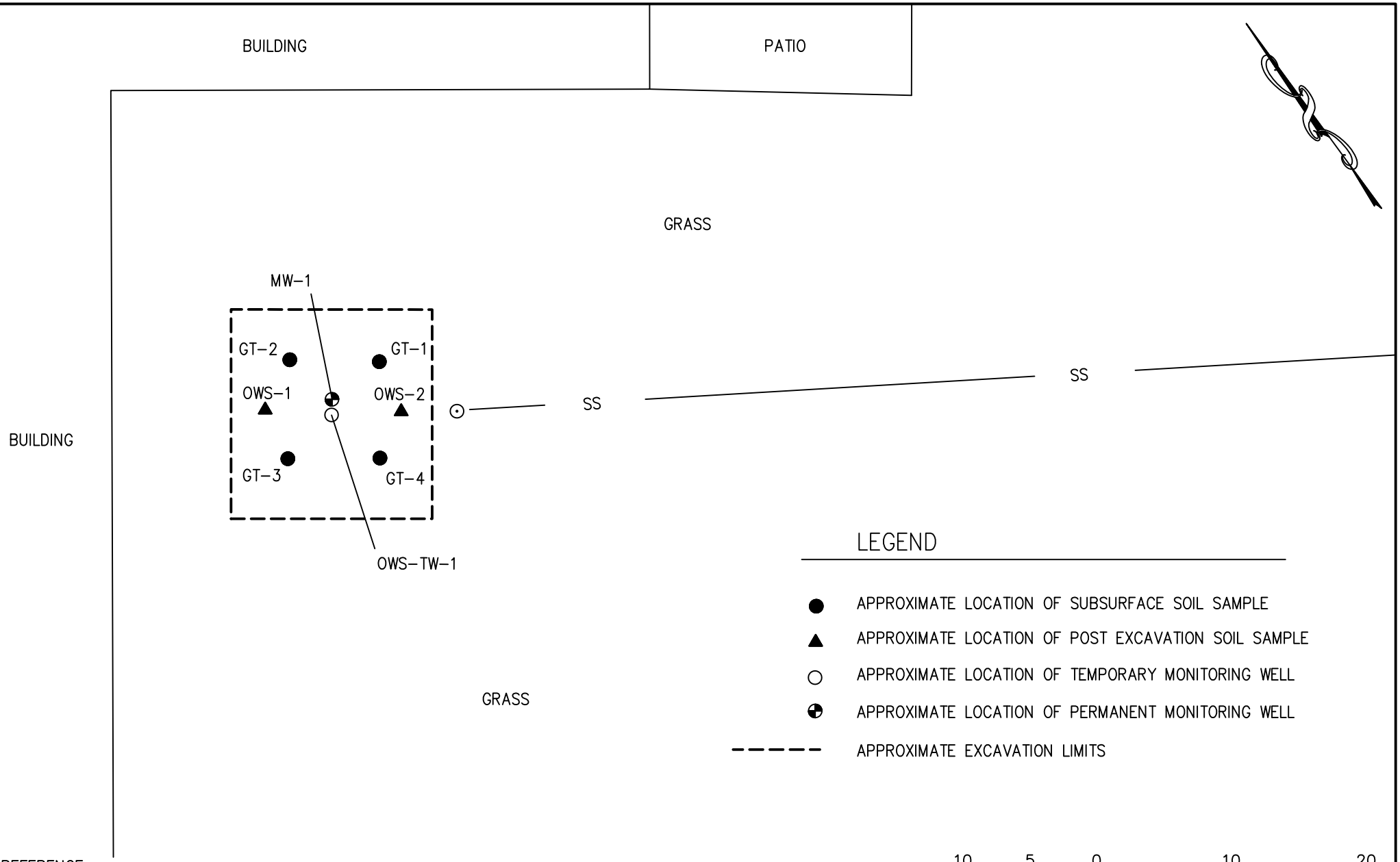
FIGURE 17

TOL. ON MACHINED DIMENSIONS UNLESS OTHERWISE SPECIFIED		
DECIMAL	.0	±.1
	.00	±.010
	.000	±.003
	.0000	±.0003
ANGLES		±.50°
BREAK SHARP EDGES		
REFERENCE DWG.		

NO.	REVISIONS	BY	DATE

VERMONT AMERICAN CORP.
FOUNTAIN INN DIVISION
FOUNTAIN INN, S.C.

SCALE: 1/16" = 1'-0"	DATE: 8-30-90	UNIT
DRAWN: JAGODZINSKI	CHECK:	
TITLE: SANITARY & PROCESS SEWER LINES		DWG. NO. 43001
		SHEET 11 OF 12



REFERENCE:
BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND MACTEC ENGINEERING & CONSULTING, INC. FIELD NOTES.

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GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

OIL WATER SEPARATOR AREA
FORMER VERMONT AMERICAN FACILITY
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 18

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 18.DWG	CHB	GWW	PSJ	5/14/12	6251121007.01.01

APPENDICES

APPENDIX A

DOCUMENTATION

LEGEND

TS-1 ○	SURFACE SOIL SAMPLING LOCATION
STB-1 ●	SUBSURFACE SOIL SAMPLING LOCATION
HA-1 ◒	SUBSURFACE SOIL SAMPLING LOCATION (HAND AUGER)
0.35	ACETONE CONCENTRATION IN MILLIGRAMS PER KILOGRAM

50 25 0 50 100

SCALE 1" = 100'

DRAWN	CHB	DATE	5/25/12
CHECKED	GWW	FILE	FIGURE A2-A4.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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ACETONE CONCENTRATIONS IN SURFACE SOIL SAMPLES

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A1

LEGEND

TS-1 ○	SURFACE SOIL SAMPLING LOCATION
STB-1 ●	SUBSURFACE SOIL SAMPLING LOCATION
HA-1 ◒	SUBSURFACE SOIL SAMPLING LOCATION (HAND AUGER)
204	ACETONE CONCENTRATION IN MILLIGRAMS PER KILOGRAM

50 25 0 50 100

SCALE 1" = 100'

DRAWN	CHB	DATE	5/25/12
CHECKED	GWW	FILE	FIGURE A2-A4.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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Fax: (864) 552-9699

ACETONE CONCENTRATIONS IN SUBSURFACE SOIL SAMPLES (1-5')

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A2

LEGEND

TS-1 ○	SURFACE SOIL SAMPLING LOCATION
STB-1 ●	SUBSURFACE SOIL SAMPLING LOCATION
HA-1 ◒	SUBSURFACE SOIL SAMPLING LOCATION (HAND AUGER)
5.25	ACETONE CONCENTRATION IN MILLIGRAMS PER KILOGRAM

50 25 0 50 100

SCALE 1" = 100'

DRAWN	CHB	DATE	5/25/12
CHECKED	GWW	FILE	FIGURE A2-A4.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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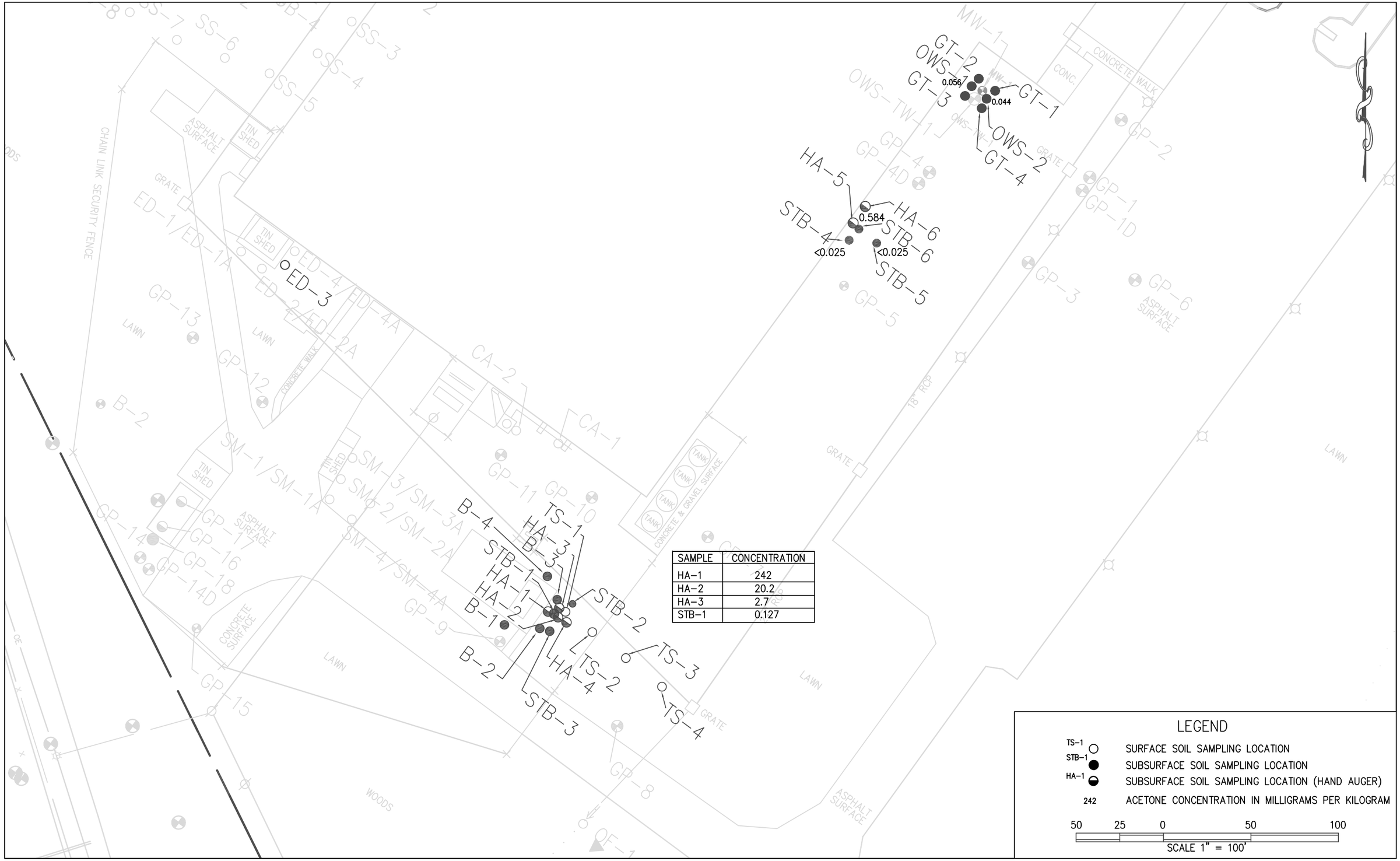
Fax: (864) 552-9699

ACETONE CONCENTRATIONS IN SUBSURFACE SOIL SAMPLES (6-11')

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A3



SAMPLE	CONCENTRATION
HA-1	242
HA-2	20.2
HA-3	2.7
STB-1	0.127

LEGEND

- TS-1 ○ SURFACE SOIL SAMPLING LOCATION
- STB-1 ● SUBSURFACE SOIL SAMPLING LOCATION
- HA-1 ◒ SUBSURFACE SOIL SAMPLING LOCATION (HAND AUGER)
- 242 ACETONE CONCENTRATION IN MILLIGRAMS PER KILOGRAM

50 25 0 50 100

SCALE 1" = 100'

DRAWN	CHB	DATE	5/25/12
CHECKED	GWW	FILE	FIGURE A2-A4.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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GREENVILLE, S.C. 29607
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Fax: (864) 552-9699

ACETONE CONCENTRATIONS IN SUBSURFACE SOIL SAMPLES (12-20')

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A4

RBTC FOUNTAIN INN DIVISION

FORMER SHERWIN WILLIAMS PROPERTIES

OF-2 11
OF-1 19
MB-1 68
MB-2 180
MB-3 170
SM-3 36
SM-2 32
SM-1 140
SM-4 21
B-1
B-2
B-3/B3A
B-4
B-5
TW-1
TW-2
SS-1 SS-2 SS-3 SS-4 SS-5 SS-6 SS-7 SS-8 SS-9 SS-10 SS-11 SS-12 SS-13 SS-14
GT-1 GT-2 GT-3 GT-4
OWS-1 OWS-2 OWS-TW-1
MW-1
GP-1 GP-2 GP-3 GP-4 GP-5 GP-6 GP-7 GP-8 GP-9 GP-10 GP-11 GP-12 GP-13 GP-14 GP-15 GP-16 GP-17 GP-18
GP-1D GP-4D GP-14D
HA-1 HA-2 HA-3 HA-4 HA-5 HA-6
STB-1 STB-2 STB-3 STB-4 STB-5 STB-6
TS-1 TS-2 TS-3 TS-4
CA-1 CA-2
ED-1/ED-1A ED-2/ED-2A ED-3/ED-3A ED-4/ED-4A
WOODS
LAWN
GRAVEL
ASPHALT SURFACE
CONCRETE SURFACE
CONCRETE WALK
CONCRETE CURB
GRATE
CATCH BASIN
STORM DRAIN MANHOLE
GAS SERVICE
DOCK
TIMBER
24" RCP
18" RCP
15" RCP
12" RCP
24" CCP
CONC
GULLY
SHED
CHAIN LINK SECURITY FENCE

LEGEND

MB-1 ● SURFACE SOIL SAMPLE LOCATION

140 CHROMIUM CONCENTRATION IN MILLIGRAMS PER KILOGRAM

100 50 0 100 200

SCALE 1" = 100'

DRAWN	CHB	DATE	5/25/12
CHECKED	GWW	FILE	FIGURE A5-A7.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

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Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

CHROMIUM CONCENTRATIONS IN SURFACE SOIL SAMPLES

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE A5

LEGEND

MB-1 ●	SURFACE SOIL SAMPLE LOCATION
140	CHROMIUM CONCENTRATION IN MILLIGRAMS PER KILOGRAM
NA	NOT ANALYZED

100 50 0 100 200

SCALE 1" = 100'

DRAWN	CHB	DATE	5/25/12
CHECKED	GWW	FILE	FIGURE A5-A7.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

REVISIONS		
No.	DESCRIPTION	BY

amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

CHROMIUM CONCENTRATIONS IN SUBSURFACE SOIL SAMPLES (1 - 1.5')

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A6

DRAWN	CHB	DATE	5/25/12
CHECKED	GWW	FILE	FIGURE A5-A7.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

REVISIONS		
No.	DESCRIPTION	BY

amec

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GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

NICKEL CONCENTRATIONS IN SURFACE SOIL SAMPLES

RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A7

D H E C
PROMOTE PROTECT PROSPER
2600 Bull Street
Columbia, SC 29201-1708

BUREAU OF
UNDERGROUND STORAGE TANK MANAGEMENT

Phone (803) 734-0723 Fax (803) 734-3604

Mr. John Young
Vermont American Corporation
National City Tower, Suite 2300
101 South Fifth Street
Louisville, Kentucky 40202

JUN 2 4 1997

Re: Vermont American Corporation
Site ID # 04235
Site Specific Action Levels for Acetone received March 3, 1997
Greenville County

Dear Mr. Young:

The Bureau of Underground Storage Tank Management of the South Carolina Department of Health and Environmental Control (SCDHEC) has reviewed the referenced report. Based on the data obtained from the monitoring reports, the release does not present a significant threat to human health or the environment. Therefore, this office recommends that no further action will be required at this time. **The no further action decision is conditional** and is based on the following assumptions and conditions and is valid under these assumptions and conditions:

Assumptions:

1) The property on which the release occurred and the adjacent properties are currently zoned commercial and will remain commercial.

2) Groundwater is not currently being used within the actual or predicted extent of the plume and will not be used as a source of water in the reasonably anticipated future.

3) The groundwater monitoring program has verified the continued natural degradation of the COC.

Conditions:

1) The SSTLs have been met for all the COC in soil and groundwater at the source and the monitoring verified that the COC will not exceed RBSLs at all potential receptors and/or exposure points.

RECEIVED
JUN 26 1997

2) The groundwater should not be used as a source of potable or nonpotable water.

3) Land use or zoning ordinance should remain the same (i.e., commercial or residential).

4) If, in the future, the COC are detected at levels that present a risk to human health or the environment, and COCs can be attributed to the release from the UST system, this office may require site rehabilitation activities per South Carolina Underground Storage Tank Control Regulations (SCUSTR) R.61-92 Part 280.51/280.52.

5) This correspondence does not exempt the UST system from any additional requirements (i.e., notification, operation, release detection, corrective action, and closure) of subparts B, C, D, E, F, and G of the SCUSTCR.

This letter is a notice of "Conditional No Further Action". The assumptions and conditions must remain true for the referenced site until COC levels are below the MCLs. Should the above assumptions and conditions change the Bureau of Underground Storage Tank Management must be notified. The decision for the Conditional No Further Action will be reevaluated and further site assessment and rehabilitation activities may be required.

By agreeing to the "Conditional No Further Action", the referenced site will be placed on a registry of releases that have been closed with concentrations of COC's above RBSLs but do not pose any significant threat to human health or the environment. The following options are offered at this time:

Option 1:

You may choose to abandon all the monitoring wells at this time. Abandonment of the well must be in accordance with South Carolina Well Standards and Regulations (R. 61-71). If you choose to remove the site from the registry, verification that all the COC are below the RBSLs, by analytical samples from temporary wells will be required.

Option 2:

You may choose to keep all the wells for future monitoring in order to verify that the intrinsic remediation process has been successful in reducing all concentrations below RBSLs. Analytical results will be required for verification and the removal of the release from the registry. It will be your responsibility for the maintenance, monitoring and abandonment of these wells. It will also be your responsibility to ensure that any possibility of leakage of potential future spills into the groundwater aquifer is minimized.

On all correspondence related to this site, please reference the Site ID number given above. Should you have any questions, please feel free to contact Chuck Williams at (803) 734-5455.

Sincerely,

State Lead and Field Services Section
Assessment and Corrective Action Division
Bureau of Underground Storage Tank Management

Charles J. Williams III, Hydrogeologist

Christopher S. Doll, P.G., Manager

enc: Option Letter for Abandonment

cc: Paul S. Johnstone, Law Environmental, (w/enclosures)
Technical File

DHEC/UST/ACAD/SLS/CJW/062497

D H E C
PROMOTE PROTECT PROSPER
2600 Bull Street
Columbia, SC 29201-1708

March 22, 2002

Mr. John Young
Vermont American Corp.
National City Tower, Suite 2300
101 S. 5th Street
Louisville KY 40202

Re: Vermont American Corp, Site ID #01817
Assessment Report received March 22, 2002
Greenville County

Dear Mr. Young:

The Department has reviewed the referenced assessment report. Based upon the groundwater data in the referenced report, the geotechnical data is below EPA Maximum Contaminant Levels (MCLs).

As this data was not specifically requested by the Department, and the work conducted at this site received no prior review by the Department, we cannot provide any comments on the completeness of the work performed or the overall environmental conditions of the site. Based on the information and analytical data submitted, there is no evidence to indicate that a violation of the Pollution Control Act has occurred. Consequently, no investigation will be required at this time. Please note, this statement pertains only to the data submitted and does not apply to other areas of the site and/or any other potential regulatory violations. Further, the Department retains the right to request further investigation if deemed necessary.

On all correspondence regarding this site, please reference Site ID #01817. If you have any questions, please call me at (803) 898-4155 or email boyntosj@columb32.dhec.state.sc.us.

Sincerely,

Jennifer Boynton, Hydrogeologist
Groundwater Quality Section
Water Monitoring, Assessment, and Protection Division
Bureau of Water

B. Thomas Knight, P.G., Manager

cc: Appalachia II District EQC
Mr. Paul Johnstone, Law Eng., 4 Interchange Blvd, Greenville SC 29607

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN

QUALITY ASSURANCE PROJECT PLAN
REVISION 5.0

FORMER VERMONT BOSCH SITE
FOUNTAIN INN, SOUTH CAROLINA

Prepared for:

ROBERT BOSCH TOOL CORPORATION

1800 West Central Road
Mount Prospect, Illinois 60056

Prepared by:

Paul S. Johnstone and W. Paul Brafford
AMEC Environment & Infrastructure, Inc.
555 North Pleasantburg Drive, Suite 202
Greenville, South Carolina 29607

AMEC Project 6251121007.01.01

August 24, 2012

A PROJECT MANAGEMENT

A1 TITLE

QUALITY ASSURANCE PROJECT PLAN
REVISION 5.0

FORMER VERMONT BOSCH SITE
FOUNTAIN INN, SOUTH CAROLINA

Prepared for:

ROBERT BOSCH TOOL CORPORATION
1800 West Central Road
Mount Prospect, Illinois 60056

Prepared by:

Paul S. Johnstone and W. Paul Brafford
AMEC Environment & Infrastructure, Inc.
555 North Pleasantburg Drive, Suite 202
Greenville, South Carolina 29607
(864) 552-9624

August 24, 2012

AMEC Project 6251121007.01.01

A1 APPROVAL SHEET

AMEC Project Manager:

Paul S. Johnstone
Signature

Paul S. Johnstone / 10/18/12
Printed Name/Date

AMEC Project QA Officer:

William Paul Brafford
Signature

William Paul Brafford / 10/18/12
Printed Name/Date

AES Project QA Officer

Signature

Printed Name/Date

AES Laboratory Director

Signature

Printed Name/Date

SCDHEC Project Manager:

Signature

Printed Name/Date

SCDHEC Project QA Officer:

Signature

Printed Name/Date

A1 APPROVAL SHEET

AMEC Project Manager:

_____
Signature

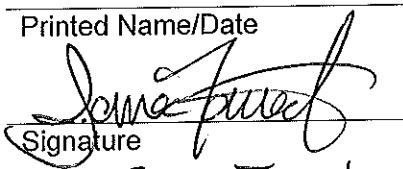
_____
Printed Name/Date

AMEC Project QA Officer:

_____
Signature

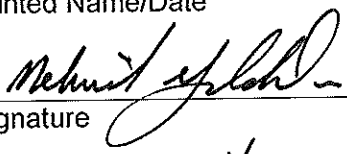
_____
Printed Name/Date

AES Project QA Officer


Signature

James Forrest 10/18/12
Printed Name/Date

AES Laboratory Director


Signature

MEHMET YILDIRIM 10/18/12
Printed Name/Date

SCDHEC Project Manager:

_____
Signature

_____
Printed Name/Date

SCDHEC Project QA Officer:

_____
Signature

_____
Printed Name/Date

A2 TABLE OF CONTENTS

LIST OF TABLES

Table

LIST OF FIGURES

Figure

LIST OF APPENDICES

Appendix

ABBREVIATIONS AND ACRONYMS

Acronym	Definition
AALA	American Association for Laboratory Accreditation
AA	Atomic Absorption
AES	Atomic Emission Spectroscopy
AOC	Area of Concern
BERA	Baseline Ecological Risk Assessment
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Register
CHMM	Certified Hazardous Materials Manager
COC	Chain of Custody
CPR	Cardio Pulmonary Resuscitation
CVAF	Cold Vapor Atomic Fluorescence
DO	dissolved oxygen
DQO	Data Quality Objectives
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
ERA	Ecological Risk Assessment
FDRs	Field Data Records
FID	flame-ionization detector
FOL	Field Operations Leader
FSAP	Field Sampling and Analysis Plan
GC	Gas Chromatograph
GPC	Gel Permeation Chromatography
GPS	global positioning system
HASP	Health and Safety Plan
HHRA	Human Health Risk Assessment
HSA	hollow stem augers
ICP	Inductively Coupled Plasma
ID	inside diameter
IDW	investigative derived waste
LCS	laboratory control sample
LD	laboratory duplicate
MACTEC	MACTEC Engineering and Consulting, Inc.
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mm	millimeter

LIST OF ABBREVIATIONS AND ACRONYMS - Continued

Acronym	Definition
mL	milliliter
MS	Mass Spectroscopy
MS/MSD	Matrix Spike and Matrix Spike Duplicate
MQO	Measurement Quality Objectives
NAVD88	North American Vertical Datum, 1988
NELAP	National Environmental Laboratory Accreditation Program
NCP	National Contingency Plan
NIST	National Institute of Standard Testing
NPL	National Priority List
OD	outside diameter
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Act
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PE	Professional Engineer
PG	Professional Geologist
PID	photo-ionization detector
PM	Project Manager
POC	Point of Contact
PQL	Practical Quantitation Limits
PVC	polyvinyl chloride
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCSR	Quality Control Summary Report
RCRA	Resource Conservation and Recovery Act
RBTC	Robert Bosch Tool Corporation
RI/FS	Remedial Investigation and Feasibility Study
RL	Reporting Limit
RPD	relative percent difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SCDHEC	South Carolina Department of Health and Environmental Control
SDG	Sample Delivery Group
SHSO	Site Health and Safety Officer
SOP	Standard Operating Procedure
STP	Sample Tracking Program
SVOC	semi-volatile organic compound

LIST OF ABBREVIATIONS AND ACRONYMS - Continued

Acronym	Definition
TAL	Target Analyte List
TCL	Target Compound List
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound
WTP	Work and Test Procedure

A3 DISTRIBUTION LIST

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Robert Bosch, LLC	Mr. John Young 401 North Bendix Drive South Bend, IN 46628	1 CD with PDF
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Robert Bosch Tool Corp.	Mr. David Luepke David.Luepke@us.bosch.com	
Robert Bosch, LLC	Mr. John Young John.Young@us.bosch.com	

A4 PROJECT/TASK ORGANIZATION

Robert Bosch Tool Corporation (RBTC) has contracted with AMEC Environment & Infrastructure, Inc. (AMEC), formerly MACTEC Engineering and Consulting, Inc. (MACTEC), to perform environmental investigations and remediation of contaminated media at the Former Vermont Bosch site (Site) located in Greenville County, South Carolina. The primary goal of the environmental investigation and remediation is to protect human health and the environment by determining the horizontal and vertical extent of contaminated media and the reduction and/or removal of contaminated media resulting from former facility operations. This Quality Assurance Project Plan (QAPP) was developed to outline the procedures and methodologies that will be used to assure the quality of the sampling and analytical data collected at the Site. Sampling and analysis activities at the site include but are not limited to:

- A water supply well inventory within a one-mile radius of the site.
- Surface soil samples collected at Area of Concern (AOC) #7 (Storm Water Outfalls).
- Subsurface soil samples collected at AOC #4 (Former Scrap Metal Rolloff), AOC #6 (Compounding Room Blower Exhaust), AOC #8 (Former Oil/Water Separator), and AOC #9 (Former Hazardous Waste Accumulation Building).
- Groundwater samples collected from permanent monitoring wells installed at AOC #3 (Former Metals Baghouse), AOC #4 (Former Scrap Metal Rolloff), AOC #8 (Former Oil/Water Separator), Pore-water samples collected from the bank of the unnamed tributary to Stoddard Creek.
- Surface water and sediment samples collected from the unnamed tributary to Stoddard Creek.
- Hydraulic conductivity (slug) testing in selected monitoring wells.

This QAPP has been prepared in general accordance with the South Carolina Department of Health and Environmental Control (SCDHEC) "*Guidance Document For Preparing Quality Assurance Project Plans (QAPPs) For Environmental Monitoring Projects/Studies*" (SCDHEC, 2008). Additional references from the United States Environmental Protection Agency (USEPA) used in preparation of this QAPP include "*Requirements for Quality Assurance Project Plans for Environmental Data Operation EPA QA/R-5*" (USEPA, 1994), "*Requirements for Quality Assurance Project Plans EPA/240/B-0/003 QA/R-5*" (USEPA, 2001), "*Guidance for the Data Quality Objectives Process, EPA QA/G-4, EPA/600/R-96/055*" (USEPA, 2000), and the "*Guidance for Data Quality Objective Process for Hazardous Waste Sites, EPA QA/G-4HW, EPA/600/R-00/007*" (USEPA, 2000). The organization of this QAPP follows the 2007 South Carolina Department of Health and Environmental Control (SCDHEC) guidance.

Project Organization

AMEC will provide a team of AMEC professionals, including the assistance of appropriate subcontractors, to complete the work assignments in accordance with the procedures described in this QAPP. Field sampling locations and media sampling will be conducted by AMEC employees. An organizational chart and flow of responsibilities is shown in the

flowchart in **Figure A-1**, and a brief description of AMEC's responsibilities is listed below. The anticipated team and their responsibilities are as follows:

Project Manager and Project Principal

Mr. Paul S. Johnstone, Professional Geologist (P.G.) will serve as the Project Manager (PM) and Project Principal for this project. The PM is responsible for overall project scope, organization, schedule, budget, and quality. He is the primary point of contact for the SCDHEC, RBTC, and AMEC's subcontractors, and is responsible for projecting resource needs and facilitating the assignment of those resources to the project. The Project Principal is responsible for the project's technical quality. The Principals' responsibility is to assure that the scope, organization, and schedule for each task will meet the project's quality objectives, the appropriate technical Principal Professional is involved with each task, and the review process for report writing is followed.

Mr. Johnstone will also be responsible for distributing and maintaining the approved QAPP and preparing and redistributing any revisions to the QAPP.

Discipline Leaders

Discipline leaders will be utilized to execute specific tasks related to the investigation and remediation of the Site. The discipline leaders will work directly with the PM and Project Principal to help execute the assignment and prepare project deliverables.

Field Operations Leader

The Field Operation Leader (FOL) will manage and coordinate field activities related to the investigation and have the responsibility of verifying and validating field measurements. Mr. Christopher H. Bruce is AMEC's FOL. The FOL will work directly with the PM to assist in the prioritization of scheduling tasks for investigation, interpretation of the data collected, and preparation of reports in addition to site management duties.

Quality Assurance Officer

Mr. Paul Brafford, Certified Hazardous Materials Manager (CHMM) is AMEC's Quality Assurance Officer (QAO)/Data Validator and is responsible for the overall quality of the fieldwork associated with the project, validation of the field measurements, and the quality of the chemical data generated. The QAO will work directly with the Project Principal to ensure that all work being performed follows AMEC's QA policies and the QA requirements of the project. Specific responsibilities include:

- Reviewing analytical protocols for measuring and monitoring;
- Assuring that laboratory personnel are trained and qualified in specified laboratory quality control (QC) and analytical procedures prior to receiving samples;
- Reviewing QA/QC results with laboratory QA staff and the RBTC/AMEC PMs;

- Making laboratory QC evaluations and, if problems are detected, making recommendations to the RBTC/AMEC PMs concerning repeat sampling and analyses;
- Informing the RBTC/AMEC PMs that appropriate QA/QC procedures have been established and are being implemented by the analytical laboratory.

Site Health and Safety Officer

Analysis of health and safety issues, including preparation of site-specific health and safety plans (HASPs) for each assignment, will be the responsibility of Mr. Gary W. Wise, AMEC's Local Health and Safety Representative (LHSR). A Site Health and Safety Officer (SHSO), generally the Site Manager/Field Coordinator, will be designated from the work crew assigned to each site, and will serve as the on-site resource for health and safety issues or concerns and administering the site specific HASP. A copy of the site-specific HASP is included in the RI/FS Work Plan (Appendix D).

Project Engineer

The Project Engineer for the site is Mr. Gary W. Wise, Professional Engineer (P.E.). The Project Engineer will supervise activities related to the remedial alternative selection and design of site modifications including remedial systems. Mr. Wise will be responsible for review and oversight of the remediation contractor and procedures, assurance that remedial goals are being met by the process, and review of plans and reports.

Project Chemist/Data Verifier

The Project Chemist/Data Verifier will supervise activities related to the chemical analysis, verification of chemical data quality, and reviewing and documenting the data validity. Ms. Judy Hartness is AMEC's Project Chemist for the Site. Ms. Hartness will work with the PM and AMEC's QAO. Specific responsibilities include:

- Tracking sample chain-of-custody through the laboratory;
- Verifying that laboratory QC and analytical procedures are being followed as specified in the QAPP and reviewing sample and QC data. This review will include examination of raw data such as chromatograms and checking arithmetic calculations for the samples analyzed, and inspection of reduced data, calibration curves and bound laboratory notebooks;
- Producing and/or reviewing a detailed validation report of the data collected

<u>Laboratory</u>

One laboratory is approved for receiving and analyzing samples collected at the Site. Analytical Environmental Services, Inc. (AES) located in Atlanta, Georgia will be responsible for chemical analytical testing of soil, sediment, surface water, and groundwater sampling at the site. AES has been assigned certification #98016003 by the SCDHEC. The laboratory's point of contact (POC) is presented below:

Analytical Environmental Services, Inc.
3785 Presidential Parkway
Atlanta, Georgia 30340-3704
Mr. James Forrest
(770) 457-8177
(770) 457-8188 (fax)

The laboratory is responsible for:

- Receiving samples from the field and verifying that incoming samples correspond to the chain-of-custody sheet;
- Maintaining records of incoming samples. Tracking samples through processing, analysis, and appropriate disposal at the conclusion of the program;
- Informing the Site Manager/Field Coordinator of discrepancies between chain-of-custody forms and sample package contents;
- Submitting quality control samples for analysis prior to and during the program;
- Reviewing raw data with laboratory analysts against calibration and QC records; and
- Preparing analytical data including QC data for audit by the QAO.

Subcontractors

Subcontractors may be used by AMEC to perform project elements that AMEC does not perform. Subcontractors must pass AMEC's QA policies, health and safety policies, as well as accept AMEC's terms and conditions.

Two drilling subcontractors and one surveying subcontractor have been identified for the project. Direct-push technology drilling will be provided by Probe Technology, Inc. from Concord, North Carolina. Hollow-stem auger drilling will be provided by Metro Drill, Inc. from Cowpens, South Carolina. Surveying will be provided by Freeland and Associates, Inc. from Greenville, South Carolina. The subcontractors POC are identified below.

Probe Technology, Inc. (SC License # 1415B, expires June 30, 2013)
Post Office Box 1369
Concord, North Carolina 28026
Mr. Arlen Burney
(704) 933-5538
(704) 933-5539 (fax)

A.E. Drilling Services, LLC (SC License # 562A, expires June 30, 2012)
Two United Way
Greenville, South Carolina 29607
Mr. William E. Barnes
(864) 288-1986
(864) 288-2272 (fax)

Freeland and Associates, Inc. (SC License # 4781, expires June 30, 2014)
323 West Stone Avenue
Greenville, South Carolina 29609
Mr. Michael Austin
(864) 217-4924
(864) 233-0315 (fax)

A5 PROBLEM DEFINITION/BACKGROUND

Problem Definition

Past manufacturing operations at the Site may have resulted in elevated concentrations of volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), metals (arsenic, barium, cadmium, chromium, lead, nickel, and selenium), nitrate, nitrite, and total petroleum hydrocarbon, oil and grease (TPH O&G) in soil, sediment, surface waters, and groundwater. Based on a comparison of detected concentrations from studies previously conducted at the Site to United States Environmental Protection Agency (USEPA) Residential Regional Screening Levels (RSLs) or Soil Screening Levels (SSLs) in soils and MCLs or Tap Water RSLs in groundwater, the principal constituents of concern (COCs) are volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs).

The objectives for the project are to determine the source, nature, and extent of contaminated media (soil, groundwater, surface water, and sediment) present at the site; determine if the concentrations of constituents present in Site media present an unacceptable risk to human health or the environment; and develop and evaluate alternatives for remedial action to prevent, mitigate, or otherwise respond to the migration or release or threatened release of hazardous substances, pollutants, or contamination at or from the Site.

Reason for the Study

The environmental investigation at the Site is being conducted under a Responsible Party Voluntary Cleanup Contract (RPVCC) between RBTC and the SCDHEC.

Decisions To Be Made

Evaluate Site media (soil, sediment, surface water, and groundwater) to determine impact from historical facility operations and determine if detected concentrations are greater than regulatory screening levels or action levels resulting in impacts to human health and the environment.

Regulatory Information

Previous environmental investigations at the Site were conducted under the SCDHEC Bureau of Water. This QAPP describes the proposed work to be conducted under SCDHEC RPVCC #05-5613-RP, managed by the SCDHEC Bureau of Land and Waste Management.

Background

The Site is located at 800 Woodside Avenue in Fountain Inn, Greenville County, South Carolina. A site location map is included as **Figure A-2**. The Site is located northwest of the intersection formed by South Carolina Highway 418 (McCarter Road) and Woodside Avenue. Access to the site is from either South Carolina Highway 418 (McCarter Road) or Woodside Avenue. The site is presently developed with a 124,793 square foot vacant former manufacturing facility where screwdrivers and spade bits were manufactured. The Site is approximately 22.85 acres in size and consists of a single parcel of land.

The plant is located in the approximate center of the property. Parking areas are located southeast of the plant followed by a mowed grassy field with a stand of trees between the parking area and McCarter Road. Northeast of the plant are landscaped areas, mowed grassy fields, and Woodside Road. Northwest of the plant are a mowed grassy field and woodlands. Southwest of the plant are a tank containment area, access road, and hazardous waste accumulation area with mowed grassy areas in between.

Past History

The site was developed with the manufacturing plant in 1984 and operations commenced in 1985 as Rosco Tools, a division of Vermont American Corporation (VAC) which subsequently became RBTC. Screwdrivers were manufactured initially and spade bit manufacturing was added in 1992. Nickel plating and associated wastewater pretreatment was present in the facility from 1985 to the early 1990s. A self-contained vapor degreaser was used at the facility from 1985 to the early 1990s. Manufacturing operations ceased in November 2003 and the facility was vacant until it was sold in September 2005 to Fountain Inn Investments I, LLC (assignee of Liberty Property Development Corporation).

Three primary manufacturing processes were performed at the site: manufacture of screwdriver handles and other specialty items; screwdriver head manufacturing; and spade bit manufacturing. The process areas discussed below are shown on Figure 2.

Screwdriver Handles and Other Specialty Items

Raw materials included flake cellulose acetate in 50 pound bags, scrap plastic from outside sources, plasticizer (Diethyl Phthalate, or DEP), and various colorants and powders. The cellulose acetate and scrap plastic were stored in Raw Materials Storage, the DEP was stored in two 6,000-gallon aboveground storage tanks (ASTs) located in a Tank Containment Area outside the plant, and the colorants and powders were stored in the Compounding Room. The DEP was transported to the Compounding Room from the Tank Containment Area by overhead piping.

The cellulose acetate and/or scrap plastic were combined with the DEP to form plastic pellets in two compounding lines (one for virgin cellulose acetate and one for scrap plastic). The plastic pellets underwent a water quench prior to being stored in four 30,000-gallon silos located outside the Compounding Room.

Water vapor containing DEP was condensed and accumulated in a settling tank in the Compounding Room. The DEP was skimmed off the settling tank, pumped into drums, and recycled in the compounding process. Initially, from 1985 to 1996, the water from the

settling tank was discharged directly to the sanitary sewer. After 1996, water from the settling tank was periodically pumped into drums for off-site disposal. The settling tank bottoms were cleaned out approximately every two years, and containerized in drums for off-site disposal. Plastic sweepings and DEP waste entrained in oil dry were accumulated and containerized in drums for off-site disposal. An exhaust blower system was used to remove vapors from the Compounding Room. A baghouse located outside the Compounding Room collected cellulose acetate dust, which was disposed in a dumpster on the site. Bag filters in the piping were containerized in drums for off-site disposal.

The plastic pellets in the silos were pumped to extruding machines that used electric burners to heat and melt the plastic, which were extruded as plastic rods that ultimately would become screwdriver handles or other specialty items. Flexible hoses were located on the extruders to add striping to the rods. The hoses were cleaned with acetone. The spent acetone was accumulated for subsequent distilling and reuse. Each extruder had a quench trough fed with a water hose. Overfills were collected in floor drains that discharged to the sanitary sewer.

The finished rods were transported to Rod Handle Storage prior to further processing in the Handle Machining Lathes. The Handle Machining Lathes used an aqueous coolant stored in a sump located on each lathe. The containers were pumped out manually and transported to an oil/water separator located in the Grinding Area. The oil from the separator was accumulated in drums and subsequently transferred to the Hazardous Waste Accumulation Building pending off-site disposal. The water from the separator was discharged to the sanitary sewer. The oil/water separator was previously located near the Quality Control (QC) lab located in the mezzanine under the stairs near the Acetone Room.

Plastic turnings from the lathes were captured by an overhead vacuum system that transported the waste plastic to the Grinding Area where the plastic was ground to a usable size and then transferred to the Plastic Scrap Regrinding Room. Floor sweepings in the Handle Machining Lathe area were collected and disposed in a dumpster. The sump bottoms at each lathe were drummed and accumulated for subsequent off-site disposal.

Some finished rod handles were further processed in the Drilling area with pneumatic drilling machines to drill special holes. Plastic turnings were captured in the same fashion as at the lathes. Floor sweepings in the Drilling area were collected and disposed in a dumpster. After drilling the holes, the rod handles were processed in the Handle Wash area where they were first placed in a tumbler to remove plastic burrs and then washed in a 180-gallon barrel washer containing water and detergent. The rod handles were then placed in a 120-gallon rinse tank. The wash and rinse water was discharged to sanitary sewer every couple of days. Bag filters in the wash line and rinse line were disposed in a dumpster.

Following drilling and washing, the vast majority of the rod handles went to the Polishing Room where they were polished with acetone. The acetone was located in a sump in the Acetone Line that was heated to create vapors. The handles were transported through the vapors on racks in a conveyor belt system. The majority of the acetone vapor condensed and returned to the sump while other vapors discharged to the air. An acetone dip tank and paint dip tank were also located in the polishing room. The acetone

dip tank was used to polish mallet heads and other specialty items. The paint dip tank was used to paint ends and noses for special order items.

Virgin acetone was stored in a 6,000 gallons underground storage tank (UST) located in the Tank Containment Area and initially piped to the Acetone Line via underground piping. At a later date, the underground piping was abandoned in place and replaced with aboveground piping.

The acetone sump was drained once a week into drums that were transferred to the Hazardous Waste Accumulation Building. The drums were initially disposed off site. Later, the acetone was reclaimed by an outside contractor who came in approximately every 90 days to distill the acetone. The recycled acetone was pumped to the UST and the still bottoms were containerized in drums and stored in the Hazardous Waste Accumulation Building until they were disposed off site.

After polishing, most handles were transferred to the Handle Storage area. Some handles were painted using a dip tank in the Painting area or with a Binks spray paint system also located in the Painting area. In the early 1990s, the Binks spray paint system was transferred to another RBTC facility. The paint used in the dip tank and Binks system was an oil-based paint thinned with acetone. Cleaning of spray nozzles and other parts was performed using acetone. The paint/acetone mixture from painting and cleaning was containerized in drums, transferred to the Hazardous Waste Accumulation Building, and recycled by an outside contractor.

On some handles and specialty items, printing was affixed to the plastic materials. Hot stamp printing using a foil tape was performed in the Hot Stamp area. Spent foil was accumulated and disposed in a dumpster. In the Ink Pad Printing area, a pad printer using organic ink was used to print the plastic materials. The ink was thinned with ink-specific thinners. Isopropyl alcohol was used to clean inks from the plastic materials so that the plastic wouldn't be affected. Acetone was used to clean printer components and was accumulated in a satellite accumulation area nearby. The spent acetone was processed as described previously. Waste paint rags and plastic cups and cardboard coated with paint were containerized in drums in a satellite accumulation area before they were transported to the Hazardous Waste Accumulation Building for off-site disposal.

Screwdrivers

Raw materials consisted of coiled carbon steel. Flat-head screwdrivers were fashioned in the Press area using mechanical, hydraulic presses. Phillips-head screwdrivers were fashioned in the ESCO Manufacturing area using cutting oils. Steel scrap from the processes was collected in totes and when full were transported to the Scrap Metal Rolloff for off-site disposal. Waste oils were drummed and transported to the Hazardous Waste Accumulation Building for off-site disposal.

Vapor degreasing was performed from 1985 to 1992/1993 in a self-contained system that initially utilized 1,1,1-Trichloroethane and later Freon 113. Vapor degreasing took place in what is now the ESCO Manufacturing area. At the time vapor degreasing operations were discontinued, the floor drains in the area were plugged. The vapor degreasing was replaced by an aqueous two-stage alkaline dip degreaser.

The screwdrivers were then processed in a heat treat line located in the Heat Treat area. From 1985 to 1988/89 heat treating was accomplished using a vacuum furnace.

Following heat treatment in the vacuum furnace, the screwdrivers were quenched in an oil bath then processed through wash, rinse, and drying tanks. The vacuum furnace was replaced in 1988/89 with a heat treat system that used a sodium nitrate quench bath and two rinse tanks. Periodically, the quench tank was cleaned of caked quench residue, which was disposed in a dumpster. Prior to 1997, the cleaning rinse water and water from the rinse tanks were discharged to the sanitary sewer. After 1997, the cleaning rinse water and water from the rinse tanks were pumped to the ground outside the facility.

Following heat treating, the majority of the screwdriver blades were processed in the Grit Blasting area to remove scale and burrs. There were three grit blasters that used steel shot as the blast media. One of the grit blasters was used to blast the tips of screwdriver blades that had been nickel plated at the facility or chrome-plated screwdriver blades that were received from other RBTC facilities. Spent blast media was collected in traps in the back of each machine and periodically emptied into the Scrap Metal Rolloff. Dust generating during the grit blasting operations was processed through a Metals Baghouse located outside the facility. Filters from the Metals Baghouse were initially disposed in the Scrap Metal Rolloff but subsequently were rebuilt and reused.

The screwdrivers and spade bits were next processed in the Screwdriver and Spade Bit Grinding area where the final faces and edges were ground. From 1985 to 1988, dry grinding was performed on screwdriver blades. The dry grinding operations were connected to the Metals Baghouse. Wet face grinding was also performed. Each wet-face grinder had an individual sump to collect grinding swarf and grinding oil. The sumps were cleaned out on a semi-annual basis and the grinding swarf was disposed in the Scrap Metal Rolloff. Sometime in the late 1980s, the wet face grinders were connected to a central coolant system. The grinding swarf was removed from the central coolant system and disposed in the Scrap Metal Rolloff and the grinding oil was recirculated. In 1992, with the addition of the spade bit line, a below-grade Henry Filter was installed in what was previously the wastewater pre-treatment room. At this time, all grinding operations were attached to the Henry Filter. Grinding swarf was removed from the Henry Filter and disposed in the Scrap Metal Rolloff and grinding oils were recirculated. On at least two occasions, all of the grinding oil in the Henry Filter system was pumped out and disposed off site.

After the grinding operations were completed, the screwdriver blade and spade bit surfaces were given their final treatment which included nickel plating, vibratory finishing, black painting, or lacquer coating.

Nickel plating was performed from 1985 to the early 1990s in the Nickel Plating Area. The process included barrel plating and reverse osmosis was used to reclaim the nickel. Wastewater from the plating process flowed to a settling tank then through a filter press. Solids from the filter press were containerized in drums for offsite disposal. The resulting wastewater was treated for pH in a wastewater pretreatment system before being discharged to the sanitary sewer. The wastewater pre-treatment system was located in what was subsequently the Henry Filter Area. At the time that nickel plating was discontinued, the wastewater pre-treatment system was removed.

Screwdriver blades and spade bits that required cleaning and polishing prior to plating were processed in the Vibratory Finishing area. Some blades and bits were nickel plated at the facility and others were shipped to other RBTC locations for plating. There were two vibratory bowls that contained a ceramic and plastic media for polishing and a

detergent for cleaning. Spent vibratory media was collected in a moat around each vibratory bowl and was periodically shoveled out and disposed off site.

Other screwdriver blades and spade bits were coated in a black paint process in the Oven and Black Painting Line. The blades and bits were dipped in a tank of paint and then cured in a gas-fired oven. The spent paint was removed from the tank and disposed off site twice a year.

The rest of the screwdriver blades and spade bits were coated in a drip spin lacquer process for rust prevention in the Dip Spin Lacquer area. Initially, an oil-based lacquer was used in a two-step process was used where the parts were first dipped in the lacquer, removed from the dip tank, placed in a spinner, spun, and then removed. Later a space age process with a solvent-based lacquer was used where the parts were dipped in the lacquer and then baked in an oven. Eventually, the solvent-based lacquer was replaced with a water-based lacquer. Since 1990, a centrifugal spin process was used similar to the initial lacquer coating process.

Following completion of the finishing process, the screwdriver blades were transported to the Bulk Screwdriver Storage area.

Spade Bits

Raw materials consisted of carbon steel blanks. The spade bits were stamped in the Press Area and then underwent similar heat treating, grit blasting, grinding, and finishing processes as described for the screwdrivers. Following completion of the finishing process, the spade bits were transported to the Packaging Area to be packaged and then shipped.

Assembly

Screwdriver blades, rod handles, and specialty items were assembled in the Screwdriver Assembly area. The assembled screwdrivers and specialty items were transported to the Packaging Area to be packaged and then shipped.

Previous Work

Several environmental investigations have been conducted by MACTEC at the Site. They include the following, which have previously been provided to the SCDHEC Bureau of Land and Waste Management:

- Preliminary Site Contamination Assessment, Acetone Release Site, Vermont American Corporation Facility, Fountain Inn, South Carolina (MACTEC Project 30290-6-7856.02), dated December 10, 1996;
- Report of Acetone Tank and Line Testing and Diethyl Phthalate Line Testing, Vermont American Corporation Facility, Fountain Inn, South Carolina (MACTEC Project 30290-7-8046.01), dated August 15, 1997;
- Underground Storage Tank (UST) Assessment Report, Vermont American Corporation, Fountain Inn Division, Fountain Inn, South Carolina, SCDHEC Permit #04235 (MACTEC Project 30200-1-9316-04-917), dated August 21, 2002;

- Report of Phase II Environmental Site Assessment, Vermont American Corporation, Fountain Inn Division, 800 Woodside Avenue, Fountain Inn, South Carolina (MACTEC Project 30200-1-9316-04-917), dated February 4, 2003;
- Second Revised Draft Report of Environmental Services, Vermont American Corporation, Fountain Inn Division, Fountain Inn, South Carolina (MACTEC Project 30200-1-9243-01-917), dated February 5, 2003;
- Results of Field Screening Ground-Water Sampling, Robert Bosch Tool Corporation (former Vermont American Corporation), Fountain Inn Division, Fountain Inn, South Carolina (MACTEC Project 3020019316-2, Task 04), dated October 8, 2003;
- Results of Field Screening Sampling, Robert Bosch Tool Corporation (former Vermont American Corporation), Fountain Inn Division, Fountain Inn, South Carolina (MACTEC Project 3020019316-2, Task 05), dated April 18, 2005.

The previous environmental work conducted at the site is described in more detail in Section 3.1 of the RI/FS Work Plan (Pages 3-1 to 3-10) to which this QAPP is included as Appendix B.

Regulatory Context

The activities described in this QAPP will be performed pursuant to RPVCC #05-5613-RP, executed August 29, 2005, between the SCDHEC and RBTC, for a Remedial Investigation/Feasibility Study (RI/FS) at the Site.

A6 PROJECT/TASK DESCRIPTION AND SCHEDULE

Summary of Work

The RI/FS proposed for the Site will evaluate the extent and risk posed by facility-related constituents in soil, groundwater, surface water, and sediment. The objective of the RI/FS is to:

- Determine the source, nature, and extent of contaminated media (soil, groundwater, surface water, and sediment) present at the Site.
- Determine if the concentrations of constituents present in Site media present an unacceptable risk to human health or the environment as defined by the USEPA Regional Screening Levels (RSL) for Chemical Contaminants at Superfund Sites (May 2012), SCDHEC Maximum Contaminant Levels (MCLs), USEPA MCLs, USEPA Tap Water RSLs, and SCDHEC Water Quality Criteria (WQC).
- Develop and evaluate alternatives for remedial action to prevent, mitigate, or otherwise respond to the migration or the release or threatened release of hazardous substances, pollutants, or contaminants at or from the Site.

The work to be conducted during the RI/FS is described more fully in the Field Sampling and Analysis Plan (FSAP) in the RI/FS Work Plan (Appendix C).

Work Schedules

The project schedule is described in Section 7.0 (Page 7-1) of the RI/FS Work Plan.

Investigation Areas

The investigation areas are described in Section 3.2 (Pages 3-11 to 3-20) of the RI/FS Work Plan and the FSAP (Section 4.0, Pages 4-1 to 4-9).

Time or Resource Constraints

There are no time or resource constraints that are expected to impact the environmental investigation or project schedule.

A7 DATA QUALITY OBJECTIVES AND DATA QUALITY INDICATORS

Data Quality Objectives (DQOs)

Data Quality Objectives (DQOs) are used to determine the type, quantity, and quality of data needed to reach defensible decisions. DQOs define the performance criteria and are part of a systematic planning process. This framework is used to ensure that the level of detail is commensurate with the intended data use and available resources. A seven step process has been developed by SCDHEC (as outlined in the "*Guidance Document for Preparing Quality Assurance Project Plans (QAPPs) for Environmental Monitoring Projects/Studies*" (September, 2008)). Each step is summarized below.

State the Problem

Past manufacturing operations at the Site may have resulted in elevated concentrations of VOCs, SVOCs, metals (arsenic, barium, cadmium, chromium, lead, nickel, and selenium), nitrate, nitrite, and TPH O&G in soil, sediment, surface waters, and groundwater. Based on a comparison of detected concentrations from studies previously conducted at the Site to USEPA Residential RSLs or SSLs in soils and MCLs or Tap Water RSLs in groundwater, the principal constituents of concern (COCs) are VOCs and SVOCs.

Identify the Decision

Site investigation data is needed to assess if the Site presents a risk to human health and the environment. Risk will be determined by comparing laboratory analytical results for Site media to regulatory screening levels to develop preliminary contaminants of potential concern (PCOPCs). The PCOPCs will be evaluated in the Feasibility Study (FS).

Identify Inputs to the Decision

Data will be collected to assess VOC, SVOC, and metals concentrations in soil, surface water, sediment, and groundwater as indicated in the various AOCs at the Site for comparison to investigation results and evaluation in human health risk assessments.

Probable parameters to be analyzed by media are summarized in **Table A-1** with the laboratory analytical methods as presented in **Table A-2** and **Table A-3**. The rationale for the parameters to be collected and method selection at the Site is discussed in the FSAP

(Section 4.0, pages 4-1 to 4-9). Both screening and definitive data will be collected and used.

Stream sediment samples will be collected from the unnamed tributary to Stoddard Creek at reference locations upstream of the suspected area of discharge of contaminated groundwater. Analytical results from the reference locations will be compared to downstream sediment data that may have been impacted by groundwater discharge.

Subsurface soil samples will be collected from the specified AOCs and analyzed for VOCs and SVOCs. These data will be used in the human health risk assessment to evaluate soil exposure pathways.

Groundwater monitoring wells and groundwater samples will be collected and analyzed for VOCs and SVOCs during the RI program.

Laboratory analysis will be requested with a Contract Laboratory Program (CLP)-like deliverable and electronic data deliverable (EDD) to allow data validation and calculation of DQIs.

USEPA RSLs will be the action levels utilized for decision making when reviewing analytical results for soils and sediment. SCDHEC Maximum Contaminant Levels (MCLs), USEPA MCLs, and USEPA Tap Water RSLs will be the action levels utilized for decision making when reviewing analytical results for groundwater. SCDHEC Water Quality Criteria (WQC) will be the action levels utilized for decision making when reviewing analytical results for surface water. An effort has been made to select the appropriate method to obtain data at or below the action levels. The selected screening or action levels are presented in **Table A-2**.

Define the Study Boundaries

The initial study boundaries include those areas defined as AOCs in the RI/FS Work Plan (Section 3.2, Pages 3-11 to 3-20 and Figure 14). The lateral extent of each AOC will be defined by visual inspection of surficial materials and laboratory analytical data. Study boundary depths are identified in the FSAP (Section 4.0, Pages 4-2 to 4-8).

Develop an Analytical Approach and a Decision Rule

If the RI results indicate media at the Site do not contain COCs above regulatory action levels or risk-based levels, then the RI data collection will be deemed complete.

If the RI results indicate media at the Site contains COCs above regulatory action levels or risk-based levels, then the RI data collection will continue until sufficient data is collected to characterize the nature and extent of the COCs and to complete human health risk assessments and a FS to identify and evaluate remedial alternatives.

Specify Limits on Decision Error

A data quality objective of 95 percent for usable data is proposed. For a description of the processes proposed to minimize decision error, see Data Quality Indicators below. Analytical sensitivity will be at levels lower then decision rule values (i.e., sufficient to

determine whether a given media contains COCs at concentrations greater than limits of interest).

Optimize the Design for Obtaining the Data

Historical data has been reviewed. Parameters have been selected based on previous studies and the large quantity of existing data. In addition, a site reconnaissance will be undertaken to select specific sample locations with suitable media for sampling. In this way, the target population will be divided into strata that are more homogeneous. Most of the sampling design is based on a phased approach.

The overall elements of the sampling program will be documented in the RI/FS Work Plan and FSAP. Assumptions supporting the sampling program will be provided in the RI/FS Work Plan and supporting documents.

The overall data quality objective is to produce data of sufficient quality for use in risk assessment, to support remedial alternative selection, and to monitor the effectiveness of remedial actions. These policies are intended to provide analytical data that will yield comprehensive and valid results and will comply with applicable federal and state regulations. Activities will comply with these policies and procedures and will be performed in accordance with the directives issued by AMEC's Project Principal Engineers or Scientists. (A qualified Principal Engineer or Scientist is one who has suitable experience with the techniques employed, conditions evaluated, and technologies involved and is authorized by corporate policy to practice in the discipline covered.)

Data Quality Indicators

Data Quality Indicators (DQIs) are qualitative and quantitative descriptors used in interpreting the degree of acceptability or utility of data. The principal DQIs are precision, bias/accuracy, representativeness, comparability, and completeness. Establishing acceptance criteria for the DQIs set quantitative goals for the quality of the data generated in the analytical measurement process. Of the five principal DQIs, precision and bias/accuracy are the quantitative measures, representativeness and comparability are qualitative, and completeness is a combination of both quantitative and qualitative measures.

The objective of this QAPP is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results that are legally defensible and of sufficiently high quality to meet the data quality objectives. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in subsequent sections. Data usability can be determined by review of field and laboratory measurement quality objectives (MQOs). The MQOs are the QA elements necessary to ensure proper chemical data quality management. MQOs for chemical data are expressed in terms of precision, accuracy/bias, representativeness, completeness, comparability (PARCC), and sensitivity. QA objectives provide the mechanism for ongoing control and evaluation of data quality throughout the project and ultimately will be used to define the data quality achieved for the various measurement parameters.

The laboratory MQO program will be assessed through internal laboratory quality control (QC), including method blanks; laboratory control samples (LCSs), surrogate standards, internal standards, and calibration standards. The laboratory MQOs are detailed in the laboratory's Quality Assurance Manual (QAM) and method Standard Operating Procedures (SOPs) in Appendix A.

The field MQO program assures that the samples being collected are representative of the Site via the media being sampled, the parameters being analyzed, and that the data generated are valid. This will be accomplished through:

- use of the standard field procedures, also known as Work and Test Procedures (WTP);
- accurate and detailed record keeping in the field notebooks and field logs;
- proper calibration of field equipment according to manufacturer's instructions; and
- collection and analysis of QA samples potentially including field duplicates, rinsate blanks, field blanks, trip blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples.

The selection of the samples within the AOCs ensures that the samples will be representative for the contamination on the property. AOC selection includes studies of historical data and location of contamination so as to follow the "plume" of contamination.

The purpose of this section is to address the specific objectives for PARCC and sensitivity. These data quality criteria are discussed below.

Representativeness

Representativeness is the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Representativeness of field data is dependent upon adequate sampling program and using proper sampling techniques and will be satisfied by following the FSAP (Appendix C of RI/FS Work Plan).

Representativeness in the laboratory is achieved by using proper analytical procedures, meeting sample-holding times, and analyzing and assessing field duplicated samples. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to existing analytical data, physical setting and processes, and constraints inherent to the sampling of the media of interest.

Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Precision is based on the relative percent difference (RPD) of duplicate analyses or duplicate spike analyses.

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 20 analytical samples. Field precision goals for this project will be 35% for water duplicates and 50% for soil duplicates. RPDs will be calculated as shown below.

Precision in the laboratory is assessed through the calculation of RPDs for two or more replicate samples. The RPD equation is given by:

$$RPD = A - B\ (100\%)/[(A + B)/2]$$

Where: RPD = Relative Percent Difference
A = First sample value
B = Second sample value

Laboratory precision will be assessed at a rate of 1 per 20 analytical samples. Laboratory precision is presented in Table A-2 and Table A-3.

Bias/Accuracy

Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction. Bias assessments are for environmental measurements are made using personnel, equipment, and spiking materials or reference materials as independent as possible from those used in the calibration of the measurement system.

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) that result from sampling and analytical operations. Accuracy in the field will be assessed through the use of field and trip blanks and through the adherence to sample handling procedures, applicable preservation techniques, and holding times.

Laboratory accuracy is assessed through the analysis of laboratory and matrix spikes (MS) or reference materials and the determination of percent recoveries. The equation to be used for accuracy is listed below.

$$R = [(A - B)/S](100\%)$$

Where: R = Percent Recovery
A = Value obtained by analyzing the sample with the spike added
B = Background value, i.e. the value obtained by analyzing the sample alone
S = Final concentration of the spike added to the sample

Accuracy/bias control limits are given in **Table A-2** and **Table A-3**. Laboratory accuracy/bias will be assessed at a rate of 1 per 20 analytical samples. Requirements for accuracy in analyzing reference materials (vendor provided QC samples) shall be according to the acceptance range given by the vendor. Requirements for laboratory spike and matrix spike accuracy/recoveries are shown on **Tables A-2 and A-3**.

Comparability

Comparability is the confidence with which one data set can be compared with another. Comparability of field data is dependent upon adequate sampling program design and using proper sampling techniques and will be satisfied by following the QAPP and the FSAP.

Analytical data will be comparable when similar sampling and analytical methods are used and documented in accordance with the QAPP. Consideration will be given to seasonal conditions, river flow, or other environmental factors that could influence analytical results.

Historical laboratory analytical data will be considered comparable when similar and comparable sampling and analytical methods are used and documented in accordance with the QAPP. Previous work conducted at the site included soil and water sampling that were analyzed for VOCs and SVOCs using gas chromatograph/mass spectrometer (USEPA Methods 8260B and 8270C, respectively) and metals using inductively coupled plasma/atomic emission spectrometry (USEPA Method 6010B), and mercury by manual cold vapor (USEPA Method 7470A/7471A). The laboratory analyses proposed in this QAPP will use the same or similar analytical methods and therefore the historical data will be comparable to the newly acquired data.

Completeness

Completeness is a measure of the amount of valid data obtained compared to the amount that was collected. The equation for completeness is presented in below.

$$\text{Percent Completeness (\%)} = \frac{\text{(Number of accepted data points)} \times 100}{\text{Total number of samples collected}}$$

The SCDHEC has requested a completeness goal of 100% unless the SCDHEC PM concurs that a lower completeness goal is appropriate. In the event that there is a problem with a sample, the SCDHEC PM will be contacted for consultation. Because of the requirement of 100% completeness, every sample is critical. Invalid samples shall be recollected.

Method Sensitivity

Sensitivity is the required method detection limits (MDLs) and the reporting limits (RLs) established to meet the project DQOs. The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that value is above zero. It is established and maintained as described in 40 CFR Part 136 Appendix B (Federal Register, 1992). The RL is sometimes called the practical quantitation limit (PQL) and is the lowest level that can be achieved within specified limits of precision and accuracy during routine laboratory operating conditions as defined by SW 846.

In general, the RLs for the COCs will be at or below risk-based screening levels for exposure to soil, sediment, surface water, and groundwater to assure both qualitative and quantitative results. Constituents with an RL higher than the screening level will be qualitatively assessed only and viewed as an uncertainty in technology screening and risk assessment. The analytical methods and sensitivities for VOCs, SVOCs, and inorganics are summarized on **Table A-4**, **Table A-5**, and **Table A-6**, respectively.

Rinsate blanks, method blanks, field blanks, duplicates, and matrix spike/matrix spike duplicate (MS/MSD) samples will be analyzed to assess the data quality from the sampling and analytical programs. Blanks must be lower than the PQL. All data associated with blanks above the PQL for an analyte must be flagged (see Section D).

Rinsate blanks consisting of distilled water poured over decontaminated sampling equipment will be submitted to the analytical laboratory to assess the data quality from the field sampling program. Rinsate blanks are analyzed to check for procedural contamination during sampling. Rinsate blanks will only be collected on non-dedicated sampling equipment. A rinsate blank will be collected and analyzed per sampling device per media per sampling event.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. Matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One MS/MSD will be analyzed for every 20 samples, or one per sample data group (SDG).

One field blank sample per media (soil, sediment, surface water, and groundwater) per day of sampling will be collected. Organic-free water will be used to fill a clean sample container while in the field for each media sampled during a given day.

Duplicate samples are analyzed to check for sampling and analytical reproducibility. One field duplicate will be collected and analyzed for every 10 samples for surface water, groundwater, sediment, and soil. Field duplicate precision goals for the project samples are: 35% for water matrices and 50% for solid matrices.

A8 TRAINING AND CERTIFICATIONS

Training of field personnel will be provided by the FOL. Routine training will be completed at the beginning of each field event if required. The FOL will review applicable procedures and the QAPP with each field personnel to verify that the project requirements and procedures are understood and implemented properly. A copy of the QAPP will be given to each person participating in this study. Personnel working at the Site will be required to read, understand, and conform to the requirements of the QAPP.

Experienced and qualified professionals with appropriate licenses and certifications will perform project fieldwork. Subcontractors utilized during the project (e.g., drillers, laboratories, professionals) will also maintain appropriate licenses or certifications required by the applicable regulatory agency.

Personnel conducting fieldwork at the Site will be appropriately trained in health and safety procedures. If appropriate, personnel conducting work covered by this QAPP will have obtained at a minimum, the 40-hour hazardous waste-site worker training program and the eight-hour annual refresher course in compliance with regulations stated in 29 CFR Part 1910.120. All certificates or documentation representing completion of training shall be maintained in personnel files archived at AMEC. The PM or FOL will verify that personnel have the necessary training and certifications prior to the implementation of the project.

A Site Health and Safety Plan (HASP) has been developed specific to the Site activities discussed in this QAPP and is included in the RI/FS Work Plan (Appendix D). The HASP applies to AMEC employees and AMEC subcontractors, only. Each field team will have a copy of the HASP during field activities. Personnel working at the Site will be required to read, understand, and conform to the requirements of the HASP. As site activities progress and if new information arises, the HASP will be updated, as necessary, to ensure compliance with the Occupational Safety and Health Act (OSHA) and safe working conditions.

A9 DOCUMENTATION AND RECORDS

Upon approval of the QAPP (see Section A1), the approved QAPP will be distributed to the individuals listed in Section A3. Each QAPP will be given a unique number based on the total number of copies published (e.g., 1 of 5, 2 of 5, etc.) and each QAPP will show the current revision number and date. The QAPP will be distributed by AMEC's PM either as a hard copy or in portable document format (PDF) on compact disk (CD). Copies will be distributed by hand delivery, courier, electronic mail, or surface mail. A hard copy of the QAPP will be archived in AMEC's Greenville, South Carolina office and an electronic copy will be archived on the Greenville office server.

Approved updates to the QAPP will be distributed to the individuals shown in Section A3. Each update will have a revision number and the date of revision. The updates will be distributed either as a hard copy or in PDF on CD. Updates will be distributed by AMEC's PM by hand delivery, courier, electronic mail, or surface mail. Hard copies of QAPP updates will be archived in AMEC's Greenville, South Carolina office and an electronic copy will be archived on the Greenville office server.

AMEC

Documentation of field activities will be completed using a combination of logbooks, field data records (FDRs), and sample custody records. Site and field logbooks are completed to provide a general record of activities and events that occur during each field task. FDRs have been designed for each exploration and sample collection task, to provide a complete record of data obtained during the activity. Laboratory data will be presented in hard copy via portable document format (PDF) and in an electronic data deliverable (EDD) in Microsoft Excel© formate. Project reports will be generated from the collected and validated data.

Deviations from the procedures specified in the QAPP and FSAP will be documented in the field logbooks and applicable FDRs. Such deviations may be dictated by site-specific conditions encountered during the sampling activity.

All project files, documents, reports, and data will be retained for a minimum of five years. Final documents will be maintained in the project files of AMEC. Field, laboratory, and raw data will be maintained in AMEC's project file for future use. Electronic files will be archived using Sharepoint.

<u>Field Logbooks</u>

The field logbooks provide a daily hand written account of all field activities. Logbooks are hardcover books that are permanently bound. All entries are made in permanent black or

blue ink, and corrections are made with a single line with the author initials and date. Each page of the logbook will be dated and signed by the person completing the log. Partially completed pages will have a line drawn through the unused portion at the end of each day. The following information is generally entered into the field logbooks:

- The date and time of each entry. The daily log generally begins with weather conditions;
- A summary of important tasks or subtasks completed during the day;
- A description of field tests completed in association with the daily task;
- A description of samples collected including documentation of any quality control samples that were prepared (rinse blanks, duplicates, matrix spikes, split samples, etc.);
- Documentation of equipment maintenance and calibration activities;
- Documentation of equipment decontamination activities; and
- A summary of any problems encountered during the day, which may include a description of corrective actions taken.

Following completion of the project, the log books are stored in the project file for a period of five years. Periodically during project execution, the log book pages are scanned to PDFs and archived in the project file on the Greenville office server. The office server is backed up to tape on a weekly basis.

Field Data Records

Sample FDRs contain sample collection and/or exploration details. A FDR is completed each time a field sample is collected. The goal of the FDR is to document exploration and sample collection methods, materials, dates and times, and sample locations and identifiers. Field measurements and observations associated with a given exploration or sample collection task are recorded on the FDR. FDRs are maintained throughout the field program in files that become a permanent record of field program activities. Periodically during project execution, the FDRs are scanned to PDFs and archived in the project file on the Greenville office server. The office server is backed up to tape on a weekly basis.

Laboratory Records

The following information will be included in each laboratory data report package. Hard copies of the laboratory report packages are maintained in the project file in the Greenville office for a period of five years. The electronic copy of the laboratory package is archived in the project file on Greenville's office server. The office server is backed up to tape on a weekly basis.

- Cover Letter with Laboratory Manager (or designee's) signature.
- Data reports for each sample submitted which include at a minimum:
  - Results and reporting units for each parameter;
  - Project defined reporting limits;

  - Date of extraction(s) and analyses;
  - List of project specified methodologies for each parameter; and
  - Dates of sample collection and laboratory receipt.
- Quality Control Summary Forms with method blank results, MS/MSD recoveries, and RPD calculations.
- Original Chain-of-Custody forms.
- A Sample Receipt Record documenting the condition of the samples upon receipt by the laboratory.
- A Case Narrative, as necessary, to discuss quality control limit exceedances, specific sample problems, and analytical methodology problems observed.

LABORATORY

The laboratory has a record keeping system that allows for the reconstruction of all laboratory activities that produced the analytical data. Each department has a different system for storing data.

Entries in manually recorded records are not obliterated by methods such as erasures, overwriting, whiteout or markings. All corrections to record-keeping errors are made by one line marked through the error. The individual making the correction initials and dates the correction. Corrections to electronic records are made by a manual notation that indicates the change to the record. This notation is kept with the affected record.

Metals

All raw data (including QC information), summary data, copies of prep logs and back log reports for each batch are placed in a folder with the batch ID number on the folder. Checklists for prep and analysis are placed in the front of the folder. Any non-conformances for the batch are placed in the folder. These folders are stored in numerical order, according to the batch ID, in the metals department. In addition, the electronic data associated with each instrument is downloaded into a separate file and saved periodically onto a CD-ROM to be stored off site.

Wet Chemistry

All raw data, prep information and QC data is recorded in logbooks for most analysis. If samples are analyzed on the Lachat, ion chromatograph or TOC analyzer, the raw data (including QC information) and the prep log (where applicable) for each batch are placed in a folder with the batch ID number on the folder. Any non-conformances for the batch are placed in the folder. These folders are stored in numerical order, according to batch ID, in the wet chemistry manager's office. In addition, the electronic data associated with each instrument is downloaded into a separate file and saved periodically onto a CD-ROM to be stored off site.

Organics

All QC data and copies of the extraction logs for each batch are placed in a folder with the batch ID number on the folder. In addition, any non-conformances for the batch are placed in the folder. These folders are stored in numerical order, according to the batch ID, in the organic manager's office. All organic raw data is given to the project manager and kept with the client's folder. In addition, the electronic data associated with each instrument is downloaded into a separate file and saved periodically onto a CD-ROM to be stored off site.

Project Management

Each project manager has a folder with the COC, sample receipt checklist (SRCL) and organic data (if analyzed for organics) in their office until the project is completed. Once the project is completed, the report and invoice are printed, along with a cover letter and case narrative (if necessary). If everything is correct, the project is reported to the client. A copy of the COC, SRCL, invoice and report are printed and placed in the folder before the report is mailed. If the raw data is to be sent to the client, a copy of the data is printed and placed in the folder before it is sent out. Once the project has been mailed, it is filed in numerical order in the file storage room. Reports are kept for five years.

Laboratory Information Management System (LIMS)

The LIMS holds all the information relevant to each project that is received at the laboratory, including all client information, and prep and analysis information for each test performed. LIMS data is backed up daily onto CDs. Copies are stored both on and off site.

Quality Assurance Records

Where necessary, records are generated and maintained for all quality associated activities conducted during all phases of the analytical work. QA records provide sufficient evidence that all specified QA requirements have been accomplished and satisfied and provide sufficient documentation to substantiate all reported findings and conclusions. These records are maintained by the laboratory for a minimum of five years after the initial issuance of the report (10 years for lead analysis). This ensures availability of the QA historical information. The following types of records shall be identifiable and retrievable: general QA records, inspection and test data records, and generated raw data and reports, etc.

Record Storage

All records for each project that is received at the laboratory must be held for a minimum of five years (10 years for lead analysis). Once the file has been reported and moved to the file storage area, it can only be retrieved again through the access log. This log is located in the sample-receiving department.

Hard copies of records are stored and filed numerically, alphabetically or chronologically by date or batch as appropriate for the type of record. Periodically, all records are transferred to storage boxes that are labeled with the month(s) and

year(s) in which the records were generated. Each box is given a unique number and entered into an archive log that includes a description of the contents of each box and the box location. The archived boxes are stored on-site for approximately one year and then transferred to an off-site storage facility. Boxes are stored in such a way to allow easy retrieval of records upon request. Final reports are also maintained electronically on computer hard drives and daily back-up tapes.

Electronic records are stored by department on the laboratory's portal server after scanning or converting the documentation to a PDF format. Customer Service stores the client reports by work order number. Laboratory data is downloaded and stored by department (asbestos, inorganic chemistry, metals, microbiology, sample prep, semi-volatile organics, volatile organics, and wet chemistry). Data contained in the LIMS and on other servers is backed up daily onto CDs.

Archive areas are protected against fire, theft, loss, environmental deterioration and vermin. Electronic records are also protected from electronic or magnetic sources. Archive areas are regularly inspected as part of the Internal Audit program. Representatives of the accrediting authority may have access to archived information.

In the event that the laboratory transfers ownership, the new proprietors retain sole custody and responsibility for all records. If the laboratory were to close, records shall be maintained at a commercial archival facility or maintained by another laboratory within the network. Records may also be transferred back to clients, if requested.

B MEASUREMENT/DATA ACQUISITION

B1 SAMPLING PROCESS/EXPERIMENTAL DESIGN

The sampling programs include analyses for the parameters in **Table A-1**. The sampling programs, including the number and depths of samples along with the required parameters, are described in the FSAP (Section 4.0, Pages 4-1 to 4-9). VOCs and SVOCs will be the focus of most studies.

The placement of sampling locations for investigations conducted at Site will primarily use the biased sampling approach. Since a biased sampling approach is proposed, all sample locations will be considered critical. The project DQOs will aid in the determination of the number and media of samples to be collected for the appropriate data set. General criteria for the determination of sample location, number, size and media are presented below.

- Sampling locations will be selected based on historical data, access, site reconnaissance, where previous contamination was detected, river characteristics, and project objectives. The anticipated or known groundwater flow direction will also be considered in selection of sample locations.
- The number of samples and environmental media sampled will be selected based on knowledge of the Site, the area potentially affected by site activities, the conceptual site model, and/or will be statistically derived based on the overall decision rule.
- Analytical parameters will be selected based on identification and quantification of Site COCs, knowledge of the Site history, media affected (or potentially affected), and project objectives. Analytical methods selected for this project will be USEPA-promulgated, consistent with federal and/or state regulations, and acceptable for generating data for comparison to risk-based screening levels.
- The sampling program assumes that conditions will be generally consistent throughout the sampling locations and that the analytical instrument response will be consistent with samples within the same medium. Due to the nature of the geology in the site area, there may be considerable variation in concentration of metals in soil. Detected concentrations will be compared to published literature (e.g., Canova, 1999) to determine if metals in soil will be considered representative of background or a release from site activities.
- It is anticipated that three sampling events will be conducted at different times during the assessment activities. The first event will be the collection of surface soil samples and subsurface soil samples from soil test borings. The second event will be the collection of pore water, surface water, and sediment samples. The third event will be the collection of groundwater samples. Samples will be collected during each day of each event and the group of samples (sample delivery group) collected on that day will be shipped to the laboratory via overnight courier.

Based on the location of the AOCs at the site, it is not likely that the sample locations will become physically inaccessible. However, it is possible that weather conditions may result in temporary inaccessibility. In this situation, sampling will be delayed until the conditions that result in inaccessibility have abated. AMEC will notify the SCDHEC PM when and if such conditions arise.

The number of field QC samples to be collected and the parameters to be analyzed at the Site is presented in **Table B-1**.

B2 SAMPLING METHODS

Project specific sampling methods are discussed in the FSAP (Section 4.0, Pages 4-1 to 4-9). This approach for collecting environmental information and samples is based on USEPA Region IV, Science and Ecosystem Support Division (SESD), *Field Branches Quality System and Technical Procedures*. General environmental sampling protocols are discussed below. Preservations, holding times, container types, and required sample volumes for environmental chemistry parameters are shown in **Table B-2**.

Surface Soil Samples

Surface soil samples will be collected to determine the nature and distribution of near surface (typically zero to two feet vertically) contamination. The specific sampling approach will be specified in the FSAP (Section 4.0, Pages 4-1 and 4-2). Surface soil samples collected for the purpose of human health risk assessments will be taken from the zero to two feet (depth) sampling interval.

Surface soil samples may be selected from pre-determined (historical detection of contaminants) or field-determined sampling locations (field screening of samples). Surface soil samples will be collected using a hand auger, split-spoon sampler, or stainless steel spoon. Excavated soil shall be placed in a stainless steel bowl. If the sample location is grassy, an eight-inch (or more) square piece of sod should be removed before collecting the sample. Soil materials shall be placed in a stainless steel bowl if composite samples are desired. Composite samples will be collected as follows:

1. Soil samples for VOC analysis will be collected in accordance with USEPA SW-846 Method 5035A. SW-846 Method 5035A is a closed-system, purge-and-trap process for the analysis of VOCs in solid materials (e.g., soils, sediments, and solid waste). While this method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs. Samples are collected using sampling kit provided by the laboratory that contains a disposable plastic coring device and four pre-weighed 40 milliliter (mL) glass vials with septum-sealed screwed caps ("5035 Kit"). Two of the vials contain stirring bars and five mL of reagent water (e.g., deionized water) and are used for low-level VOC analysis. One vial is unpreserved for screening and dry-weight determination and one vial contains 10mL of methanol for high-level VOC analysis. The plastic coring device produces a sample volume of approximately five grams. Four separate five-gram sample volumes are collected from the same soil stratum within close proximity to one another. It is very important to minimize the disturbance of the sample and the transfer of the sample

be made as quickly as possible to avoid loss of volatile components. One five-gram sample is placed in each sample vial. The soil samples in reagent water (deionized water) must be frozen within 48 hours and all samples must be analyzed within 14 days of sample collection. The closed-system purge-and-trap equipment employed for low concentration VOC samples is not appropriate for soil samples preserved in the field with methanol; therefore these samples are analyzed using Method 5030.

2. Place the remaining sample into a stainless steel bowl for mixing or perform sample homogenization in-situ.
3. Mix the sample using a dedicated clean stainless steel spatula.
4. Soil intended for other types of analyses (e.g., SVOCs, metals, etc.) should be placed in appropriate containers, capped, and sealed.
5. Immediately after the samples are collected, labeled vials and jars are checked for completeness of the sampling objective and chain-of-custody procedures are initiated. Samples are then placed in coolers for sample shipment.
6. Decontaminate the sampling equipment in accordance with the procedures described on page B-15 and the FSAP (Section 7.0, Page 7-1).

Discrete soil samples will be collected directly from the sampling tool (without placing in a bowl and mixing) and provisions 1, 4, 5, and 6 above apply. Appropriate sample containers are described in **Table B-2**.

Subsurface Soil Samples

Subsurface soil samples will be collected to determine the nature and distribution of contamination in the subsurface. The specific sampling approach will be specified in the FSAP (Section 4.0, Pages 4-2 to 4-4). Subsurface soil samples will be collected from variable depths in the unsaturated zone, which is anticipated to be approximately 30 feet in thickness.

Subsurface soil samples will be collected using a hand auger, split-spoon sampler, direct-push macrocore or other similar drilling/sampling technique. Soil materials shall be placed in a stainless steel bowl if composite samples are desired. Composite samples will be collected as follows:

1. Soil samples for VOC analysis will be collected in accordance with USEPA SW-846 Method 5035A. SW-846 Method 5035A is a closed-system, purge-and-trap process for the analysis of VOCs in solid materials (e.g., soils, sediments, and solid waste). While this method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs. Samples are collected using a 5035 Kit. Two of the vials contain stirring bars and five mL of reagent water (e.g., deionized water) and are used for low-level VOC analysis. One vial is unpreserved for screening and dry-weight determination and one vial contains 10mL of methanol for high-level VOC analysis. The plastic coring device produces a sample volume of approximately five grams. Four separate five-gram sample

volumes are collected from the same soil stratum within close proximity to one another. It is very important to minimize the disturbance of the sample and the transfer of the sample be made as quickly as possible to avoid loss of volatile components. One five-gram sample is placed in each sample vial. The soil samples in reagent water (deionized water) must be frozen within 48 hours and all samples must be analyzed within 14 days of sample collection. The closed-system purge-and-trap equipment employed for low concentration VOC samples is not appropriate for soil samples preserved in the field with methanol; therefore these samples are analyzed using Method 5030.

2. Place the remaining sample into a stainless steel bowl for mixing or perform sample homogenization in-situ.
3. Mix the sample using a dedicated clean stainless steel spatula.
4. Soil intended for other types of analyses (e.g., SVOCs, metals, etc.) should be placed in appropriate containers, capped, and sealed.
5. Immediately after the samples are collected, labeled vials and jars are checked for completeness of the sampling objective and chain-of-custody procedures are initiated. Samples are then placed in coolers for sample shipment.
6. Decontaminate the sampling equipment in accordance with the procedures described on page B-15 and the FSAP (Section 7.0, Page 7-1).

Discrete soil samples will be collected directly from the sampling tool (without placing in a bowl and mixing) and provisions 1, 4, 5, and 6 above apply. Appropriate sample containers are described in **Table B-2**.

Sediment Samples

Sediment samples will be collected in conjunction with surface water samples to characterize the nature and distribution of the contaminant. Sediment samples will be collected near the edge of the water body nearest the site. A boat should not be necessary in the collection of sediment samples. Sediment samples will be collected no sooner than 24 hours after a precipitation event, unless the FOL determines that the precipitation will not adversely affect the technical objective of the sample. The following equipment and supplies may be used during completion of sediment sampling explorations:

- Sample FDR;
- field log book;
- weighted tape measure;
- stainless steel gravity corer, hand corer, spoon, or other sediment dredge or similar type sampling device;
- stainless steel bowl;
- stainless steel spoon;
- stainless steel spatula;

- wooden stakes or pin flags to mark the sample location (if required);
- global positioning system unit to locate sampling location (if applicable);
- boat, anchors, electric motor, floatation devices, and oars;
- 5035 Kit (if sampling for VOCs);
- sample containers;
- decontamination supplies; and
- disposable or digital camera.

The sediment samples will be collected in the following manner:

1. Select the sample location, identify it on a Site map, and set a wooden stake or pin flags, as close as practicable, onshore.
2. Verify sediment sampling point is within the depositional area identified during the initial site reconnaissance.
3. Remove large stones and plant debris that are not an integral component of this sediment media. Exercise caution to avoid disturbing the sediments at the sampling point.
4. Use a gravity corer, hand corer, hand auger, trowel, or other equivalent equipment to collect sediment samples. A stainless steel spoon and bowl may be used for locations that are shallow (i.e., less than six inches). If the water is shallow enough, push the gravity corer or hand auger directly into the substrate until approximately one inch or less of the sampling device is above the sediment/water interface. If the substrate is hard or coarse, the corer may be rotated gently while it is pushed to facilitate greater penetration and reduce core compaction.
5. Remove the sampling apparatus gently from the sediment to avoid losing the sample, bring it to the surface.
6. Hold the sampling device above the water to allow residual surface water to run off the device. When water is no longer running off the device, transfer the sediment sample to a stainless steel bowl.
7. Soil samples for VOC analysis will be collected in accordance with USEPA SW-846 Method 5035A. SW-846 Method 5035A is a closed-system, purge-and-trap process for the analysis of VOCs in solid materials (e.g., soils, sediments, and solid waste). While this method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs. Samples are collected using a 5035 Kit. Two of the vials contain stirring bars and five mL of reagent water (e.g., deionized water) and are used for low-level VOC analysis. One vial is unpreserved for screening and dry-weight determination and one vial contains 10mL of methanol for high-level VOC analysis. The plastic coring device produces a sample volume of approximately five grams. Four separate five-gram sample volumes are collected from the same soil stratum within close proximity to one another. It is very important to minimize the disturbance of the sample

and the transfer of the sample be made as quickly as possible to avoid loss of volatile components. One five-gram sample is placed in each sample vial. The soil samples in reagent water (deionized water) must be frozen within 48 hours and all samples must be analyzed within 14 days of sample collection. The closed-system purge-and-trap equipment employed for low concentration VOC samples is not appropriate for soil samples preserved in the field with methanol; therefore these samples are analyzed using Method 5030.

8. For samples to be collected for other than VOCs, homogenize the remaining sediment within the stainless steel bowl with a stainless steel spoon so that each sample aliquot is representative of the whole. Take care to ensure that sufficient sediment is present in the stainless steel bowl to fill all of the associated sample fractions (containers) and duplicate fractions, if necessary. Collect the sample fractions using a stainless steel spoon and transfer the sediment into the sample containers.

Appropriate sample containers are described in **Table B-2**.

Surface Water Samples

Surface water samples may be collected to characterize the nature and distribution of contaminants in the unnamed tributary to Stoddard Creek. Surface water samples will be collected no sooner than 24 hours after a precipitation event, unless the FOL determines that the precipitation will not adversely affect the technical objective of the sample. In the event that the FOL proposes to collect surface water samples within 24 hours of a precipitation event, the SCDHEC PM will be contacted for concurrence. The surface water column will be measured using a weighted tape, engineer's rule, or other device. If possible, the position of the sampling point to the shoreline will also be measured and recorded.

Sampling locations for surface water samples will be specified in the FSAP (Section 4.0, Page 4-9) and collected in the following manner:

1. Collect the sample from the surface water body by immersing a sample collection device or sample bottle. Direct collection of surface water with the sample containers will be done in shallow water only (less than one foot). Otherwise, a clean one liter amber bottle will be used to collect the water sample which will then be transferred to the final sample container. Avoid collecting floating debris from the water surface. Take precautions to avoid stirring up silt or sediment in the area where surface water samples are obtained. At shallow water bodies (less than one to two feet), samples may be collected by wading or from a location on the shore. If wading is necessary, enter the sample area from downstream without disturbing the sediment and stand downstream of the sample location. If a disturbance is observed, wait until the water is visually clear before collecting the samples.
2. For informational comparison purposes, the following parameters will be measured by AMEC personnel, if possible, directly in the water body (if direct measurement is not possible, measure the parameters from the water remaining in the sampling device):

- temperature;
- pH;
- specific conductance; and
- any other task-specific field measurements required.

Appropriate sample containers are described in **Table B-2**.

Pore Water Samples

Pore water samples will be collected to characterize contaminants at the groundwater/surface water interface along the northern bank of the unnamed tributary to Stoddard Creek. Pore Water Samplers (Push Point/Henry Samplers), consisting of a thin (~1/8" diameter) steel tube, will be hand pushed into the soils along the bank of the creek. The following equipment and supplies may be used during collection of pore water samples:

- Sample FDR;
- field log book;
- weighted tape measure;
- stainless steel push point (Henry) samplers;
- wooden stakes or pin flags to mark the sample location (if required);
- global positioning system unit to locate sampling location (if applicable);
- decontamination supplies; and
- disposable or digital camera.

Sampling locations for pore water samples will be specified in the FSAP (Section 4.0, Page 4-8) and collected in the following manner:

1. Select the sample location, identify it on a Site map, and set a wooden stake or pin flags, as close as practicable, onshore.
2. Hand-push the sampler to the desired sampling depth and remove the inner steel rod to expose the screen.
3. Using a peristaltic pump, purge pore water from the sampler at a rate of approximately 100 mL per minute for one to three minutes until the water is visually clear.
4. AMEC personnel will measure the following parameters during purging for informational comparison purposes, if possible:
   - temperature;
   - pH;
   - specific conductance; and
   - any other task-specific field measurements required.
5. Collect the pore water directly into the sample containers.

Appropriate sample containers are described in **Table B-2**.

Groundwater Investigation

Groundwater investigations may include tap sampling of water supply wells, sampling existing monitoring wells, and installing and completing, developing, and sampling new groundwater monitoring wells. This section presents methods to complete these activities.

Borehole Completions

Borings will be completed at locations and by methods specified in the FSAP (Section 4.0, Pages 4-4 to 4-8). Prior to implementing and during any proposed drilling plan, the drilling subcontractor will:

- clean drilling equipment prior to mobilizing to the site;
- construct an on-site decontamination pad to clean downhole drilling equipment;
- arrange drill sites to minimize the potential for the possibility of spills and leaks from the drilling operation entering the borehole;
- store well drilling equipment and well installation supplies in a staging area as directed by AMEC;
- decontaminate drilling tools between borings;
- provide bentonite and granular backfill materials for AMEC approval; and
- clean well screens and riser, not prepackaged and documented clean by the manufacturer.

AMEC field personnel will be present during the drilling of borings, installation of monitoring wells, and decontamination activities. The field personnel will observe the exploration activities, maintain drilling logs, and collect appropriate samples.

Management of drill cuttings will be specified in the FSAP (Section 8.0, page 8-1). The boring methods employed at a given AOC will be selected on the basis of subsurface conditions and objectives for that exploration location.

Overburden Borings

Drilling of shallow borings may be required for the installation of overburden groundwater monitoring wells or collection of deeper soil samples. Overburden boreholes may be completed using 4.25-inch inside diameter (ID) hollow stem augers (HSAs). Borings to be completed as overburden groundwater monitoring wells will usually be advanced using 4.25 or 6.25-inch ID HSAs. The resulting nominal diameter of the borehole for monitoring wells will be approximately eight to ten inches.

During advancement of a soil boring, samples may be collected for geologic characterization or chemical analysis. Soil samples will be collected using a two-inch outside diameter (OD) by 24-inch long split-spoon sampler or by continuous soil cores. All samples will be obtained by driving the split-spoon sampler or advancing the soil core into undisturbed ground beneath the bottom of the HSAs. The frequency of sample collection

will be specified in the FSAP (Appendix C of the RI/FS Work Plan). The split-spoon will be driven by a free-falling dropweight weighing 140 pounds and falling 30 inches. The sampler is usually driven using standard A-rods connected between the sampler and drive head unless use of other equipment is approved by AMEC. The sampler will be driven 24 inches with the number of blows for each six inches of penetration recorded. In soils requiring 100 or more blows per foot of penetration, the sampling barrel will be driven 12 inches with the number of blows for each successive six inches of penetration observed and recorded. Refusal is considered 50 blows with no penetration.

The following equipment and supplies may be used during completion of overburden borings:

- flame-ionization detector (FID) or photo-ionization detector (PID) for field screening;
- water level indicator;
- Soil Boring Log;
- field logbook;
- soil color and or soil logging reference material;
- 5035 Kit (if sampling for VOCs);
- stainless steel bowl;
- stainless steel spoon;
- stainless steel spatula;
- sample containers;
- plastic sheeting to establish a clean area for equipment staging and sample collection; and
- disposable or digital camera.

The construction and development of monitoring wells is discussed below.

Bedrock Borings

Borings may be completed in bedrock to construct bedrock groundwater monitoring wells. Several drilling techniques including rock coring, rotosonic coring or air rotary/air hammer drilling methods may be used.

Cored Boreholes. Bedrock coring may be completed by installing a casing into the upper bedrock surface. A 7 7/8-inch OD air hammer or roller-cone bit is advanced approximately five feet into the bedrock or until competent bedrock is encountered. A six-inch ID surface casing with a casing shoe is installed to the bottom of the borehole. The six-inch casing is tremie grouted into place using Portland cement/bentonite grout. After the grout has sufficiently cured, approximately 12 hours at a minimum, core samples will be collected using a NX- to HX-size diamond bit and wire line core barrel which produces a 3.5-inch to 4.5-inch OD borehole in the bedrock and a 3.25-inch to 4.25-inch OD core sample.

The geologist will collect bedrock core samples for purposes of geologic classification. Core samples will be collected from designated boring locations. The core samples will be placed and stored in wooden, waxed cardboard, or corrugated plastic core boxes. Collection of these samples will allow for the characterization (if necessary) of the type of bedrock, weathering features, fractures, joints, and bedding planes. Rock quality designation (RQD) values for each core sample will be determined. A Rock Coring Log will be completed for each corehole.

Prior to installation of a monitoring well, the bedrock corehole may be over reamed with a 4 7/8 inch OD or larger roller-cone bit to allow for installation of a two- inch ID polyvinyl chloride (PVC) monitoring well.

Air Hammer/Rotary Methods. Air rotary/air hammer drilling methods may be used to install boreholes in bedrock to characterize groundwater and construct permanent monitoring wells. A
7 7/8-inch OD air hammer or roller-cone bit is advanced approximately five feet into the bedrock or until competent bedrock is encountered. A six-inch ID surface casing with a casing shoe is installed to the bottom of the borehole. The six-inch casing is tremie grouted into place using Portland cement/bentonite grout. After the grout has sufficiently cured, approximately 12 hours at a minimum, a 5 7/8 inch OD air hammer bit is advanced into bedrock and the hole is drilled to the target depth.

If overburden water is not present, the surface casing may be installed with a casing shoe without cement grout, allowing the soil materials and cuttings to backfill the annulus around the casing. An air hammer/air rotary drilling log will be completed for each borehole.

Monitoring Well Installation

AMEC field personnel will be present during monitoring well installation to record details of the well construction. Monitoring wells will be installed in designated borings unless difficult drilling condition requires abandoning the boring. Abandoned borings will be tremie grouted in the presence of Site field personnel from the bottom of the boring to the ground surface in one continuous operation, as required by South Carolina Well Standards R.61-71.H.2.e.

The following equipment and supplies may be used during monitoring well installation:

- water level indicator;
- Monitoring Well Log;
- field logbook; and
- disposable or digital camera.

Well installations will begin as soon as possible following borehole completion. If sufficient time has passed (i.e., greater than 24 hours), the borehole must be measured or checked to make sure that the boring has remained open to the termination depth. Once begun, well installations will continue uninterrupted, to the extent possible, until grout placement is completed. Well screen and riser, if not pre-cleaned and packaged to maintain cleanliness by the manufacturer, will be cleaned with deionized water prior to

installation in the borehole. Well screens will have a bottom plug. Where a protective casing is desired, solid riser will extend from the screen to approximately 2.5 feet above ground surface. If the well is to be completed as a flush mount, the solid riser will extend to approximately 0.25 feet below ground surface (bgs). When depth to groundwater is 15 feet bgs or greater, sand pack or filter material will be installed around the well screen to approximately two feet above the well screen. A minimum two-foot-thick seal consisting of bentonite pellets, or bentonite slurry, will be placed above the sand pack. The bentonite pellets will be hydrated prior to placement of the grout seal. In the event that groundwater is shallower than 15 feet bgs, the well construction dimensions will be adapted to help ensure that the aquifer is optimally characterized while maintaining well integrity. Modifications to monitoring well installation requirements may be necessary if the depth to groundwater is shallow. The bentonite seal and sand-pack extension may be reduced, though it is preferred that the bentonite seal not be less than two feet. Modifications to installation requirements will only be implemented with approval from the FOL. Monitoring Well Logs for aboveground and flush mount wells will be completed.

A cement-bentonite grout will be placed in the annular space above the bentonite seal layer. The mixed grout will be recirculated through the grout pump prior to placement to produce thick lump free mixture. The cement-bentonite grout seal will extend from the top of the bentonite seal to ground surface. Grouting will be completed as a continuous operation in the presence of the field personnel. The grout will be pumped into the annular space under pressure using a rigid tremie pipe placed at the top of the bentonite seal to ensure a continuous grout seal. The protective casing or flush mount will be sealed in the grout.

A steel protective casing or flush mount manhole will be installed around the well to prevent damage to the wells by vehicular operation. If an above ground protective casing is used, a two-foot by two-foot concrete pad, six inches thick, will be placed around each well. For bedrock well, the surface casing installed to bedrock may also be used as the protective casing.

For wells installed for the purpose of monitoring the deeper portions of the overburden aquifer, a 10-inch nominal diameter boring will be advanced through the overburden soils using air rotary, mud rotary, or auger drilling techniques to the desired casing depth, which will be at least five feet deeper than the bottom of the well screen in any nearby shallow overburden monitoring well. A six-inch diameter casing will be installed to the termination depth of the borehole. The annular space between the borehole and the six-inch casing will be pressure grouted with a cement/bentonite grout mixture to near ground surface. The grout will be allowed to set-up and cure for 24 hours. After the grout cures, the boring will be advanced into the underlying overburden to the desired total depth. A monitoring well will be installed as described above.

The following materials will be used in well construction:

- Riser will be flush-threaded PVC, Schedule 40, two-inch to four-inch (nominal) ID. No PVC solvent or lubricant will be used. The well screen will be factory-slotted, with a slot width of 0.010-inch. A loose-fitting PVC cap will be used to cover the top of the well riser and will allow equilibration of the well water level with atmospheric pressure. Water table well screen lengths will typically be 10 feet. The screen in wells that monitor the water table will extend two to five feet above the water table.

- Grout will be composed by weight of 20 parts Portland Type II cement to one part bentonite, with a maximum of eight gallons of water per 94-pound bag of cement. These proportions may be modified with approval. Bentonite will be added after mixing the cement and water.
- Bentonite pellets or slurry used in the seal will be a commercially available product designed for well sealing purposes.
- Sand material used in the filter pack around the well screen will be selected to be compatible with both the screen slot size and aquifer materials.
- A four-inch to six-inch diameter stick-up protective steel casing will be installed around all wells to be completed above ground surface. This casing will extend approximately 2.5 feet above ground surface and will be seated 2.5 feet into the well seal grout. The protective steel casing will extend a maximum of 0.2 feet above the top of the well cap. A mortar collar will be placed between the protective casing and the PVC riser prior to well development. This collar will extend 0.5 feet above the ground surface. A weep hole will be drilled in the protective casing above the mortar collar. The casing will be closed with a lockable, hinged cover. The cover will prevent entry of water but not air. Therefore, the well will be open to the atmosphere via a loosely fitting cap to allow for water level stabilization. The same key will be used for all padlocks placed on the new monitoring wells at the time of installation. Alternatively, a flush-mounted manhole cover may be used.
- After the grout seal has set, it will be checked for settlement. If needed, additional grout of approved composition will be added to fill any depressions.

Monitoring Well Development

Monitoring well development will be performed, as soon as practical, after well installation but not sooner than 48 hours following placement of the grout seal. The following data will be recorded on a Well Development FDR:

- well designation;
- date of well installation;
- date of development;
- static water level before and after development;
- quantity of drilling fluid lost during drilling or used during well installation;
- quantity of standing water in well and annulus (30 percent porosity of saturated annulus assumed for calculation) prior to development;
- specific conductivity, temperature, turbidity, and pH measurements will be taken by AMEC personnel for informational purposes and recorded at the start, at the end of each development volume, and at the conclusion of development. Calibration standards will be run prior to each day's operation in the field, and as necessary during the course of the day. Daily

instrument calibration data will be recorded on the Field Instrumentation Calibration Record;

- depth from top of well casing to bottom of well;
- screen length;
- depth from top of well casing to top of sediment inside well, before and after development;
- physical character of removed water, including changes during development in clarity, color, and particulates;
- type and size/capacity of pump and/or bailer used;
- height of well casing above/below ground surface;
- typical pumping rate; and
- quantity of water removed and time for removal.

Development of wells will be accomplished with a submersible pump, peristaltic pump, and/or bailer. Bailers will be used to develop wells only where the volume of water is so small that other development methods are clearly inappropriate. The pump will be periodically raised and allowed to drain back into the hole in order to induce flow out through the well screen. A surge block may be used in instances where field personnel expect that development may be improved by its use. Water will not be added to the well to aid in development. Non-dedicated pumps will be decontaminated prior to use in the next well (see page B-17 and FSAP, Section 7.0, Page 7-1). Development fluids will be containerized and handled as described on page B-17 and in the FSAP (Section 8.0, page 8-1).

A well is considered fully developed when all the following criteria are met:

- the well water is clear to the unaided eye;
- the sediment thickness remaining in the well is less than one percent of the screen length; and
- the total volume of water removed from the well equals five times the standing water volume in the well (including the well screen and casing plus saturated annulus, assuming 30 percent porosity).

Where possible, well development will continue until turbidity measurements in nephelometric turbidity units (NTUs) from well development volumes vary by less than approximately 10 percent.

Purging and Sample Collection

Depending on the constituents of concern, standard purging techniques or low flow purging techniques may be used to collect groundwater samples from monitoring wells. Appropriate sample containers are described in **Table B-2**.

Low Flow Purging

The following procedures were developed in accordance with the USEPA guidance document "*Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells*", dated July 30, 1996.

The following steps outline the purging and sample collection activities for low-flow purging and sampling. A stainless steel or PVC submersible bladder pump with a Teflon® bladder will be used to conduct the low-flow purging and sampling. Alternatively, a peristaltic pump may be used if the depth to groundwater is shallow and low flow rates can be obtained to minimize sample turbidity. Field parameter measurements will be made using instrumentation and a commercially manufactured flow through cell. Sample collection information will be recorded on the Low Flow Groundwater Sampling FDR. The USEPA guidance will be used for purging and sampling procedures. Procedures to be followed for decontamination, quality control sampling, logbook entries, etc., also presented in the USEPA guidance, are specified in other subsections within this QAPP.

1. Determine target depth for location of the pump intake. Target depth should be the portion of the screened interval that intersects the zone of highest hydraulic conductivity. If the zone of highest hydraulic conductivity is unknown, or if the screen is placed within homogenous material, then the target depth will be the midpoint of the saturated screen length. Primary flow zones should be identified in wells with screen lengths longer than 10 feet, or in wells with open boreholes in bedrock.
2. Measure and record the depth to water. Care should be taken to minimize disturbance of the water column within the well during pre-sample measurements.
3. Decontaminate pump and tubing prior to use (if pumps and tubing are dedicated then this applies to the initial effort only).
4. Carefully lower the pump to the predetermined target depth. Start the pump at a purge rate low enough to achieve 0.3 feet of drawdown or less based on historical data. If sampling the well for the first time, start the pump at the lowest possible setting (or approximately 100-milliliter [mL] per minute) and slowly increase the speed until discharge occurs. Check the water level. Adjust pump speed until there is little or no drawdown (less than 0.3 feet if possible). If the drawdown achieved at a pump rate of approximately 100 mL per minute exceeds 0.3 feet, but remains stable, continue purging until indicator field parameters stabilize.
5. Monitor and record pumping rate and water levels every three to five minutes (or as appropriate) during purging. Record any adjustments to pumping rates.
6. To monitor the effectiveness of the purging process, field parameters for informational purposes will be collected by AMEC personnel using a flow through cell (the flow through cell cannot be used for turbidity measurements and the sample for turbidity measurement must be collected prior to entering the flow through cell). Purging is considered complete and sampling may begin when the field parameters have stabilized. Stabilization is considered to be achieved when three

consecutive readings, taken at three to five minute intervals, are within the following limits:

- turbidity (+/- 10% for values >1 NTU)
- dissolved oxygen (+/- 10%)
- specific conductance (+/- 10%)
- temperature (+/- 10%)
- pH (± 0.1 standard unit)
- oxidation-reduction potential (± 10 millivolts)

7. The final purge volume must be greater than the stabilized drawdown volume plus the tubing extraction volume.
8. During purging and sampling the tubing should remain filled with water.
9. Disconnect the tubing from the flow through cell to collect the analytical samples. Water samples for laboratory analyses must not be collected after water has passed through the flow through assembly. Fill sample containers directly from the tubing without alterations to the pumping rate.
10. If a VOC sample is to be collected, that fraction will be collected first. The VOC sample container will be completely filled without air space within the container. The remaining samples will be collected for SVOCs, metals, and any other fraction specified in the FSAP (Appendix C of the RI/FS Work Plan) for the sample location.
11. For subsequent sampling efforts, duplicate the pump intake depth and final purge rate from the initial sampling event (use final pump dial setting information).
12. If using a non-dedicated pump, remove the pump and perform an external rinse with deionized water on the pump and external tubing. Obtain and record a depth to bottom of well measurement before closing the well.

Special Cases:

If the above sampling criteria cannot be met after four hours of purging, the following options are available:

- continue purging until stabilization is achieved;
- collect sample using a bailer; or
- discontinue purging and collect samples.

If the recharge rate of the well is less than the lowest possible extraction rate of the pump (i.e., drawdown does not stabilize at a purge rate of approximately 100 mL per minute or less), the purge rate will be increased and the water will be evacuated down to the pump intake level. This will result in several feet of stagnant water below the pump intake that will not be evacuated. The pump should then remain in place, and the well should be sampled after the water level has recovered to at or near the initial static water level. Collect the sample from the pump at a pumping rate of approximately 100 mL per minute.

For wells where the low flow protocol could not be followed due to recharge conditions or prior knowledge of well characteristics, a Groundwater Sampling FDR will be completed.

Standard Purging

Monitoring wells will be purged prior to sample collection to remove any stagnant water from the well so that the samples collected will be representative of the groundwater quality in the vicinity of each well. For wells that recover quickly, a minimum of three volumes of water will be evacuated. For informational purposes, specific conductance, pH, and water temperature will be measured by AMEC personnel periodically during well evacuation. Wells that evacuate to dryness with less than three well volumes being removed will be sampled as soon as the well has recovered enough to yield sufficient volume for a sample.

The monitoring wells will be purged using a three-foot long by 1.6-inch diameter disposable polypropylene or Teflon® bailer attached to an unused polypropylene cord, or by a submersible electric pump. The wells will be sampled using a bailer as described above or with unused polypropylene tubing attached to the submersible pump sampling port immediately upon completion of purging. To minimize the potential for cross-contamination between wells, a new clean bailer will be used or the submersible pump will be decontaminated. A Groundwater Sampling FDR will be completed by the sampler.

Residential Well Sample Collection

For collection of samples from residential or private water supplies the following method will be used.

The sample will be collected from a tap on the well, if available, and if not, the closest source to the well that is not affected by any water treatment, filtering, or water softening devices of any sort. The sampler will inspect the plumbing system to verify the sampling point.

The tap will be run for approximately 15 minutes to cause the pump system to turn on bringing fresher water from the well. Samples will be collected directly from the tap. Following sample collection, field parameters consisting of pH, conductivity, temperature, and turbidity will be measured by AMEC personnel for informational purposes from a separate sample container. A Groundwater Sampling FDR will be completed by the sampler. Appropriate sample containers are described in **Table B-2**.

Hydraulic Conductivity (Slug) Tests

Rising or falling head slug tests will be performed in selected monitoring wells to determine the hydraulic conductivity of the formation material exposed to the well screen. Hydraulic conductivity is a constant of proportionality relating to the ease with which a fluid passes through a porous medium. The field procedure used to determine this parameter is as follows:

- Measure the static groundwater elevation in the well to be tested;
- Effect a sudden change to the static water level in the well by using a slug of a known volume of water; and

- Measure the rate (feet per second) at which the water column recovers to its original level.

Data will be reduced and the hydraulic conductivities computed using commercially available software and techniques described in: Bouwer, H. and R. C. Rice "*A Slug Test for Determining Conductivity of Unconfined Aquifers with Completely or Partially Penetrating Wells*," Water Resources Research, 12 (1976): 423-28, or other appropriate methodology.

Decontamination

The objective of this section is to provide procedures to ensure the removal of COCs from sampling, drilling, and other field equipment to concentrations that do not adversely impact the investigation objectives. The procedures outlined below should be adhered to, unless instructed otherwise within the FSAP (Section 7.0, Pages 7-1 and 7-2).

Cleaning procedures outlined in this section are intended to be used by field personnel for field cleaning of sampling and other equipment. Deviations from these procedures must be approved by the Project Manager and documented in the field logbook.

Specifications for Cleaning Materials

Specifications for standard cleaning materials referred to in this section are as follows:

- Soap shall be a standard brand of phosphate-free laboratory detergent such as Liquinox®.
- Distilled water shall be used as a decontamination water source.
- Solvent will be pesticide-grade isopropanol.

Handling and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used for containing the specified cleaning solutions:

- Soap must be kept in clean plastic, metal or glass containers until used. It will be poured directly from the container during use.
- Distilled water may be kept in its original one-gallon containers or transferred into clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.

Safety Procedures for Field Cleaning Operations

All field personnel will exercise caution and follow all applicable safety procedures contained in the HASP (Appendix D of the RI/FS Work Plan) when handling cleaning materials. At a minimum, the following precautions will be taken in the field during these cleaning operations:

- Safety glasses with splash shields or goggles, and latex or nitrile gloves will be worn during all cleaning operations.
- Eating, smoking, drinking, or any hand to mouth contact will not be permitted during the cleaning operations.

Field Cleaning Procedure for Downhole Drilling Equipment

The following procedures are to be used to clean downhole drilling equipment between drilling boreholes in which monitoring wells will be installed:

1. Drilling rods and bits will be placed on racks or sawhorses and steam cleaned (high pressure hot water). Sampling equipment that is steam cleaned will be placed on racks or sawhorses at least two feet above the floor of the temporary decontamination pad. Special care will be taken when steam cleaning polyvinyl chloride (PVC) or plastic items.
2. Sampling devices (split spoons) will be scrubbed with potable water and soap then rinsed thoroughly with distilled water between each subsurface soil sampling interval. The split spoon samplers will be steam cleaned between boring locations.

Field Cleaning Procedure for Sampling Equipment

All field sampling equipment used to collect soil, sediment, surface water and groundwater samples (e.g., water level meters, tapes and non-dedicated field equipment) will be cleaned between each sampling locations follows:

1. Clean with tap water and soap using a brush if necessary to remove particulate matter and surface films. Equipment may be steam cleaned (soap and high pressure hot water) as an alternative to brushing. Sampling equipment that is steam cleaned will be placed on racks or saw horses at least two feet above the floor of the decontamination pad. PVC or plastic items will not be steam cleaned.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with analyte free water.
4. Rinse thoroughly with solvent (pesticide grade isopropanol). Do not solvent rinse PVC or plastic items.
5. Rinse thoroughly with organic/analyte free water. If organic/analyte free water is not available, equipment will be allowed to completely dry. A final rinse will not be applied with analyte water. Organic/analyte free water may be generated on-site utilizing a portable system.
6. Remove equipment from the decontamination area and cover with aluminum foil (non-metal sampling) or plastic (metals sampling). Equipment stored overnight will be wrapped in aluminum foil and covered with clean, unused plastic.

Handling of Cleaned Equipment

After field cleaning, only personnel wearing clean nitrile or other appropriate material (non-latex) gloves will handle equipment to prevent re-contamination. In addition, the cleaned equipment will be moved away from the cleaning area to prevent re-contamination. If the equipment is not to be immediately re-used it will be covered with or wrapped in aluminum foil and stored in an area away from potential contaminants. Equipment rinsates will be collected on undedicated equipment to verify the decontamination of the equipment.

Investigative Derived Waste

All investigation-derived waste (IDW) generated during proposed assessment activities will be containerized. The following identifies the types of IDW that could be generated during the investigation and their disposition:

- Tyvek®, gloves, paper towels, and other miscellaneous trash generated during investigation will be double bagged, placed in a commercial dumpster, and managed as municipal solid waste.
- Based on anticipated compounds, their respective concentrations, and quantities generated, drill cuttings and unused sample material (soil) will be containerized and characterized for disposal as appropriate.
- Development and purge water will be containerized and characterized for disposal as appropriate.
- Spent decontamination fluids will be containerized and characterized for disposal as appropriate.
- Containerized IDW will be stored on-site in a designated controlled area (i.e., within the locked security fence of the site) while analytical results are pending and until removal is scheduled.
- IDW will be manifested, removed, and transported from the site by a properly licensed waste hauler.
- IDW will be disposed of at a properly licensed waste disposal facility.

Sample Containers

The laboratory will provide sample containers. The containers are commercially prepared by the vendor and will be certified by production lot to be contaminant free.

The laboratory will assemble a sampling kit for each sampling event. The sampling kit includes the following items, when applicable: cooler(s), sample containers (with appropriate preservative), and Chain-of-Custody forms.

The laboratory will provide rinsate blank containers containing the preservative for each type of sample collected. The same preservative will be used for both blanks and samples.

Glass bottles will be adequately protected from breakage by placing bottle in bubble bags, bubble wrap, or equivalent protection. Forms will be placed in a waterproof bag. The

cooler will then be sealed with packing tape and shipped to the laboratory. Samples will be shipped via overnight delivery (e.g., Federal Express) or hand carried to the laboratory by a laboratory courier.

Preservatives

Preservatives, when applicable, are provided by the laboratory by adding the preservative to the sample containers. The preservatives used are American Chemical Society reagent grade or better or the equivalent. Container and preservatives required for each method is presented in **Table B-2**.

B3 SAMPLE HANDLING AND CUSTODY

Sample Identification

In order to ensure that each field sample number for every sample collected is unique, a 14-digit sample identification system will be used. The first nine place holders will be the sample matrix identification (ID). The tenth through fourteenth place holders will be used to describe the vertical depth of the sample and the sample type as shown below:
An exploration and sample identification number will look like:

SS-04-01X000XX

where:

Digit 1,2 A two-letter site type designation will be used. The following list shows the different types of samples and letter designations to be used for this project.

SS	=	surface soil (zero to two feet)
PW	=	pore water
SW	=	surface water
SD	=	sediment
HA	=	hand auger (deeper soil > two feet)
SB	=	soil boring
MW	=	overburden monitoring well
BW	=	bedrock monitoring well
RW	=	residential water supply well
RB	=	rinse blank (equipment)
FB	=	field blank
TB	=	trip blank

Digit 3 Mandatory hyphen (-)

Digits 4,5 Indicates the AOC.

01	=	Tank Containment and Underground Piping Area
02	=	Heat Treat Cleaning Water Disposal Area
03	=	Former Metals Baghouse
04	=	Former Scrap Metal Rolloff
05	=	Former Empty Drum Storage Pad
06	=	Compounding Room Blower Exhaust

07 = Storm Water Outfalls
08 = Former Oil/Water Separator
09 = Former Hazardous Waste Accumulation Building

Digit 6 Mandatory hyphen (-)

Digits 7,8 Used to indicate the number of the boring, monitoring well, sediment sample, etc. from 00 to 99.

Digit 9 Used for monitoring well clusters or multilevel piezometers where A through Z indicates individual wells, or screened intervals within the cluster. An X will be used for all other samples. This digit can also be used for sample matrix types that may exceed 100 locations.

Digits 10-12 Used to indicate the top depth of the sample vertically below ground surface.

Digit 13, 14 Used to designate type of sample or round collected

XX = normal field sample
XD = duplicate field sample
MS = matrix spike
MD = matrix spike duplicate
X1 = Round 1 groundwater sample
X2 = Round 2 groundwater sample (etc.)

Field duplicates MS/MSD and/or split samples are collected from the same sampling location, depth, or interval as the original field sample. Where duplicate, MS/MSD, or split samples are to be collected, samplers should fill all containers for a given analytical parameter before moving on to the next parameter.

The quality control samples must also have a unique number; therefore the TB will be designated as:

TB-00-001 through TB-00-099

Sampler or equipment rinsate blanks (RB) will be designated as:

RB-00-001 through RB-00-099

The field blank (FB) is the investigation source water used for decontamination purposes. These samples will be designated as:

FB-00-001 through FB-00-099

Field Custody Procedures

Field logbooks and FDRs will provide the means of recording data collection activities. As such, entries will be described in enough detail so those individuals participating in the sampling can reconstruct a particular situation without reliance on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified with the project specific document number.

The logbook will contain the following information for each activity:

- Location;
- Date and Time;
- Individuals performing the activity; and
- Weather conditions.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of sampling team members present, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, signed, and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, which is initialed and dated by the sampler. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station will be recorded along with the sample identification number. The number of the photographs taken of the station, if any, will also be noted. Equipment used to make measurements will be identified along with the date of calibration.

Samples will be collected in accordance with the sampling procedures documented in the FSAP (Section 4.0, Pages 4-1 to 4-9). The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, and volume and number of containers used. Sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive a separate sample identification number, will be noted under sample description. General field sampling responsibilities and protocols include the following.

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. Samples will not be left unattended at any time during the sampling activities. It is anticipated that samples will be sent to the lab daily. Should the samples not be sent to the lab at the end of the day, they will be secured by returning the samples to the AMEC Greenville office, locking them in the office, and retrieving the samples from the office the next day for shipment.
- Bottles will be identified by use of sample tags with sample numbers, sampling locations, date/time of collection, sample matrix (soil, sediment, water, groundwater, etc.) and type of analysis.
- A properly completed chain-of-custody form accompanies samples. The sample numbers and identifications will be listed on the chain of custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record.

The chain-of-custody documents the transfer of custody of samples from the sampler to another person, to the laboratory, or to/from a secure storage area.

- Water, groundwater, sediment, and soil samples will be properly packaged on wet ice at 4°C, but protected from intrusion of meltwater from the ice. A separate, signed custody record will be enclosed in and secured to the inside top of each sample box or cooler. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.
- The chain-of-custody record identifying the contents will accompany all shipments. The original record and a second copy will accompany the shipment. A third copy will be retained by the sampler and placed in the project files. A copy of the laboratory's Chain-of-Custody form is included in the laboratory's QAM provided in Appendix A.
- If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact. However, the bills and return receipts for shipping will be included as part of the chain-of-custody documentation.
- Samples will be transported to the laboratory in sufficient time to insure that holding times are not exceeded prior to analysis. Maximum holding times from sample collection to extraction and/or analysis for each sample type are documented in Table B-2.

Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and login, sample storage and numbering, tracking during sample preparation and analysis, and storage of data is described in the laboratory's QA Manual (Appendix A-1) and will be evaluated by AMEC to document appropriate custody and integrity of the submitted samples.

Final Evidence Files

The final evidence file will include at a minimum:

- Field logbooks;
- Field data and data deliverables;
- Photographs;
- Drawings;
- Soil boring logs;

- Monitoring well installation diagrams
- Laboratory data deliverables;
- Data validation reports;
- Data assessment reports;
- Progress reports, QA reports, interim project reports, etc.; and
- Custody documentation (tags, forms, air bills, etc.).

B4 ANALYTICAL METHODS

The following sections outline the analytical methods used for this project. These methods were selected to meet the overall project DQOs.

Field Screening Analysis

Color Tec Method General Information

The Color Tec (CT) Method is a qualitative method that will be used to field-screen pore water and surface water samples. The CT Method was developed by Ecology and Environment, Inc. for determining total chlorinated ethene concentrations in the headspace above soil and groundwater samples. The method employs colorimetric gas detector tubes to analyze the headspace gases. Each colorimetric tube contains a catalyst that decomposes the chlorinated ethene, releasing hydrogen chloride, which discolors the reagent (4-phenylazodiphenylamine) in the tube. Any color change within the detector tube indicates the presence of chlorinated ethenes. The detector tubes are constructed of glass and printed with calibration scales to facilitate measurement of the linear extent of the reaction within the tube. Tubes are provided for a variety of concentration ranges. The lowest concentration tube is used initially to screen the sample. When a positive result is observed, the concentration level is obtained by matching the linear extent of the discolored reagent inside the tube to the calibration scale printed on the outside of the tube. If the calibrated range of the tube is exceeded by the reaction, a tube with a higher concentration range is used to screen a duplicate sample. This procedure is repeated until the approximate concentration is determined.

The practical quantitation limit for tetrachloroethene (perchloroethylene, or PCE) is five to ten parts per billion (ppb) as measured in the headspace. This method does not employ Henry's Constant or other partitioning methods to back calculate the actual concentrations of soil or water samples. Samples containing only trans-1,2-dichloroethene, 1,1-dichloroethene, or vinyl chloride are generally not detectable with Color Tec at concentrations below 25 micrograms per liter (µg/l).

Other compounds, including bromine, free chlorine, and hydrogen chloride can also indicate a positive reaction within the detector tube. The colorimetric detector tubes are manufactured to detect specific alkenes. However, if there are other chlorinated ethenes present in a sample, the identification of a specific chlorinated compound is not possible using the Color Tec method.

Spent Color Tec tubes and the liquid/solids in the vials are containerized with other investigative derived waste generated during field activities.

Color Tec with Water Samples

Fill a 40 milliliter (ml) volatile organic analysis (VOA) vial to approximately 60% of the volume of the vial and cap. Heat the sample and the detection tube in a water bath with a temperature of 100 to 110º Fahrenheit (F). When heated, the vial is shaken vigorously for 20 seconds. One end of the detection tube is broken and attached to a hand pump. The other end of the tube is broken and attached to a small extraction needle. The septum of the vial is penetrated with the extraction needle. A larger purge needle is used to penetrate the septum of the vial and the endpoint of the needle is positioned near the bottom of the vial. This serves to bring air into the vial. One stroke is pulled on the hand pump and the change in color of the tube is observed. The concentration reached by the change in color is recorded. For QC purposes, a minimum of two vials will be collected for each sample and each sample will be screened to verify the results.

Other Field Measurements

Field measurements such as pH, temperature, specific conductance, oxidation-reduction potential (ORP), dissolved oxygen (DO), and FID or PID readings will be collected for informational purposes using appropriately calibrated meters. Field monitoring equipment will be calibrated in accordance with manufacturer instructions daily, at the beginning of each day, and a Field Instrumentation Calibration Record will be completed. The instrumentation will also be checked against the calibration throughout the day to verify that the instrumentation is still providing accurate results.

Laboratory Analysis

Laboratory analysis will be provided by a laboratory certified by the SCDHEC as well as accredited by the National Environmental Laboratory Accreditation Program (NELAP) for solid and aqueous environmental samples. Analytical procedures for this project will be performed in accordance with laboratory QAM and laboratory SOPs, which are based on USEPA SW-846 (USEPA, 1997 with updates) testing procedures. Method validation information and SOPs are provided in the laboratory QAM. The laboratory's QAM is included as Appendix A-1.

Tables A-2 and A-3 provide the method performance criteria for soil and water samples, respectively. **Tables A-4 through A-6** provides the required reporting limits (RL) for each analysis and associated regulatory standards along with MCLs, Tap Water RSLs, Residential and Industrial RSLs, soil screening levels (SSLs) and South Carolina Water Quality Criteria (WQC). Field QA/QC method performance criteria are presented on **Table B-2**. **Table B-3** presents the sample analysis, container preservation and holding time requirements for this project. As shown in **Tables A-4 through A-6**, COC RLs for this project are generally lower than regulatory standards; therefore, laboratory results may be used to:

- enhance the Site conceptual model;
- further characterize the nature and distribution of contaminants in soil, groundwater, surface water, and sediment; and
- complete human health risk assessments.

The standard laboratory turnaround time (10 business days) will be utilized for reporting of laboratory analytical results.

Disposal of laboratory samples is addressed in the laboratory's QAM.

Corrective Actions

Corrective action will be initiated upon identification of problems either through systems or by standard QC data review. Essential steps in the corrective action system are:

- Identifying and defining the problem;
- Assigning responsibility for investigating the problem;
- Investigating and determining the cause of the problem;
- Determining a corrective action to eliminate the problem;
- Assigning and accepting responsibility for implementing the corrective action;
- Implementing corrective action and evaluating its effectiveness; and
- Verifying that the corrective action has eliminated the problem.

The QAO will verify that these steps are taken and that the problem that led to the corrective action has been resolved. The QAO may issue a Stop Work Order if appropriate corrective actions are not taken and the non-conformance is considered significant. Prior to issuing a Stop Work Order, the QAO, AMEC PM and RBTC PM will attempt to resolve outstanding non-conformances.

Field Corrective Action

Project members who know or suspect that an activity is not being performed in accordance with those requirements must identify project tasks or items that do not conform to the QA/QC requirements based on field procedures. The QAO and AMEC PM will be informed of such defects and will act in a timely manner to verify if corrective action is necessary.

If errors in field procedures are found during the observation and/or review of field activities by the QAO, corrective action will be initiated. The QAO will use the protocols outlined to correct the nonconforming activity to meet specified QA/QC requirements. The QAO, in conjunction with the AMEC PM, will review the activity to identify the source of the problem and develop a plan to correct the nonconforming items. Corrective actions for field sampling and testing problems will be developed with assistance from the field team as follows:

- No additional work dependent on the nonconforming activity will be performed until the problem is corrected. The AMEC PM will have the ultimate responsibility for implementation of corrective actions; and
- The QAO will be notified when corrective actions are complete and will then conduct a follow-up audit.

If the problem(s) has been corrected to the satisfaction of the QAO, the activity may resume. **Table B-4** presents a summary of the field corrective actions. Field problems, the applied corrective action, and the results of the corrective action are logged in the field logbooks. A copy of the appropriate pages of the field logbook will be submitted to the PM to be included in the validation process.

Laboratory Corrective Action

The need for laboratory corrective action originates when an inadequacy is found in the method of analysis (e.g., inappropriate calibration) or a determinate error occurs (e.g., calibration error due to standards failure; quality control failures; loss of sample due to exceedances of hold times, temperature, or actual loss). Failures of the first kind are precluded by the laboratory and regulator/client audits that evaluate analytical SOPs. The analytical SOPs incorporate mechanisms to detect the existence of determinate errors and specify the procedures to correct them. Depending on the nature of the corrective action, it is classified as one of two types, immediate or long-term. Immediate corrective actions are the correction of procedures or the repair of instrumentation that is working improperly. Long-term corrective actions eliminate analytical problems by correcting systematic errors.

Response

Many times the source of a nonsystematic problem is obvious to the analyst and can be corrected immediately. Immediate corrective action routinely made by laboratory analysts should be documented as normal operating procedures in instrument logbooks. The supervisor and analyst should compile a list of commonly encountered problems and the appropriate routine corrective actions (in addition to manufacturer's troubleshooting guides). The operations manager and QA/QC coordinator are responsible for approving corrective actions.

For calibration failures, corrective action consists of analyzing standards to establish a new initial calibration or continuing calibration. In cases where MS/MSD criteria fail but laboratory control sample (LCS) criteria are within control limits, corrective action consists of qualification of the sample. If internal standards or surrogates fail criteria, the sample is re-extracted or reanalyzed and both analyses are reported. Method/prep blanks and associated samples are re-extracted and analyzed if concentrations greater than the RL are detected in the blank sample with the exception of common laboratory contaminants (e.g., acetone, 2-butanone, etc.) which must not exceed three times the RL. A LCS sample with failures of the target analytes is re-extracted and reanalyzed along with its associated samples. Data qualification flags are added to those analytes affected by out of control QC data or non-conformance to sampling, handling, or shipping requirements.

Reestablishment of Control

Corrective action is not complete until the problem has been effectively and permanently solved. Follow-up action to ensure that the problem remains corrected is a vitally important step in the corrective action procedure. Once a problem has been technically defined, the operations manager and the QA/QC coordinator discuss the problem and jointly take the following steps:

- Determine that specific corrective action is needed to eliminate the problem and assign responsibility for investigating, implementing, and documenting the situation.
- Set a time schedule for determining the required action;
- Assign responsibility and time schedule to implement the desired action;
- Establish desired effectiveness of the corrective action and implement the correction; and
- Verify that the corrective action has eliminated the problem and document the incident for review and lessons learned.

Documentation

To provide a complete record of QC activities, QC problems and corrective actions applied must be documented. Historical records assist laboratory management in identifying long-term corrective actions, such as personnel training, replacement of instrumentation, and improvement of sampling procedure. A corrective action requires defined responsibilities for scheduling, performing, documenting, and assuring the effectiveness of the action. A deficient incident report form is used to document the above steps and aid in written communication between the analyst and lab management. In all cases, documentation includes: the problem, the corrective action, and the results of the corrective action.

B5 QUALITY CONTROL REQUIREMENTS

A qualified AMEC Principal Engineer or Scientist will review documents involving engineering or scientific evaluation, interpretation, or judgment. A qualified Principal Engineer or Scientist is one who has suitable experience with the techniques employed, conditions evaluated, and technologies involved and is authorized by corporate policy to practice in the discipline covered.

The quality control procedures specified in the current SW-846 methodologies and specified USEPA methods will be followed in the laboratory and the field.

In case of QC failure, the sample will be reanalyzed. In the event additional sample is not available or cannot be located, the laboratory or field personnel must notify the PM within 24 hours. The laboratory is responsible for corrective action related to laboratory analyses and the FOL is responsible for corrective action related to field sampling.

Field Quality Control

Field sampling procedures call for preparation and submittal of four types of QC samples.

- Trip blanks – Prepared by the analyzing laboratory using distilled or de-ionized water that is analyte free and which is shipped with the other sample bottles to the field and then is returned to the analyzing laboratory with the other samples for analysis to check sample contamination from on-site sampling conditions. The trip blanks will not be separated from other samples. They must be packaged with the environmental samples

collected during the sampling event. One trip blank will be included with each sample cooler that contains samples for VOC analysis. The trip blank will be analyzed for VOCs.

- Rinsate blanks – Prepared in the field to demonstrate that a sampling device (e.g., auger) has been effectively cleaned. The sampling device will be filled with organic-free, deionized water that will then be poured through the device, transferred to the appropriate sample bottles, preserved, and returned to the laboratory for analysis. One rinsate blank will be collected per non-disposable sampling tool used at the site. The rinsate blank will be analyzed for constituents of concern.
- Field (blind) duplicates – Two sets of samples from a single source will be prepared, labeled with unique sample numbers, and submitted to the laboratory without cross referencing data and without identification as duplicates on the parameter request sheet. One blind duplicate will be collected for every 20 environmental samples collected for each matrix type.
- Field blank – A water blank, using water provided by the laboratory, will be prepared in the field once per sampling day. An additional field blank will be collected as a duplicate or if weather conditions notably change during a day of sampling.

Field QA/QC method performance criteria are presented on **Table B-2**.

In addition to the above QC samples, the following QC will be conducted during the field activities:

- As a QC check for the Color Tec PCE tubes, one tube from each lot of tubes will be analyzed against a known PCE standard to verify that the lot is working correctly.
- Thermometers and temperature probes will be checked prior to the beginning of the study and annually against a NIST traceable thermometer at the approximate temperature at which the thermometer will be used. The thermometer readings must agree within 1°.
- Water quality meters and turbidity meters will be calibrated prior to first use in the field and checked throughout the day to verify that the instruments work correctly. Calibration results will be maintained in the field logbook.

Laboratory Quality Control

To obtain data on the precision and bias/accuracy, the analytical laboratory will analyze the QC samples as specified in Section A7 and **Table A-2** and **Table A-3**. The control limits and corrective actions for each parameter are specified in each analytical method. The laboratory will comply with the QC procedures outlined in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. Corrective action procedures used by the laboratory are discussed in the laboratory's QAM (**Appendix A-1**). Precision and accuracy will vary with the analytical method and laboratory procedures. The laboratory will make precision and accuracy statements available upon request. The laboratory will

prepare a quality assurance report evaluating the QC measurements listed above. Formulas for calculating QC statistics are provided in Section A7.

For inorganic analyses of soil and water, the analytical methods require analyses of the following QC samples.

- Calibration verification following instrument calibration and once every tenth sample thereafter through the working day.
- Laboratory blank verification at instrument calibration and once every tenth sample thereafter through the working day to check instrument drift.
- Method blank analysis at a rate of one per batch of samples or one per 20 samples of a single matrix, whichever is more frequent, to determine contamination levels during preparation.
- Matrix spike/matrix spike duplicate (MS/MSD) analyses at a rate of one per batch of samples for each matrix type (e.g., soil, water) and concentration level (e.g., low, medium) or one in 20 samples, whichever is more frequent. The MS/MSDs are used to check for the ability to accurately and precisely recover compounds of interest from the matrix.

The results of analyses of these QC samples will be used as independent, external checks on laboratory and field contamination.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Laboratory and field instrumentation will be tested daily prior to use. All equipment will be routinely inspected and maintained according to service and instruction manuals. All instruments and equipment will be tested, inspected, and maintained according to the manufacturer's guidelines and recommendations. Project personnel that have been properly trained in these procedures will operate the instruments. Maintenance documentation for the equipment will be kept on file and will be made available upon request.

All disposable sampling equipment will be used one time only, then properly containerized and disposed. Reusable sampling and investigative equipment will be decontaminated prior to first use, between uses, and at the end of the investigation. Field decontamination methods are provided on page B-15 and in the FSAP (Section 7.0, Page 7-1).

The preventative maintenance procedures for field and laboratory equipment are presented below.

Field Equipment

Field equipment utilized during the investigation will be rented from AMEC's Field Operations Services (FOS) located in Portland, Maine. AMEC's FOS maintains field monitoring and analytical equipment in accordance with the manufacturers' recommended schedules and procedures. Maintenance activities will be documented by FOS personnel. Calibration of field equipment will be performed by FOS personnel prior to shipment to AMEC's Greenville, South Carolina office. Calibrating equipment will also be routinely recalibrated and documented. At a minimum, field calibration will be performed by AMEC

field personnel at the start of the day and checked throughout the day. Routine inspection of equipment is intended to identify problems requiring maintenance before they cause a major disruption of the field monitoring or analytical activities or adversely affect the validity and precision of the data being measured. AMEC's FOL will have sufficient spare parts to allow routine maintenance of the instrumentation. **Table B-5** identifies all field equipment needing periodic maintenance, the schedule for maintenance, the responsible party, and the availability and location of spare parts. Testing criteria for each field instrument to insure the equipment is performing properly is identified on **Table B-6**. **Table B-6** also indicates how deficiencies, if found, will be resolved, re-inspections performed, and effectiveness of corrective action determined and documented, including the personnel responsible.

Laboratory Equipment

The laboratory will maintain a full-service contract with the laboratory equipment manufacturers in order to minimize downtime of the analytical systems. A service engineer will perform necessary preventive maintenance. In the event that analytical equipment used in this study is unable to perform the necessary analyses, appropriate secondary equipment owned by the laboratory will be reconfigured and dedicated to complete the scheduled analytical laboratory work. A supply of spare parts will be maintained to minimize downtime.

Each analyst is responsible for conducting a daily inspection of critical systems on instruments under their charge. Inspections include vacuum lines and pumps for Gas Chromatography/Mass Spectrometry (GC/MS), automatic injection systems, controlled reagent-feed motors, temperature-controlled ovens in GCs, capillary columns, detectors and support systems, gas control system for Atomic Absorption (AA)'s, and many others. Wear-dependent items such as septa on GC injection systems are to be replaced as needed. The performance of instruments is to be checked against known standards at the beginning of each working day or shift. Failure to achieve proper performance indicates a system problem, which will be dealt with by laboratory personnel or by the manufacturer's service representative.

In addition, laboratory personnel or the manufacturer's service representative will service working systems according to a fixed schedule. A record of service and repairs, whether accomplished by laboratory personnel or by the manufacturer's service representative, will be maintained in a logbook kept with each instrument. **Table B-7** identifies laboratory equipment needing periodic maintenance, the schedule for maintenance, the responsible party, and the availability and location of spare parts. Testing criteria for each laboratory instrument to insure the equipment is performing properly is identified on **Table B-8**. **Table B-8** also indicates how deficiencies, if found, will be resolved, re-inspections performed, and effectiveness of corrective action determined and documented, including the personnel responsible.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

The following sections discuss calibration procedures and frequency. Availability and types of standards are also discussed.

Field Instrument Calibration

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. **Table B-9** identifies the equipment, tools, and instruments for field work that should be calibrated, frequency of calibration, how calibrations are performed and documented, test criteria, standards, certified equipment, how deficiencies are resolved and documented, and the personnel responsible for corrective action.

Equipment to be used during the field sampling will be examined to determine its operating condition. This will include review of the maintenance requirements for each instrument. Equipment calibration results from previous equipment use may be reviewed.

Surface water and groundwater may be measured for several field parameters including pH, temperature, specific conductivity, DO, ORP, and turbidity. The meter or meters will be calibrated according to manufacturer's SOP at the beginning of each day of use and the calibration will be checked throughout the day since if the calibration check fails, all data preceding the check will be deemed invalid. The manufacturer specifies the calibration procedures for the instrument. Calibration results will be documented by including the following:

- Date calibrated;
- Person who calibrated the instrument;
- The instrument number (serial number or other identification number);
- The results of the calibration; and
- Identification of the calibration standard (source, type, concentration).

If the instrumentation cannot be calibrated, the FOL will request a replacement instrument from FOS. Field activities that require the use of the calibrated instrument will be delayed until a new instrument is obtained and calibrated.

Field logbooks and Field Instrumentation Calibration FDRs will be used to record calibration dates, results, statistics, and the resulting data measurements. These logbooks and records will also include maintenance and repair reports. Entries will be signed and dated by the personnel performing the required action.

Laboratory Calibration Standards

Calibration standards will be traceable to the National Institute for Standards and Technology (NIST) or American Association for Laboratory Accreditation (A2LA), whenever such standards are available. Commercial sources of standards and reagents are to be checked for purity, and approved prior to their use in analysis.

Standards prepared for use throughout the laboratory will be uniquely identified and will be entered in a bound standard notebook with information regarding the preparation of that standard (i.e., date, technician, name of each compound and amount used, final volume, and solvent used). Standard containers will be labeled so that the standard is traceable to the standard's identification, lot number, manufacturer, and date.

The instrument response obtained for each compound in a newly prepared standard will be compared to the response obtained from the previous standard. The two standards must pass calibration verification criteria (for all but a few compounds recognized as being chromatographably atypical) before the new standard may be used. The new standard may not be used until the discrepancy has been resolved. The working lifetime of standard preparations is dependent upon the compound types comprising the standards.

Chemical Analysis Calibration

Instruments will be calibrated before being put into service and will be recalibrated at regularly specified intervals consistent with the manufacturer's recommendations. Instrument response is subjected to checks between the regular recalibrations. The nature and frequency of such checks are dictated by the standard operating procedures practiced by the analytical laboratory. The analytical laboratory will maintain adequate records of calibrations, recalibrations, and in service checks of instruments. The schedule of checks depends on the experience of the laboratory's maintenance needs. Calibrations will be traceable to primary standards of measurement. Where the concept of traceability of measurements to primary standards is not applicable, the laboratory will provide satisfactory evidence of correlation or accuracy of test results.

Analysts, assistant managers, lab managers, and QA staff will inspect calibration data for completeness and validity. Forms will be checked for arithmetic and procedural errors. Recurring errors, either caused by individual operators or by ambiguously worded instructions, will be brought to the attention of the department senior laboratory staff or laboratory management for corrective action. Calibrations will meet criteria as specified by the applicable methods.

Table B-10 identifies the instruments for laboratory work that should be calibrated, frequency of calibration, how calibrations are performed and documented, test criteria, standards, certified equipment, how deficiencies are resolved and documented, and the personnel responsible for corrective action.

B8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Two techniques will be utilized to document that supplies and consumables used for analysis are of acceptable quality. The first technique involves the supply vendor stockpiling a specific lot number of a consumable product (such as solvents or reagents) for use by the laboratory. The quality of a reagent from one production lot is usually consistent. The second technique involves quality verification of newly obtained supplies by analysis of blanks and/or control samples to verify consistency between the new and old supply of material. The laboratory will be proactive in verifying the quality of new reagents prior to the consumption of the existing supply to facilitate an acceptable transition to the new supply. **Table B-11** presents the supplies and consumables for field and laboratory, vendors, acceptance criteria, handling/storage/retrieval conditions, and the personnel responsible.

B9 DATA ACQUISTITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

Non-direct measurements anticipated to be used for the project include previous environmental reports prepared by AMEC, published literature, and computer models.

Previous environmental reports prepared by AMEC, including text, figures and tables were reviewed by at least two people for acceptability. The reports are signed by two people, one of whom is the technical lead for the project (Principal), evidencing the review process. Tables and figures review for acceptability are evidenced by the "Prepared By" and "Checked By" initials on the figures and tables. Laboratory analytical data used in the preparation of tables and figures was also reviewed for acceptability by the technical lead for the project. Copies of AMEC previous environmental reports are archived in the project file in AMEC's Greenville, South Carolina office.

Published literature will include information on physiography, climate, geology, hydrogeology, meteorology, demographics, land use, and endangered species in South Carolina. The published literature will be used in the evaluation of fate and transport of contaminants and potentially affected receptors. The published literature will be considered acceptable if it is obtained from governmental web sites or peer-reviewed journals. Due to advancements in information technology, access to this type of information should be readily available and no key resources or support facilities are necessary. Copies of published information obtained and used for the project will be archived in the project file in AMEC's Greenville, South Carolina office. Cited literature is presented in the References section of this document.

Computer models may be utilized to assist in the interpretation of fate and transport of contaminants. The computer model(s) will be selected based on their intended use and will be obtained from commercial vendors. Any computer software utilized by AMEC undergoes internal QA review and calibration checks prior to use. Commercial computer models are readily available and no key resources or support facilities are necessary. Computer models, if used, will be archived in the project directory on AMEC's computer server located in the Greenville, South Carolina office. The results of AMEC's internal QA review and calibration checks will be archived in the project file.

B10 DATA MANAGEMENT

Hardware and software used in the data management process has been demonstrated to be acceptable and is the current norm for the environmental consulting and laboratory analytical businesses.

Field Data

Data collected during field activities will be entered into field logbooks and FDRs. The field logbooks and FDRs will be in the custody of AMEC personnel while in the field. Following completion of the field activities, the entries in the field logbook and FDRs are reviewed by the AMEC PM and initialed as to acceptability. Any corrections to the field logbook or FDRs will be made by striking out the incorrect entry with a single line and initialed by the personnel making the correction. Following review, the pages of the field logbook and FDRs are scanned into a PDF and archived in the project directory in the Centric project server. The Centric project server is backed up weekly to a tape. Access to the Centric project server is restricted and only the PM and FOL will have access or be

able to save data to the project file on the Centric project server. In between field activities, the field logbook and FDRs are stored in the project file located in the AMEC Greenville, South Carolina office. The project file is kept in the AMEC Greenville, South Carolina office for five years at which time it is moved to an off-site secured storage facility. Electronic data files are maintained indefinitely.

Sampling Data

Information related to field sampling activities is entered into a chain-of-custody form by AMEC personnel. The chain-of-custody form and samples will remain in the custody of AMEC personnel at all times until the chain-of-custody form and samples are relinquished to the laboratory. A copy of the signed chain-of-custody form will be retained by AMEC to compare to the final chain-of-custody form provided by the laboratory. The copy of the chain-of-custody form will be archived in the project file in AMEC's Greenville, South Carolina and a PDF version of the chain-of-custody form will be archived in the project file on the Greenville Centric project server. The project file is kept in the AMEC Greenville, South Carolina office for five years at which time it is moved to an off-site secured storage facility. Electronic data files are maintained indefinitely.

Laboratory Data

The laboratory has defined protocols for receiving samples and for the "logging in" process as documented in the laboratory's QAM (Appendix A-1). These protocols provide information to the analysts regarding the requested analysis, holding times, types of preservation, matrices, etc. Upon receipt, each sample is identified by a laboratory-issued project number and a unique individual sample number. Properly followed, the preceding procedures provide court defensible documentation related to sample release to the lab, proper preservation and handling, and traceability throughout the analytical and reporting process.

Samples usually arrive at the laboratory in one of three ways: 1) delivered by carrier (UPS, Federal Express, and US Mail), 2) delivered by courier, or 3) delivered by client personnel. In all cases, a chain-of-custody must accompany the samples. This document, supplied by the laboratory to clients, is designed to provide to the laboratory all the necessary information about the client, samples, and which analyses are required. In addition, this document provides evidentiary information indicating who had the samples in their possession at any time and when possession was changed.

Once samples have been relinquished to the laboratory, they are checked for condition including the type(s) of preservation employed (temperature, pH, etc.), correctness of containers, and if the chain-of-custody has been properly completed and signed.

If the chain-of-custody matches the samples it represents, the sample custodian, through the Laboratory Information Management System (LIMS), will issue individual numbers for each sample received. All samples are properly logged into the computer with all pertinent information, including comments about improper preservation or holding times. This information is compiled into a spreadsheet called the "daily" and the information is distributed to the analysts. A folder is prepared with a cover sheet that gives the project number and lists the analyses needed. All information pertaining to the project is placed inside the folder including the chain-of-custody, client contact information, and any special documentation.

After all sample information is logged into the computer, a printout of the entered data is made. A second individual must verify the accuracy of the sample information entered. If the log-in, chain-of-custody, and all sample information are approved, the checking individual initials the work and the project folder is given to the project manager.

Before placement in a storage area, samples must be checked for integrity. If any bottles are broken or have leaked, the client will immediately be contacted. It may be necessary to resample for the incomplete tests. Samples are checked against the chain-of-custody for accuracy and discrepancies. Custody seals must be intact if used. If a discrepancy is found, the variance is noted on the sample receipt checklist and the client is contacted to clarify the problem. Samples are checked for holding time. Holding times begin the moment the sample is taken, not when it is received. The results of all observations are noted on a Sample Receipt Check List during the logging in procedure.

Samples are then placed in the sample holding area, either in the appropriate cooler or on the correct shelf. If the project requires a continuous chain-of-custody, they must be logged out of the area by the analyst and logged back in when the analysis is completed using the logbook provided. Sample bottles are segregated according to the required analysis. Samples to be analyzed for volatile organics are placed in a separate cooler/refrigerator from semi-volatile organics or inorganics because of the high probability of cross-contamination from inorganic and waste samples. Samples for metals analysis do not require cooling and may be placed on the shelf at room temperature.

Data resulting from laboratory analysis will be consistent with the appropriate methods and equations stated in the procedure. Individual laboratory supervisors will review data before forwarding it to the data management supervisor. The laboratory QA Manager will review final reports for error or deviations before release. Final reports will include the Quality Control Summary data required to perform data assessment. Procedures used for analyses will be compared with the reference methods. Discrepancies or deviations will be noted and explained.

The data generated during the sample collection and analysis will be centralized into one project file including information about the instrument conditions. The data management system allows review by project personnel.

The laboratory must submit an electronic data deliverable (EDD). AMEC's data manager uploads the data into a temporary database where the EDD undergoes a review process. Any errors that cannot be resolved at AMEC are communicated to the laboratory. The laboratory may be required to submit a corrected EDD. Once an EDD is processed without errors, the data manager uploads the data into a permanent database specific to the project. All outputs from the database are checked for accuracy by another person other than the person who produced the output. Both persons initial and date the output deliverable. The data manager maintains a record of any data transactions and an electronic copy of all outputs. Only the data manager has access to the permanent database and any changes and or edits can be performed by the data manager. Edits to the database require a "Change Form" be submitted. "Users" can be set up to access information and download reports specific to a project.

The laboratory will provide deliverables to AMEC in PDF. The PDFs will be archived in the project file on the Greenville Centric project server. Electronic data files are maintained indefinitely.

C ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Assessment of field activities will be performed by the FOL and reviewed by the QAO. Field assessments will ensure that proper field methods are followed and that each sample and associated field analysis is documented completely. At the end of each day of sampling, the FOL will review the work completed during that day. This includes a review to determine that both calibration and QC were acceptable. If field methods were not adequately followed, affected samples will be recollected along with repeated field analysis.

The Laboratory PM will be responsible for ensuring that laboratory activities are performed properly. Laboratory data will be reviewed for accuracy prior to reporting. Any discrepancies will be addressed internally with the Laboratory PM. Significant issues that affect data usability will be discussed directly with the AMEC QAO at the time of discovery. Other QC deviations will be noted as part of the laboratory QA/QC summary in the laboratory report. If necessary, samples will be reanalyzed or recollected and analyzed.

The AMEC QAO and Laboratory PM will be responsible for directing assessments and corrective actions and may issue stop-work orders, if necessary. If a corrective action is implemented, the QAO or Laboratory PM will verify that the corrective action was adequate and was properly documented. Non-conforming items observed by the AMEC FOL during field sampling and/or laboratory testing will be documented and corrected. Non-conformance reports will be completed and retained in the project files.

The AMEC PM will audit the site during the field activities. No proficiency testing is planned for field analyses as they are being collected for informational purposes only. During the audit, the sampling design, equipment, instrumentation, supplies, personnel, training, sampling procedures, chain of custody, sample handling and tracking, data reporting, data handling and management, data tracking and control, and data review procedures will be examined for conformance with the QAPP. The PM will prepare a field assessment audit report(s) to document observations made during these visits. Assessment activities are summarized on **Table C-1**

C2 REPORTS TO MANAGEMENT

This QAPP provides the necessary QA/QC procedures so that site investigations are performed in accordance with acceptable protocols, and that the data generated meet the overall project objectives for precision and accuracy. This QAPP provides traceable sampling and analysis procedures, personnel requirements, chain of custody and documentation requirements, and specific criteria for determining data acceptability. This QAPP also establishes the procedures to address data deficiencies, data reduction and evaluation, and preparation of field investigation reports, which will be produced so that outputs are accurate and technically sound.

A report will be generated or obtained for any field or laboratory performance or system audits performed, including the subcontracted laboratory audits conducted concurrent with sample analyses being performed for this project. Reports will also be submitted when analysis or sample collection problems cause samples to be invalidation. These reports

will be prepared by the QAO for AMEC activities and by the Laboratory Manager for laboratory activities and submitted to the PM and kept as part of the project file. Any major problems encountered will be reported to the PM.

D DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION AND VALIDATION

Data review is the in-house examination to ensure that the data have been recorded, transmitted, and processed correctly. Data verification is the process for evaluating the completeness, correctness, and conformance/compliance of the data against the method, procedural, or contractual specifications. Data validation is an analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the quality of a specific data set relative to the end use.

The criteria that will be used for accepting, qualifying, or rejecting the data are described below.

Field Measurements

Field measurements (temperature, pH, specific conductance, DO, turbidity, and ORP) will be collected for informational purposes only. If the initial calibration and periodic calibration checks indicate that calibration is maintained throughout the day, the field measurements will be accepted. If the field equipment fails a calibration check, the measurements collected from the last acceptable calibration check (or initial calibration if the first calibration check fails) to the failed calibration check will be rejected.

Field QC Samples

Field QC samples will consist of trip blanks, rinsate blanks, field blanks, and duplicates. If no constituents are detected above the PQL in the trip blanks, rinsate blanks, and field blanks, the QC samples will be accepted. If constituents are detected above the PQL in the QC samples and the same constituents are detected in the samples collected in the SDG corresponding to the QC samples, the constituents detected in the SDG samples will be evaluated and flagged as necessary.

For field duplicate samples, if the RPD between the two samples (primary and duplicate) meet the acceptance criteria, then the samples will be accepted. Should the RPD be outside the acceptance criteria, then both samples will be rejected and the sample will be recollected.

Sample Handling

Sample handling consists of sample integrity, sample preservation (temperature and preservatives), and hold times. For sample integrity, if a sample has a single container that is unbroken with no signs of leakage, then the sample will be accepted. If the single container is broken or exhibits signs of leakage, then the sample will be rejected. In the case where a sample has multiple containers for a specific analyte (e.g., VOCs) and only one container is broken or exhibits signs of leakage, then the sample will be accepted if sufficient sample volume remains in the unbroken containers to complete the analysis. If there is insufficient volume, then the sample will be rejected and the sample will be recollected.

If the samples in a SDG are within the acceptance criteria for sample preservation, the samples will be accepted. If the samples in a SDG are outside the acceptance criteria for

sample preservation, the samples will be flagged and qualified for informational purposes only and the samples may be recollected.

Samples analyzed within the applicable hold time will be accepted. If the hold time is exceeded, the samples will be rejected and recollected.

Laboratory Analytical Data

The criteria for accepting, qualifying, or rejecting laboratory data are summarized on **Table D-1**.

D2 DATA VALIDATION AND VERIFICATION METHODS

To ensure that data generated are of appropriate quality, all data will be verified and validated. This is a systematic process for reviewing a body of data against a set of established criteria to provide a specified level of assurance of its validity prior to its intended use. The techniques used will be applied to the body of the data in a systematic and uniform manner. The process of data validation will be close to the origin of the data, independent of data production, and objective in approach. All data, as applicable, will be validated in accordance with USEPA policy, per the DQO process. Any deviations will be documented and provided in the final Verification, Validation and Usability Report.

If verification or validation indicates that samples have been collected and/or analyzed out of compliance with the QAPP, resampling may be required. If data is accepted that deviates from the QAPP, the data will be used for screening purposes only and the data will be annotated as such (see **Section D1**).

If the laboratory is found to have lost certification for any of the performed analysis, the data will be used for informational purposes only and annotated as such. The laboratory will provide a list of data qualifier flags and the definition of each.

Verification of the sample data is done by the laboratory. Verification of the entire project, including the data, is performed by AMEC. A checklist will be completed by AMEC to ensure that the check is thorough not only on the completeness of the data, but adherence to the QAPP. The checklist will include any anomalies in the field notes, the data, or the sample narrative from the laboratory. All deviations from the acceptable criteria and potential impacts affecting the usability of the data shall be documented.

AMEC will validate the analytical and project data supplied to ensure compliance with the formal and/or informal DQOs stated in the QAPP. Validation of the data by AMEC will include a check on the following.

- Completeness of the data;
- Adherence to proper sample preservation, transport, or handling protocols;
- Proper use of sample collection procedures;
- Proper use of QC criteria;
- Documentation of all data;

- Ability to reconstruct all field sampling procedures through documentation and records of such procedures;
- Ability to trace data in the final report to a specific sampling site, date, and time;
- Appropriateness of the data as related to specific DQOs.

Prior to issuing the RI report, the QAO will validate the project data by first reading the report and reviewing the checklists, noting the anomalies listed as well as those seen in the RI report. The QAO will determine that all samples have been collected or a reason was given why a sample was not collected. Chain-of-custody for samples will be examined to ensure that it is properly completed and documented the condition of samples during their preparation, packing, transportation, and analyses. The time the sample was collected until it was received by the laboratory will be checked for consistency and for time travel (meaning the sample was received before it was collected or other inconsistencies). The temperature upon receipt is also checked. The QAO will determine if the laboratory was certified throughout the study. The FOL and laboratory are responsible for reporting and correcting all sample handling procedures that deviate from the specified DQO and/or other project-specific requirements.

The QAO will examine the laboratory reports to make sure that all required analytes are present and were analyzed according to the requirements of the QAPP. The data will be compared to historical data to note changes and anomalies. QC data will be examined for completeness and adherence to the requirements of the QAPP. The examination will include an evaluation to ensure that necessary corrective actions have been taken when the QC does not meet QAPP or method requirements.

Validation will be performed on well construction and boring logs. The records will be reviewed for completeness and anomalies. Certification of the well driller will be checked. Field measurements will be analyzed to ensure that wells are purged in accordance with the QAPP. The disposal manifest will be checked and included in the final RI report.

Field Measurements

Raw data from field measurements and sample collection activities will be appropriately recorded in the field logbook. If the data are to be used in the project reports, they will be reduced or summarized and the method of reduction will be documented in the RI report.

AMEC will perform validation of data obtained from field measurements (pH, specific conductivity, temperature. DO, ORP, and turbidity). Data validity will be determined by checking calibration procedures utilized in the field as appropriate and by comparing the data to previous measurements obtained at the Site. Variations in data that cannot be explained will be assigned a lower level of validity and will be used for limited purposes. The FOL will summarize the data obtained from the field measurements and will include this information in field logbooks.

Laboratory Analysis

The following sections describe the data reduction, validation, and reporting procedures to be performed by the laboratory and AMEC. As part of the laboratory review of the data,

the analyst will verify their calculations. A second analyst will also verify a percentage of the sample data and ensure that there are no transcription and calculation errors.

Data Reduction

The analyst will perform the analysis and enter the data on the parameter bench sheet and corresponding data station(s). Bench sheets contain necessary information to establish sample identity, integrity, calibration evaluation, and analytical observations and results. A bench sheet key is provided to the analyst that specifies the way in which bench are sheets to be filled out (i.e., notation, significant figures, etc.), the data reduction formula, and the QC samples required and their control criteria. QC samples include duplicates, MS, or MSDs, continuing calibration verification samples (CCVs), etc. The use of rounding rules and significant digits for numerical data are in accordance with EPA-600/4-79-019 publication, "Handbook for Analytical Quality Control in Water and Wastewater Laboratories."

The laboratory for the duration of the study will keep raw, preliminary, and final data and instrument readouts (e.g., chromatograms, printed digital readouts, etc.). Ultimately, data will be archived along with other project records.

Data Reporting and Validation

Data will be summarized as they are generated and submitted to the project team. The data will be considered preliminary until completion of review and validation.

AMEC will perform a Level II with ten percent Level III validation on one hundred percent of the data prior to use. Data validation will be conducted on data used in risk assessment and will be performed by AMEC's project chemist, who is an individual separate from the sampling team. A Level I validation will be used for other data that are not used in risk assessment. Data validation will be performed using criteria described in this QAPP and specific analytical methods.

The data review and validation consists of checking samples and QC results to demonstrate that the analyses are within prescribed criteria for precision, accuracy, completeness, sensitivity, selectivity, blank contamination, etc. In addition to tabulated results, instrument readouts (e.g., calibration curves, summary reports, etc.) are checked. No raw data review is anticipated; however, if sample data needs clarification, raw data may be required and will be requested for review from the laboratory.

The Level II review will consist of an evaluation of the routine QA/QC performed by the laboratory. This will include review of the following QA/QC controls:

- Extraction blanks;
- Matrix spike and matrix spike duplicates (MS/MSD);
- Surrogate spikes, if applicable;
- Laboratory control samples (LCS);
- Preparation blanks;
- Sample preservation;
- Holding times;
- Equipment and trip blanks; and

- Field duplicate precision.

Level III validation will consist of a Level II review with the following QA/QC controls:

- Initial and continuing calibration standards;
- Initial and continuing method blanks;
- Internal standard (IS) recoveries (organics);
- Post digestion spikes (PDS) (metals);
- Serial dilutions (metals); and
- Interference check samples (metals).

Validation will also include an examination of the chain-of-custody and the sample case narrative. If data points are qualified, they will receive data qualifiers (see Section D1). The qualifiers will indicate if results are usable as-is, usable as-estimated or unusable (rejected). A case narrative will be generated for each analytical package submitted by the laboratory. This narrative represents a summary on the quality of the data. Standard data qualifiers will be used to classify data as to their conformance to QA/QC requirements. The laboratory data qualifiers to be used in this project are described in **Table D-1**.

The validator will also look at the data as a whole to determine if there are anomalies or inconsistencies within the project data and as compared to historical data. If anomalies are detected, the validator will investigate to determine what is responsible and how variability, errors, and anomalies will affect the data usability.

Any anomalies that do not meet the requirements of the QAPP will be noted in a Verification, Validation and Usability Report. This report will be generated by the QAO and is based on his findings during the validation and how the findings affect the data. **Table D-2** presents the data validation and verification checklist.

D3 RECONCILIATION WITH USER REQUIREMENTS

AMEC shall ensure that the data collected address the needs to evaluate the Site and meet the specific DQOs specified previously. This will be done in conjunction with the data verification and validation. The Usability Report will be part of the Verification, Validation, and Usability report discussed in Section D2. This will document problems and corrective action throughout the project and discuss findings in the data and report that appear to be anomalous, but do not significantly impact the usability of the data as a whole. Because data generated with significant deviations from the requirements of the QAPP will be rejected and because of the nature of the work (biased sampling), all data will have the same expected uncertainties and there will be no limitations on data use. A data usability assessment checklist is presented on **Table D-3**.

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TABLES

TABLE A-1

Sample Media and Parameters
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Sample Matrix	Field Parameters	Laboratory Parameters	Responsible Laboratory
Groundwater	pH Conductivity Temperature Dissolved Oxygen Oxidation-Reduction Potential Turbidity ColorTec	TCL VOCs TCL SVOCs TAL Metals TPH-DRO	AES
Surface Water	pH Conductivity Temperature Dissolved Oxygen Oxidation-Reduction Potential Turbidity ColorTec	TCL VOCs	AES
Soil	None	TCL VOCs TCL SVOCs	AES
Sediment	None	TCL VOCs	AES

Notes:
TCL = Target Compound List
VOCs = Volatile Organic Compounds
SVOCs = Semi-Volatile Organic Compounds
TAL = Target Analyte List
TPH-DRO = Total Petroleum Hydrocarbons-Diesel Range Organics
AES = Analytical Environmental Services, Inc.
Field parameters collected for informational purposes only

Prepared By/Date: PSJ 5/22/12
Checked By/Date: JAH 5/31/12

TABLE A-2

Analytical Precision and Accuracy for Soil Samples
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Analyte	Analytical Method	Reporting Limit (mg/kg)	Laboratory Control Sample (LCS) % Recovery	Matrix Spike Samples % Recovery	Precision Relative Percent Difference (RPD) % RPD
Acetone	5030B with 8260B	0.02	70-130	Not in spike	50
Benzene	5030B with 8260B	0.005	70-130	50-150	50
Bromodichloromethane	5030B with 8260B	0.005	70-130	50-150	50
Bromoform	5030B with 8260B	0.005	70-130	50-150	50
Bromomethane (Methyl bromide)	5030B with 8260B	0.005	70-130	Not in spike	50
2-Butanone (MEK)	5030B with 8260B	0.01	Not in spike	Not in spike	50
Carbon disulfide	5030B with 8260B	0.005	70-130	Not in spike	50
Carbon tetrachloride	5030B with 8260B	0.005	70-130	50-150	50
Chlorobenzene	5030B with 8260B	0.005	70-130	50-150	50
Chloroethane (Ethyl chloride)	5030B with 8260B	0.005	70-130	Not in spike	50
Chloroform	5030B with 8260B	0.005	70-130	50-150	50
Chloromethane (Methyl chloride)	5030B with 8260B	0.005	70-130	Not in spike	50
1,2-Dibromo-3-chloropropane	8011	0.005	70-130	50-150	50
Dibromochloromethane	5030B with 8260B	0.005	70-130	50-150	50
1,2-Dibromoethane (EDB)	8011	0.005	70-130	50-150	50
1,2-Dichlorobenzene	5030B with 8260B	0.005	70-130	50-150	50
1,3-Dichlorobenzene	5030B with 8260B	0.005	70-130	50-150	50
1,4-Dichlorobenzene	5030B with 8260B	0.005	70-130	50-150	50
1,1-Dichloroethane	5030B with 8260B	0.005	70-130	50-150	50
1,2-Dichloroethane	5030B with 8260B	0.005	70-130	50-150	50
1,1-Dichloroethene	5030B with 8260B	0.005	70-130	50-179	50
cis-1,2-Dichloroethene	5030B with 8260B	0.005	70-130	50-150	50
trans-1,2-Dichloroethene	5030B with 8260B	0.005	70-130	50-150	50
1,2-Dichloropropane	5030B with 8260B	0.005	70-130	34-166	50
Ethylbenzene	5030B with 8260B	0.005	70-130	50-150	50
2-Hexanone	5030B with 8260B	0.01	Not in spike	Not in spike	50
Methyl tertiary butyl ether (MTBE)	5030B with 8260B	0.005	70-130	Not in spike	50
4-Methyl-2-pentanone	5030B with 8260B	0.01	Not in spike	Not in spike	50
Methylene chloride	5030B with 8260B	0.005	70-130	50-150	50
Naphthalene	5030B with 8260B	0.005	70-130	50-150	50
Styrene	5030B with 8260B	0.005	70-130	50-150	50
1,1,2,2-Tetrachloroethane	5030B with 8260B	0.005	70-130	50-150	50
Tetrachloroethene	5030B with 8260B	0.005	70-130	50-150	50
Toluene	5030B with 8260B	0.005	70-130	50-154	50
1,1,1-Trichloroethane	5030B with 8260B	0.005	70-130	50-150	50
1,1,2-Trichloroethane	5030B with 8260B	0.005	70-130	50-150	50
Trichloroethene	5030B with 8260B	0.005	70-130	50-150	50
Vinyl chloride	5030B with 8260B	0.01	70-130	4-196	50
Xylenes (total)	5030B with 8260B	0.005	70-130	50-150	50

TABLE A-2

Analytical Precision and Accuracy for Soil Samples
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Analyte	Analytical Method	Reporting Limit (mg/kg)	Laboratory Control Sample (LCS) % Recovery	Matrix Spike Samples % Recovery	Precision Relative Percent Difference (RPD) % RPD
Acenaphthene	8270D	0.330	70-130	50.4-120	50
Acenaphthylene	8270D	0.330	70-130	50-150	50
Anthracene	8270D	0.330	70-130	50-150	50
Benzo(a)anthracene	8270D	0.330	70-130	50-150	50
Benzo(a)pyrene	8270D	0.330	70-130	50-150	50
Benzo(b)fluoranthene	8270D	0.330	70-130	50-150	50
Benzo(g,h,i)perylene	8270D	0.330	Not in spike	Not in spike	50
Benzo(k)fluoranthene	8270D	0.330	Not in spike	Not in spike	50
4-Bromophenyl phenyl ether	8270D	0.330	70-130	50-150	50
Butyl benzyl phthalate	8270D	0.330	Not in spike	Not in spike	50
Carbazole	8270D	0.330	Not in spike	Not in spike	50
4-Chloro-3-methyl phenol	8270D	0.330	50-130	45.1-120	50
4-Chloroaniline	8270D	0.330	Not in spike	Not in spike	50
bis(2-Chloroethoxy)methane	8270D	0.330	70-130	49.2-164.7	50
bis(2-Chloroethyl)ether	8270D	0.330	70-130	42.9-150	50
bis(2-Chloroisopropyl)ether	8270D	0.330	70-130	50-150	50
2-Chloronaphthalene	8270D	0.330	Not in spike	Not in spike	50
2-Chlorophenol	8270D	0.330	70-130	44.8-120	50
4-Chlorophenyl phenyl ether	8270D	0.330	Not in spike	Not in spike	50
Chrysene	8270D	0.330	70-130	50-150	50
Di-n-butyl phthalate	8270D	0.330	70-130	8.4-150	50
Di-n-octylphthalate	8270D	0.330	70-130	18.6-150	50
Dibenzo(a,h)anthracene	8270D	0.330	70-130	50-150	50
Dibenzofuran	8270D	0.330	Not in spike	Not in spike	50
1,2-Dichlorobenzene	8270D	0.330	50-130	50-150	50
1,3-Dichlorobenzene	8270D	0.330	50-130	50-150	50
1,4-Dichlorobenzene	8270D	0.330	50-130	50-150	50
3,3'-Dichlorobenzidine	8270D	0.830	40-130	Not in spike	50
2,4-Dichlorophenol	8270D	0.330	70-130	50-150	50
Diethylphthalate	8270D	0.330	70-130	10-150	50
Dimethyl phthalate	8270D	0.330	70-130	10-150	50
2,4-Dimethylphenol	8270D	0.330	70-130	50-150	50
4,6-Dinitro-2-methylphenol	8270D	0.830	Not in spike	Not in spike	50
2,4-Dinitrophenol	8270D	0.830	Not in spike	Not in spike	50
2,4-Dinitrotoluene	8270D	0.330	70-130	40.3-120	50
2,6-Dinitrotoluene	8270D	0.330	70-130	50-150	50
bis(2-Ethylhexyl)phthalate	8270D	0.330	70-130	28.9-150	50
Fluoranthene	8270D	0.330	70-130	50-150	50
Fluorene	8270D	0.330	70-130	50-150	50
Hexachlorobenzene	8270D	0.330	70-130	7.8-150	50
Hexachlorobutadiene	8270D	0.330	70-130	37.8-150	50
Hexachlorocyclopentadiene	8270D	0.830	Not in spike	Not in spike	50

TABLE A-2

Analytical Precision and Accuracy for Soil Samples
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Analyte	Analytical Method	Reporting Limit (mg/kg)	Laboratory Control Sample (LCS) % Recovery	Matrix Spike Samples % Recovery	Precision Relative Percent Difference (RPD) % RPD
Hexachloroethane	8270D	0.330	Not in spike	Not in spike	50
Indeno(1,2,3-c,d)pyrene	8270D	0.330	Not in spike	Not in spike	50
Isophorone	8270D	0.330	Not in spike	Not in spike	50
2-Methylnaphthalene	8270D	0.330	Not in spike	Not in spike	50
2-Methylphenol	8270D	0.330	70-130	50-150	50
3-Methylphenol	8270D	0.670	70-130	50-150	50
4-Methylphenol	8270D	0.670	70-130	50-150	50
N-Nitrosodi-n-propylamine	8270D	0.330	Not in spike	Not in spike	50
N-Nitrosodiphenylamine	8270D	0.330	40-130	50-150	50
Naphthalene	8270D	0.330	70-130	50-150	50
2-Nitroaniline	8270D	0.330	Not in spike	Not in spike	50
3-Nitroaniline	8270D	0.330	Not in spike	Not in spike	50
4-Nitroaniline	8270D	0.330	Not in spike	Not in spike	50
Nitrobenzene	8270D	0.330	70-130	50-157.6	50
2-Nitrophenol	8270D	0.330	Not in spike	Not in spike	50
4-Nitrophenol	8270D	0.830	Not in spike	Not in spike	50
Pentachlorophenol	8151A-1	0.830	70-130	Not in spike	50
Phenanthrene	8270D	0.330	70-130	Not in spike	50
Phenol	8270D	0.330	Not in spike	Not in spike	50
Pyrene	8270D	0.330	70-130	47.9-115	50
1,2,4-Trichlorobenzene	8270D	0.330	Not in spike	Not in spike	50
2,4,5-Trichlorophenol	8270D	0.330	70-130	50-150	50
2,4,6-Trichlorophenol	8270D	0.330	70-130	50-150	50
TPH-DRO	8015C	6.7	70-130	47.4-128	50
Aluminum	6020A	10	80-120	75-125	50
Antimony	6020A	0.25	80-120	75-125	50
Arsenic	6020A	0.25	80-120	75-125	50
Barium	6020A	1.3	80-120	75-125	50
Beryllium	6020A	0.2	80-120	75-125	50
Cadmium	6020A	0.1	80-120	75-125	50
Calcium	6010C	250	80-120	75-125	50
Chromium	6020A	0.25	80-120	75-125	50
Cobalt	6020A	1.3	80-120	75-125	50
Copper	6020A	0.25	80-120	75-125	50
Iron	6020A	5	80-120	75-125	50
Lead	6020A	0.25	80-120	75-125	50
Magnesium	6010C	250	80-120	75-125	50
Manganese	6020A	0.75	80-120	75-125	50
Mercury	7471B	0.083	80-120	75-125	50
Nickel	6020A	2	80-120	75-125	50
Potassium	6010C	250	80-120	75-125	50
Selenium	6020A	0.25	80-120	75-125	50

TABLE A-2

Analytical Precision and Accuracy for Soil Samples
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Analyte	Analytical Method	Reporting Limit (mg/kg)	Laboratory Control Sample (LCS) % Recovery	Matrix Spike Samples % Recovery	Precision Relative Percent Difference (RPD) % RPD
Silver	6020A	0.25	80-120	75-125	50
Sodium	6010C	250	80-120	75-125	50
Thallium	6020A	0.5	80-120	75-125	50
Vanadium	6010C	2.5	80-120	75-125	50
Zinc	6020A	2.5	80-120	75-125	50

Prepared By/Date: PSJ 5/22/12
Checked By/Date: JAH 5/31/12

TABLE A-3

Analytical Precision and Accuracy for Water Samples
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Analyte	Analytical Method	Reporting Limit		Laboratory Control Sample (LCS) % Recovery	Matrix Spike Samples % Recovery	Precision Relative Percent Difference (RPD) % RPD
		Groundwater (mg/L)	Drinking Water and Receptors (mg/L)			
Acetone	5030B with 8260B	0.02	0.02	Not Spikec	Not Spiked	35
Benzene	5030B with 8260B	0.005	0.005	70-130	50-150	35
Bromodichloromethane	5030B with 8260B	0.005	0.005	70-130	50-150	35
Bromoform	5030B with 8260B	0.005	0.005	70-130	50-150	35
Bromomethane (Methyl bromide)	5030B with 8260B	0.005	0.005	Not Spikec	Not Spiked	35
2-Butanone (MEK)	5030B with 8260B	0.01	0.01	Not Spikec	Not Spiked	35
Carbon disulfide	5030B with 8260B	0.005	0.005	Not Spikec	Not Spiked	35
Carbon tetrachloride	5030B with 8260B	0.005	0.005	70-130	50-150	35
Chlorobenzene	5030B with 8260B	0.005	0.005	70-130	50-150	35
Chloroethane (Ethyl chloride)	5030B with 8260B	0.005	0.005	Not Spikec	Not Spiked	35
Chloroform	5030B with 8260B	0.005	0.005	70-130	50-150	35
Chloromethane (Methyl chloride)	5030B with 8260B	0.005	0.005	Not Spikec	Not Spiked	35
1,2-Dibromo-3-chloropropane	8011	0.00002	0.00002	60-140	47-142	35
Dibromochloromethane	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,2-Dibromoethane (EDB)	8011	0.00002	0.00002	60-140	54.3-138	35
1,2-Dichlorobenzene	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,3-Dichlorobenzene	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,4-Dichlorobenzene	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,1-Dichloroethane	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,2-Dichloroethane	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,1-Dichloroethene	5030B with 8260B	0.005	0.005	60-140	50-179	35
cis-1,2-Dichloroethene	5030B with 8260B	0.005	0.005	70-130	50-150	35
trans-1,2-Dichloroethene	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,2-Dichloropropane	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,3-Dichloropropene	5030 with 8260B	0.005	0.005	70-130	50-150	35
Ethylbenzene	5030B with 8260B	0.005	0.005	70-130	50-150	35
2-Hexanone	5030B with 8260B	0.01	0.01	Not Spikec	Not Spiked	35
Methyl tertiary butyl ether (MTBE)	5030B with 8260B	0.005	0.005	70-130	37.4-152	35
4-Methyl-2-pentanone	5030B with 8260B	0.01	0.01	Not Spikec	Not Spiked	35
Methylene chloride	5030B with 8260B	0.005	0.005	70-130	50-150	35
Naphthalene	5030B with 8260B	0.005	0.005	70-130	50-150	35
Styrene	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,1,2,2-Tetrachloroethane	5030B with 8260B	0.005	0.005	70-130	50-150	35
Tetrachloroethene	5030B with 8260B	0.005	0.005	70-130	50-150	35
Toluene	5030B with 8260B	0.005	0.005	70-130	50-154	35
1,1,1-Trichloroethane	5030B with 8260B	0.005	0.005	70-130	50-150	35
1,1,2-Trichloroethane	5030B with 8260B	0.005	0.005	70-130	50-150	35
Trichloroethene	5030B with 8260B	0.005	0.005	70-130	50-150	35
Vinyl chloride	5030B with 8260B	0.002	0.002	70-130	4-196	35
Xylenes (total)	5030B with 8260B	0.005	0.005	70-130	50-150	35
Acenaphthene	8270D	0.005	0.005	70-130	57.8-120	35
Acenaphthylene	8270D	0.005	0.005	70-130	50-150	35
Anthracene	8270D	0.005	0.005	70-130	50-150	35
Benzo(a)anthracene	8270D	0.005	0.005	70-130	50-150	35
Benzo(a)pyrene	8270D	0.005	0.005	70-130	50-150	35
Benzo(b)fluoranthene	8270D	0.005	0.005	70-130	50-150	35
Benzo(g,h,i)perylene	8270D	0.005	0.005	Not Spikec	Not Spiked	35
Benzo(k)fluoranthene	8270D	0.005	0.005	Not Spikec	Not Spiked	35
4-Bromophenyl phenyl ether	8270D	0.005	0.005	70-130	50-150	35

TABLE A-3

Analytical Precision and Accuracy for Water Samples
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Analyte	Analytical Method	Reporting Limit		Laboratory Control Sample (LCS) % Recovery	Matrix Spike Samples % Recovery	Precision Relative Percent Difference (RPD) % RPD
		Groundwater (mg/L)	Drinking Water and Receptors (mg/L)			
Butyl benzyl phthalate	8270D	0.01	0.01	Not Spikec	Not Spiked	35
Carbazole	8270D	0.005	0.005	Not Spikec	Not Spiked	35
4-Chloro-3-methyl phenol	8270D	0.005	0.005	70-130	49.8-120	35
4-Chloroaniline	8270D	0.005	0.005	Not Spikec	Not Spiked	35
bis(2-Chloroethoxy)methane	8270D	0.005	0.005	70-130	50-150	35
bis(2-Chloroethyl)ether	8270D	0.005	0.005	70-130	42.9-150	35
bis(2-Chloroisopropyl)ether	8270D	0.005	0.005	70-130	50-150	35
2-Chloronaphthalene	8270D	0.005	0.005	Not Spikec	Not Spiked	35
2-Chlorophenol	8270D	0.005	0.005	70-130	56.6-120	35
4-Chlorophenyl phenyl ether	8270D	0.005	0.005	Not Spikec	Not Spiked	35
Chrysene	8270D	0.005	0.005	70-130	50-150	35
Di-n-butyl phthalate	8270D	0.005	0.005	70-130	10-150	35
Di-n-octylphthalate	8270D	0.005	0.005	70-130	18.6-150	35
Dibenzo(a,h)anthracene	8270D	0.005	0.005	70-130	50-150	35
Dibenzofuran	8270D	0.005	0.005	Not Spikec	Not Spiked	35
1,2-Dichlorobenzene	8270D	0.005	0.005	50-130	50-150	35
1,3-Dichlorobenzene	8270D	0.005	0.005	70-130	50-150	35
1,4-Dichlorobenzene	8270D	0.005	0.005	70-130	50-150	35
3,3'-Dichlorobenzidine	8270D	0.025	0.025	40-130	40-150	35
2,4-Dichlorophenol	8270D	0.005	0.005	70-130	50-150	35
Diethylphthalate	8270D	0.005	0.005	70-130	10-150	35
Dimethyl phthalate	8270D	0.005	0.005	70-130	10-150	35
2,4-Dimethylphenol	8270D	0.005	0.005	70-130	41.8-150	35
4,6-Dinitro-2-methylphenol	8270D	0.025	0.025	Not Spikec	Not Spiked	35
2,4-Dinitrophenol	8270D	0.025	0.025	Not Spikec	Not Spiked	35
2,4-Dinitrotoluene	8270D	0.01	0.01	70-130	54.4-120	35
2,6-Dinitrotoluene	8270D	0.01	0.01	70-130	50-150	35
bis(2-Ethylhexyl)phthalate	8270D	0.005	0.005	70-130	28.9-150	35
Fluoranthene	8270D	0.005	0.005	70-130	50-150	35
Fluorene	8270D	0.005	0.005	70-130	50-150	35
Hexachlorobenzene	8270D	0.005	0.005	70-130	7.8-150	35
Hexachlorobutadiene	8270D	0.005	0.005	70-130	37.8-150	35
Hexachlorocyclopentadiene	8270D	0.025	0.025	Not Spikec	Not Spiked	35
Hexachloroethane	8270D	0.005	0.005	Not Spikec	Not Spiked	35
Indeno(1,2,3-c,d)pyrene	8270D	0.005	0.005	Not Spikec	Not Spiked	35
Isophorone	8270D	0.005	0.005	Not Spikec	Not Spiked	35
2-Methylnaphthalene	8270D	0.005	0.005	Not Spikec	Not Spiked	35
2-Methylphenol	8270D	0.005	0.005	70-130	50-150	35
3,4-Methylphenol	8270D	0.01	0.01	70-130	50-150	35
N-Nitrosodi-n-propylamine	8270D	0.005	0.005	Not Spikec	Not Spiked	35
N-Nitrosodiphenylamine	8270D	0.005	0.005	40-130	15-150	35
Naphthalene	8270D	0.005	0.005	70-130	50-150	35
2-Nitroaniline	8270D	0.01	0.01	Not Spikec	Not Spiked	35
3-Nitroaniline	8270D	0.01	0.01	Not Spikec	Not Spiked	35
4-Nitroaniline	8270D	0.01	0.01	Not Spikec	Not Spiked	35
Nitrobenzene	8270D	0.005	0.005	70-130	50-150	35
2-Nitrophenol	8270D	0.01	0.01	Not Spikec	Not Spiked	35
4-Nitrophenol	8270D	0.025	0.025	Not Spikec	Not Spiked	35
Pentachlorophenol	8151A-1	0.0005	0.0005	Not Spikec	Not Spiked	35

TABLE A-3

Analytical Precision and Accuracy for Water Samples
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Analyte	Analytical Method	Reporting Limit		Laboratory Control Sample (LCS) % Recovery	Matrix Spike Samples % Recovery	Precision Relative Percent Difference (RPD) % RPD
		Groundwater (mg/L)	Drinking Water and Receptors (mg/L)			
Phenanthrene	8270D	0.005	0.005	Not Spikec	Not Spiked	35
Phenol	8270D	0.005	0.005	Not Spikec	Not Spiked	35
Pyrene	8270D	0.005	0.005	70-130	57.9-118	35
1,2,4-Trichlorobenzene	8270D	0.005	0.005	Not Spikec	Not Spiked	35
2,4,5-Trichlorophenol	8270D	0.005	0.005	70-130	50-150	35
2,4,6-Trichlorophenol	8270D	0.005	0.005	70-130	50-150	35
TPH-DRO	8015C	0.2	0.2	43.6-115	30.5-121	35
Aluminum	6010B	0.2	0.2	80-120	75-125	35
Antimony	6020A	0.005	0.005	80-120	75-125	35
Arsenic	6020A	0.005	0.005	80-120	75-125	35
Barium	6020A	0.025	0.025	80-120	75-125	35
Beryllium	6020A	0.004	0.004	80-120	75-125	35
Cadmium	6020A	0.002	0.002	80-120	75-125	35
Calcium	6010B	5	5	80-120	75-125	35
Chromium	6020A	0.005	0.005		75-125	35
Cobalt	6010B	0.025	0.025	80-120	75-125	35
Copper	6020A	0.005	0.005	80-120	75-125	35
Iron	6010B	0.1	0.1	80-120	75-125	35
Lead	6020A	0.003	0.003	80-120	75-125	35
Magnesium	6010B	5	5	80-120	75-125	35
Manganese	6010B	0.015	0.015	80-120	75-125	35
Mercury	7471A	0.0001	0.0001	85-115	70-130	35
Nickel	6010B	0.04	0.04	80-120	75-125	35
Potassium	6010B	5	5	80-120	75-125	35
Selenium	6020A	0.005	0.005	80-120	75-125	35
Silver	6020A	0.005	0.005	80-120	75-125	35
Sodium	6010B	5	5	80-120	75-125	35
Thallium	6020A	0.0005	0.0005	80-120	75-125	35
Vanadium	6010B	0.05	0.05	80-120	75-125	35
Zinc	6010B	0.02	0.02	80-120	75-125	35

Prepared By/Date: PSJ 5/22/12
Checked By/Date: JAH 5/31/12

TABLE A-4

Comparison of PQLs and Regulatory Standards for VOCs
Quality Assurance Project Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

VOC Method 8260B			MCL	EPA Tap	EPA RSL		EPA SSL	SC WQC
Parameter	PQL		Primary	Water RSL	Residential	Industrial	(Risk Based)	Human Health
	Aqueous mg/L	Soil mg/kg	mg/L	mg/L	mg/kg	mg/kg	mg/kg	mg/L
Acetone	0.020	0.020		12	61,000	630,000	2.4	
Benzene	0.005	0.005	0.005	**0.00039**	1.1	5.4	**0.0002**	**0.0022**
Bromodichloromethane	0.005	0.005	0.08[1]	**0.00012**	0.27	1.4	**0.000032**	**0.00055**
Bromoform	0.005	0.005	0.08[1]	0.0079	62	220	**0.0021**	**0.0043**
Bromomethane (Methyl bromide)	0.005	0.005		0.007	7.3	32	**0.0018**	0.047
2-Butanone (MEK)	0.010	0.010		4.9	28,000	200,000	1.0	
Carbon disulfide	0.005	0.005		0.72	820	3,700	0.21	
Carbon tetrachloride	0.005	0.005	0.005	**0.00039**	0.61	3.0	**0.00015**	**0.00023**
Chlorobenzene	0.005	0.005	0.1	0.072	290	1,400	0.049	0.13
Chloroethane (Ethyl chloride)	0.005	0.005		21	15,000	61,000	5.9	
Chloroform	0.005	0.005	0.08[1]	**0.00019**	0.29	1.5	**0.000053**	0.0057
Chloromethane (Methyl chloride)	0.005	0.005		0.19	120	500	0.049	
1,2-Dibromo-3-chloropropane[2]	0.00002	0.005	0.0002	**0.00000032**	0.0054	0.069	**0.00000014**	
Dibromochloromethane	0.005	0.005	0.08[1]	0.00015	0.68	3.3	**0.000035**	0.0004
1,2-Dibromoethane (EDB)[2]	0.00002	0.005	0.00005	**0.0000065**	0.034	0.17	**0.0000018**	
1,2-Dichlorobenzene	0.005	0.005	0.6	0.28	1,900	9,800	0.27	0.42
1,3-Dichlorobenzene	0.005	0.005						0.32
1,4-Dichlorobenzene	0.005	0.005	0.075	**0.00042**	2.4	12	**0.00040**	0.063
1,1-Dichloroethane	0.005	0.005		**0.0024**	3.3	17	**0.00068**	
1,2-Dichloroethane	0.005	0.005	0.005	**0.00015**	0.43	2.2	**0.000042**	**0.00038**
1,1-Dichloroethene	0.005	0.005	0.007	0.26	240	1,100	0.093	0.33
cis-1,2-Dichloroethene	0.005	0.005	0.07	0.028	160	2,000	0.0082	
trans-1,2-Dichloroethene	0.005	0.005	0.1	0.086	150	690	0.025	0.14
1,2-Dichloropropane	0.005	0.005	0.005	**0.00038**	0.94	4.7	**0.00013**	**0.0005**
1,3-Dichloropropene	0.005	0.005		**0.00041**	1.7	8.3	**0.00015**	**0.00034**
Ethylbenzene	0.005	0.005	0.7	**0.0013**	5.4	27	**0.0015**	0.53
2-Hexanone	0.010	0.010		0.034	210	1,400	**0.0079**	
Methyl tertiary butyl ether (MTBE)	0.005	0.005		0.012	43	220	**0.0028**	
4-Methyl-2-pentanone	0.010	0.010		1.0	5,300	53,000	0.23	
Methylene chloride	0.005	0.005	0.005	0.0098	56	960	**0.0025**	**0.0046**
Naphthalene	0.005	0.005		**0.00014**	3.6	18	**0.00047**	
Styrene	0.005	0.005	0.1	1.1	6,300	36,000	1.2	
1,1,2,2-Tetrachloroethane	0.005	0.005		**0.000066**	0.56	2.8	**0.000026**	**0.00017**
Tetrachloroethene	0.005	0.005	0.005	0.0097	22	110	**0.0044**	**0.00069**
Toluene	0.005	0.005	1.0	0.86	5,000	45,000	0.59	1.3
1,1,1-Trichloroethane	0.005	0.005	0.2	7.5	8,700	38,000	2.6	
1,1,2-Trichloroethane	0.005	0.005	0.005	**0.00024**	1.1	5.3	**0.000077**	**0.00059**
Trichloroethene	0.005	0.005	0.005	**0.00044**	0.91	6.4	**0.00061**	**0.0025**
Vinyl chloride	0.002	0.010	0.002	**0.000015**	0.06	1.7	**0.0000053**	**0.000025**
Xylenes (total)	0.005	0.005	10	0.19	630	2,700	0.19	

Notes:
VOCs = Volatile Organic Compounds
PQL = Practical Quantitation Limit (this will also serve as the laboratory reporting limit)
mg/kg = milligrams per kilogram
mg/L = milligrams per liter
MCL = Maximum Contaminant Level
EPA = United States Environmental Protection Agency
RSL = EPA Regional Screening Level (May 2012)
SSLs = EPA Soil Screening Levels (Risk Based)
SC WQC = Water Quality Criteria, South Carolina Regulation 61-68, effective 4/25/08
Screening value is less than the PQL **0.001**
Regulatory value is less than PQL **0.001**
Concentrations detected below the respective MCL, RSL, or SSL will be reported as estimated and as such will be flagged with a "J" qualifier
[1] Total trihalomethanes
[2] Analysis by SW-846 Method 8011

Prepared By/Date: PSJ 5/22/12
Checked By/Date: LWC 5/23/12

TABLE A-5

Comparison of PQLs and Regulatory Standards for SVOCs
Quality Assurance Project Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

SVOCs Method 8270D			MCL	EPA Tap	EPA RSL		EPA SSL	SC WQC
Parameter	PQL		Primary	Water RSL	Residential	Industrial	(Risk Based)	Human Health
	Aqueous mg/L	Soil mg/kg	mg/L	mg/L	mg/kg	mg/kg	mg/kg	mg/L
Acenaphthene	0.005	0.330		0.4	3,400	33,000	4.1	0.67
Acenaphthylene	0.005	0.330						
Anthracene	0.005	0.330		1.3	17,000	170,000	42	8.3
Benzo(a)anthracene	0.005	0.330		**0.000029**	**0.15**	2.1	**0.010**	**0.0000038**
Benzo(a)pyrene	0.005	0.330	**0.0002**	**0.0000029**	**0.015**	**0.21**	**0.0035**	**0.0000038**
Benzo(b)fluoranthene	0.005	0.330		**0.000029**	**0.15**	2.1	**0.035**	**0.0000038**
Benzo(g,h,i)perylene	0.005	0.330						
Benzo(k)fluoranthene	0.005	0.330		**0.00029**	1.5	21	0.35	**0.0000038**
4-Bromophenyl phenyl ether	0.005	0.330						
Butyl benzyl phthalate	0.010	0.330		0.014	260	910	0.2	1.5
Carbazole	0.005	0.330						
4-Chloro-3-methyl phenol	0.005	0.330		1.1	6,100	62,000	1.3	
4-Chloroaniline	0.005	0.330		**0.00032**	2.4	8.6	**0.00013**	
bis(2-Chloroethoxy)methane	0.005	0.330		0.047	180	1,800	**0.0011**	
bis(2-Chloroethyl)ether	0.005	0.330		**0.000012**	**0.21**	1.0	**0.0000031**	**0.00003**
bis(2-Chloroisopropyl)ether	0.005	0.330						1.4
2-Chloronaphthalene	0.005	0.330		0.55	6,300	82,000	2.9	1.0
2-Chlorophenol	0.005	0.330		0.071	390	5,100	**0.057**	0.081
4-Chlorophenyl phenyl ether	0.005	0.330						
Chrysene	0.005	0.330		**0.0029**	15	210	1.1	**0.0000038**
Di-n-butyl phthalate	0.005	0.330		0.67	6,100	62,000	1.7	2.0
Di-n-octylphthalate	0.005	0.330						
Dibenzo(a,h)anthracene	0.005	0.330		**0.0000029**	**0.015**	**0.21**	**0.011**	**0.0000038**
Dibenzofuran	0.005	0.330		0.0058	78	1,000	**0.11**	
1,2-Dichlorobenzene	0.005	0.330	0.6	0.28	1,900	9,800	**0.27**	0.42
1,3-Dichlorobenzene	0.005	0.330						0.32
1,4-Dichlorobenzene	0.005	0.330	0.075	**0.00042**	2.4	12	**0.00040**	0.063
3,3'-Dichlorobenzidine	0.025	0.830		**0.00011**	1.1	3.8	**0.00071**	**0.000021**
2,4-Dichlorophenol	0.005	0.330		0.035	180	1,800	**0.041**	0.077
Diethylphthalate	0.005	0.330		11	49,000	490,000	4.7	17
Dimethyl phthalate	0.005	0.330						270
2,4-Dimethylphenol	0.005	0.330		0.27	1,200	12,000	**0.32**	0.38
4,6-Dinitro-2-methylphenol	0.025	0.830		**0.0012**	4.9	49	**0.002**	
2,4-Dinitrophenol	0.025	0.830		0.03	120	1,200	**0.034**	0.069
2,4-Dinitrotoluene	0.010	0.330		**0.0002**	1.6	5.5	**0.00028**	**0.00011**
2,6-Dinitrotoluene	0.010	0.330		0.015	61	620	**0.02**	
bis(2-Ethylhexyl)phthalate	0.005	0.330	0.006	**0.000071**	35	120	**0.017**	**0.0012**
Fluoranthene	0.005	0.330		0.63	2,300	22,000	70	0.13
Fluorene	0.005	0.330		0.22	2,300	22,000	4.0	1.1
Hexachlorobenzene	0.005	0.330	**0.001**	**0.000042**	**0.3**	1.1	**0.00053**	**0.00000028**
Hexachlorobutadiene	0.005	0.330		**0.00026**	6.2	22	**0.0005**	**0.00044**
Hexachlorocyclopentadiene	0.025	0.830	0.05	**0.022**	370	3,700	**0.07**	0.04
Hexachloroethane	0.005	0.330		**0.00079**	12	43	**0.00048**	**0.0014**
Indeno(1,2,3-c,d)pyrene	0.005	0.330		**0.000029**	**0.15**	2.1	**0.12**	**0.0000038**
Isophorone	0.005	0.330		0.067	510	1,800	**0.022**	0.035
2-Methylnaphthalene	0.005	0.330		0.027	230	2,200	**0.14**	
2-Methylphenol	0.005	0.330		0.72	3,100	31,000	0.58	
3-Methylphenol	0.010	0.670		0.72	3,100	31,000	**0.57**	

TABLE A-5

Comparison of PQLs and Regulatory Standards for SVOCs
Quality Assurance Project Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

SVOCs Method 8270D			MCL	EPA Tap	EPA RSL		EPA SSL	SC WQC
Parameter	PQL		Primary	Water RSL	Residential	Industrial	(Risk Based)	Human Health
	Aqueous mg/L	Soil mg/kg	mg/L	mg/L	mg/kg	mg/kg	mg/kg	mg/L
4-Methylphenol	0.010	0.670		1.4	6,100	62,000	1.1	
N-Nitrosodi-n-propylamine	0.005	0.330		**0.0000093**	**0.069**	**0.25**	**0.000007**	**0.000005**
N-Nitrosodiphenylamine	0.005	0.330		0.01	99	350	**0.057**	**0.0033**
Naphthalene	0.005	0.330		**0.00014**	3.6	18	**0.00047**	
2-Nitroaniline	0.010	0.330		0.15	610	6,000	**0.062**	
3-Nitroaniline	0.010	0.330						
4-Nitroaniline	0.010	0.330		**0.0033**	24	86	**0.0014**	
Nitrobenzene	0.005	0.330		**0.00012**	4.8	24	**0.000079**	0.017
2-Nitrophenol	0.010	0.330						
4-Nitrophenol	0.025	0.830						
Pentachlorophenol[1]	0.0005	0.830	0.001	**0.00017**	0.89	2.7	**0.0017**	**0.00027**
Phenanthrene	0.005	0.330						
Phenol	0.005	0.330		4.5	18,000	180,000	2.6	21
Pyrene	0.005	0.330		0.087	1,700	17,000	9.5	0.83
1,2,4-Trichlorobenzene	0.005	0.330	0.07	**0.00099**	22	99	**0.0029**	0.035
2,4,5-Trichlorophenol	0.005	0.330		0.89	6,100	62,000	3.3	
2,4,6-Trichlorophenol	0.005	0.330		**0.0035**	44	160	**0.013**	**0.0014**
TPH-DRO[2]	0.2	6.7	NE	NE	NE	NE	NE	NE

Notes:
PQL = Practical Quantitation Limit (this will also serve as the laboratory reporting limit)
mg/L = milligrams per liter
mg/kg = milligrams per kilogram
Screening value is less than the PQL **0.001**
Regulatory value is less than PQL **0.001**
SSLs = Environmental Protection Agency (EPA) Regional Screening Level Table Soil Screening Levels (Risk Based)
Concentrations detected below the respective MCL, RSL, or SSL will be reported as estimated and as such will be flagged with a "J" qualifier
RSL = EPA Regional Screening Level May 2012
MCL = EPA 2009 Maximum Contaminant Level
SVOC = Semivolatile Organic Compounds
SC WQC = Water Quality Criteria, South Carolina Regulation 61-68, effective 4/25/08
TPH-DRO = Total Petroleum Hydrocarbons-Diesel Range Organics
[1] Analyzed by SW-846 Method 8151A-1

Prepared By/Date: PSJ 5/22/12
Checked By/Date: LWC 5/23/12

TABLE A-6

Comparison of PQLs and Regulatory Standards for TAL Metals
Quality Assurance Project Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Metals Method 6010C/6020A/7470A/7471B	PQL		MCL Primary	EPA Tap Water RSL	EPA RSL		EPA SSL (Risk Based)	SC WQC Human Health
Parameter	Aqueous mg/L	Soil mg/kg	mg/L	mg/L	Residential mg/kg	Industrial mg/kg	mg/kg	mg/L
Aluminum	0.2	10.0		16	77,000	990,000	23,000	
Antimony	0.005	0.250	0.006	0.006	31	410	0.27	0.0056
Arsenic	0.005	0.250	0.01	**0.000045**	0.39	1.6	**0.0013**	
Barium	0.025	1.300	2.0	2.9	15,000	190,000	120	
Beryllium	0.004	0.200	0.004	0.016	160	2,000	13	
Cadmium	0.002	0.100	0.005	0.0069	70	800	0.52	
Calcium	5.0	250.0						
Chromium	0.005	0.250	0.1	**0.000031** (1)	0.29 (1)	5.6 (1)	**0.00059** (1)	
Cobalt	0.025	1.300		**0.0047**	23	300	**0.21**	
Copper	0.005	0.250	1.3	0.62	3,100	41,000	22	1.3
Iron	0.1	5.000		11	55,000	720,000	270	
Lead	0.003	0.250	0.015		400	800		
Magnesium	5.0	250.0						
Manganese	0.015	0.750		0.32	1,800	23,000	21	
Mercury (7470A/7471B)	0.0001	0.083	0.002	0.00063	10	43	**0.033**	**0.00005**
Nickel	0.04	2.000		0.3	1,500	20,000	20	0.61
Potassium	5.0	250.0						
Selenium	0.005	0.250	0.05	0.078	390	5,100	0.40	0.17
Silver	0.005	0.250		0.071	390	5,100	0.6	
Sodium	5.0	250.0						
Thallium	0.0005	0.500	0.002	**0.00016**	0.78	10	**0.11**	**0.00024**
Vanadium	0.05	2.500		0.078	390	5,200	78	
Zinc	0.02	2.500		4.7	23,000	310,000	290	7.4

Notes:
PQL = Practical Quantitation Limit (this will also serve as the laboratory reporting limit)
mg/kg = milligrams per kilogram
mg/L milligrams per liter
Screening value is less than the PQL **0.001**
Regulatory value is less than PQL **0.001**
SSLs = Environmental Protection Agency (EPA) Regional Screening Level Table Soil Screening Levels (Risk Based)
Concentrations detected below the respective MCL, RSL, or SSL will be reported as estimated and as such will be flagged with a "J" qualifier
RSL = EPA Regional Screening Level May 2012
MCL = EPA 2009 Maximum Contaminant Level
TAL = Target Analyte List
SC WQC = Water Quality Criteria, South Carolina Regulation 61-68, effective 4/25/08
(1) Value for hexavalent chromium.

Prepared By/Date: PSJ 5/22/12
Checked By/Date: LWC 5/23/12

TABLE B-1

Field and Quality Control Samples to be Collected/Analytical Parameters
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

	TCL VOCs	TCL SVOCs	TAL Metals	PAHs	TPH DRO
Field Samples					
Surface Soil				3	
Subsurface Soil	50	50			
Sediment	9				
Surface Water	9				
Pore Water	8				
Groundwater	21	6	5		5
QC Samples					
Field Duplicate	8	4	1	1	1
Rinsate Blank	6	2	1	1	1
Field Blank	10	2	2		
Trip Blank	10				

Notes:
TCL = Target Compound List
VOCs = Volatile Organic Compounds
SVOCs = Semi-Volatile Organic Compounds
TAL = Target Analyte List
PAHs = Polycyclic Aromatic Hydrocarbons
TPH = Total Petroleum Hydrocarbon
DRO = Diesel Range Organics

Prepared By/Date: PSJ 5/15/12
Checked By/Date: CHB 5/29/12

TABLE B-2

Field QA/QC Method Performance Criteria
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

ANALYTICAL METHOD	APPLICABLE PARAMETER	FIELD QUALITY CONTROL SAMPLE	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8260B	TCL VOCs	Trip Blank	1 per cooler of samples shipped to laboratory for VOCs.	No analytes detected at > RL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, qualify the data. OR 3) Resample the batch
SW8260B, SW8011, SW8270D, SW8151A, SW8015, SW6010C, SW7470A, SW7471B,	TCL VOCs, TCL SVOCs, TPH-DRO, TAL Metals	Duplicate	1 for every 20 field samples	RPD <30% for water and 50% for soil samples	1) Review lab QC data to determine if they are in control. 2) Qualify and use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
SW8260B, SW8011, SW8270D, SW8151A, SW8015, SW6010C, SW7470A, SW7471B	TCL VOCs, TCL SVOCs, TPH-DRO, TAL Metals	Rinsate	1 per sampling event per matrix if using non-dedicated equipment	No analytes detected at > RL	1) Qualify data. 2) If contamination is present in more than 1 rinsate above the RL, discuss with field personel to improve decontamination procedures.
SW8260B, SW8011, SW8270D, SW8151A, SW8015, SW6010C, SW7470A, SW7471B	TCL VOCs, TCL SVOCs, TPH-DRO, TAL Metals	Field Blank	1 per day	No analytes detected at > RL	Qualify data.

Notes:
TCL = Target Compound List
VOCs = Volatile Organic Compounds
SVOCs = Semi-Volatile Organic Compounds
TAL = Target Analyte List
TPH-DRO = Total Petroleum Hydrocarbons-Diesel Range Organics

Prepared By/Date: JAH 8/23/12
Checked By/Date: PSJ 8/23/12

TABLE B-3

Sample Container, Preservation, and Holding Times
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

	Solids					Aqueous				
Analyte	**Sample Preparation Method**	**EPA Analysis Method**	**Container Type**	**Preservative**	**Hold Time**	**Sample Preparation Method**	**EPA Analysis Method**	**Container Type**	**Preservative**	**Hold Time**
TCL-VOCs	5035	8260B	3 x 40 mL	2 vials with DI; 1 vial with MeOH; 1 vial unpreserved; Cool to 6º C	48 hours to freeze, 14 days to analyze	5030B	8260B	3 x 40 mL	HCl to pH < 2; Cool to 6º C	7 days
						none	8011	3 x 40 mL	HCL to ph< 2; Cool to 6º C	14 days
TCL-SVOCs	3550B	8270D	1 x 4 oz. jar	Cool to 6º C	14/40 days	3520C (Acid Only)	8270C	2 x 1 L amber	Cool to 6º C	7/40 days
	3550B	8151A-1	1 x 4 oz. jar	Cool to 6º C	14/40 days	3510	8151A-1	2 x 1 L amber	Cool to 6º C	7/40 days
TAL-Metals	3050B/ 7471B	6010C/ 6020A/ 7471B	1 x 2 oz. jar	Cool to 6º C	28 days for mercury; 180 days for others	3050A/ 7470A	6010C/ 6020A/ 7470A	1 x 500 ml	HNO_3 to pH < 2	28 days mercury; 180 days for others
TPH-DRO	3550B	8015C	1 x 4 oz. jar	none	14/40 days	3535	8015C	2 x 1 L amber	Cool to 6º C	7/40 days

Notes:
EPA = United States Environmental Protection Agency
TCL = Target Compound List
VOCs = Volatile Organic Compounds
SVOCs = Semi-Volatile Organic Compounds
TAL = Target Analyte List
DI = deionized water
MeOH = methylene chloride
HCl = hydrochloric acid
HNO_3 = nitric acid
mL = milliliter
L = liter

Prepared By/Date: PSJ 5/15/12
Checked By/Date: AC 5/31/12

TABLE B-4

Field Corrective Action Procedures
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

SITUATION		CALIBRATION	FREQUENCY	FIELD OBJECTIVE AFFECTED	CORRECTIVE ACTION PROCEDURE
Equipment Malfunction	pH	Calibrate with two buffer solutions that bracket expected sample pH	Twice daily	Equipment is calibrated and operating properly	Notification to Field Operations Leader (FOL) Repair or replace malfunctioning parts Recalibrate and/or replace standards Resample or repeat task if necessary Document to Project Manager (PM)
	Conductivity	Calibrate with one standard	Twice daily		
	Temperature	Calibrated per manufacturer's specifications	At start of job		
	Dissolved Oxygen	Calibrate per manufacturer's instructions	Twice daily		
	Redox Potential	Calibrate per manufacturer's instructions	Twice daily		
	Turbidity	Calibrate within expected range of sample turbidity	Twice daily		
	FID	Calibrate per manufacturer's instructions	Twice daily		
	PID	Calibrate per manufacturer's instructions	Twice daily		
Incorrect sample collection procedures		NA	NA	Samples are taken according to standard operation procedures	Notification to FOL Review of situation and correct procedures Recollect the sample Document to PM
Insufficient sample volume collection		NA	NA	Sufficient sample volume is provided to maintain sample integrity and so that all required analyses can be conducted	Notification to FOL by laboratory manager Review site affected and impact of samples on site characterization Correct procedures Recollect sample if necessary Document to PM
Incorrect measurement data collection		NA	NA	Measurements are conducted according to standard operating procedures	Notification to FOL Review of situation and correct procedures Document to PM and Quality Assurance Officer (QAO)

Prepared By/Date: PSJ 5/15/12
Checked By/Date: CHB 5/29/12

TABLE B-5

Field Equipment Maintenance
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Instrument	Serial Number	Type of Maintenance	Frequency	Parts Needed/ Location	Person Responsible
YSI 556 Water Quality Meter	TBD	Per equipment manual	Periodic	AMEC FOS Portland, ME	AMEC FOS Technical Staff
HACH 2100P Turbidity Meter	TBD	Per equipment manual	Periodic	AMEC FOS Portland, ME	AMEC FOS Technical Staff
Solinst Model 101 Water Level Meter	TBD	Per equipment manual	Periodic	AMEC FOS Portland, ME	AMEC FOS Technical Staff
Trimble GPS	TBD	Per equipment manual	Periodic	AMEC FOS Portland, ME	AMEC FOS Technical Staff
Photo Vac Micro FID	TBD	Per equipment manual	Periodic	AMEC FOS Portland, ME	AMEC FOS Technical Staff
MiniRae 2000 PID	TBD	Per equipment manual	Periodic	AMEC FOS Portland, ME	AMEC FOS Technical Staff

Notes:
Field equipment is maintained by AMEC Field Operations Services (FOS) located in Portland, Maine (ME) in accordance with the manufacturer's recommended maintenance schedule.
TBD = To Be Determined (actual serial numbers will be logged into project records upon receipt by field personnel).
Parts inventory is maintained by AMEC FOS and can be shipped overnight if required.

Prepared By/Date: PSJ 5/15/12
Checked By/Date: CHB 5/29/12

TABLE B-6

Field Equipment Testing Criteria
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Instrument/Equipment	Type of Inspection	Requirement	Individual Responsible	Resolution of Deficiencies
YSI 556 Water Quality Meter	Daily calibration checks	Method Requirements	AMEC Field Personnel	Recalibration or instrument maintenance
HACH 2100P Turbidity Meter	Daily calibration checks	Method Requirements	AMEC Field Personnel	Recalibration or instrument maintenance
Solinst Model 101 Water Level Meter	Daily battery check	Method Requirements	AMEC Field Personnel	Instrument maintenance
Trimble GPS	Daily battery check	Method Requirements	AMEC Field Personnel	Instrument maintenance
Photo Vac Micro FID	Daily calibration checks	Method Requirements	AMEC Field Personnel	Recalibration or instrument maintenance
MiniRae 2000 PID	Daily calibration checks	Method Requirements	AMEC Field Personnel	Recalibration or instrument maintenance

Prepared By/Date: PSJ 5/15/12
Checked By/Date: CHB 5/29/12

TABLE B-7

Laboratory Preventative Maintenance Procedures and Schedules
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Instrument	Maintenance Procedure/Schedules	Spare Parts in Stock
Gas Chromatograph/ Mass Spectrometer (GS/MS)	Replace pump oil as needed	Syringes
	Change septa weekly or as often as needed	Septa
	Change gas line dryers as needed	Various electronic parts
	Replace electron multiplier as often as needed	GC column
	Replace GC injector glass liner weekly or as often as needed	Glass liner
	Replace GC column as needed	
	Check to ensure the gas supply is sufficient for the day's activity and the delivery pressures are set as described in the SOP	
	Check to ensure that the pressure on the primary regulator never drops below 500 psi Autosampler-Syringe and tubing cleaned or replaced	
Gas Chromatograph (GC)	Change septa weekly or as often as needed	Syringes
	Change gas line dryers as needed	Septa
	Replace GC injector glass liner weekly or as often as needed	Detector supplies
	Replace GC column as needed	Glass liner
	Clean/replace GC detector as needed	GC column
	Check to ensure the gas supply is sufficient for the day's activity and the delivery pressures are set as described in the SOP	
	Check to ensure that the pressure on the primary regulator never drops below 500 psi	
Inductively Coupled Plasma (ICP)	Change pump tubing daily or as often as needed	Pump tubing
	Check nebulizer daily	Nebulizer
	Clean optics biannually or as needed	Argon
	Check to ensure the gas supply and liquid argon supply are sufficient for the day's activity and the delivery pressures are set as described in the SOP	
	Check to ensure that the pressure on the primary regulator never drops below 500 psi	
	Clean torch every three months or as needed	
Cold Vapor Mercury Analyzer Spectrometer (FIMS)	Inspect tubing and replace as needed	Tubing
	Inspect standard cups and drying tubes and replace as needed	Various replacement parts
	Inspect mixing coil; clean or replace Inspect sample probe; clean or replace Mercury lamp; clean or replace	

Prepared By/Date: AC 5/31/12
Checked By/Date: PSJ 5/31/12

TABLE B-8

Laboratory Equipment Testing Criteria
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Instrument/Equipment	Type of Inspection	Requirement	Individual Responsible	Resolution of Deficiencies
Volatiles Mass Spec, EPA 8260B	Daily calibration check, ICV/CCV	Method Requirements, SOP	Volatiles Analyst	Recalibration or instrument maintenance
Semivolatiles Mass Spec, EPA 8270D	Daily calibration check, ICV/CCV	Method Requirements, SOP	SVOCs Analyst	Recalibration or instrument maintenance
Semivolatiles GC, EPA 8011	Daily calibration check, ICV/CCV	Method Requirements, SOP	SVOCs Analyst	Recalibration or instrument maintenance
Semivolatiles GC, EPA 8151A	Daily calibration check, ICV/CCV	Method Requirements, SOP	SVOCs Analyst	Recalibration or instrument maintenance
Semivolatiles GC, EPA 8015C	Daily calibration check, ICV/CCV	Method Requirements, SOP	SVOCs Analyst	Recalibration or instrument maintenance
ICP, EPA 6010C	Daily calibration check, ICV/CCV	Method Requirements, SOP	Metals Analyst	Recalibration or instrument maintenance
FIMS, EPA 7470A	Daily calibration check, ICV/CCV	Method Requirements, SOP	Metals Analyst	Recalibration or instrument maintenance
FIMS, EPA 7471B	Daily calibration check, ICV/CCV	Method Requirements, SOP	Metals Analyst	Recalibration or instrument maintenance

Prepared By/Date: AC 5/31/12
Checked By/Date: PSJ 5/31/12

TABLE B-9

Field Equipment Calibration
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Instrument	Calibration Procedure	Frequency of Calibration	Calibration Checks	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
YSI 556 Water Quality Meter	3 point check for pH (3 buffers)	At beginning of day with calibration checks throughout the day	pH 7 at each stop	Within 0.1 SU	If out of range, check calibration again, recalibrate, then notify QA Manager	AMEC field personnel	SCDHEC Section 14
	1 point check to calibration standard for conductivity	At beginning of day with calibration checks throughout the day	Check that approximates sample conductivity	Within 10%	If out of range, check calibration again, recalibrate, then notify QA Manager	AMEC field personnel	SCDHEC Section 14
	Saturation check for DO	At beginning of day with calibration checks throughout the day	Checked prior to use	Within 0.2 mg/L*	If out of range, check calibration again, recalibrate, then notify QA Manager	AMEC field personnel	SCDHEC Section 14
	1 point check to calibration standard for ORP	At beginning of day with calibration checks throughout the day	Check against ORP standard	Within 10%	If out of range, check calibration again, recalibrate, then notify QA Manager	AMEC field personnel	YSI User Manual
HACH 2100P Turbidity Meter	2 point check for turbidity	At beginning of day with calibration checks throughout the day	Check against turbidity standard	Within 10%	If out of range, check calibration again, recalibrate, then notify QA Manager	AMEC field personnel	HACH User Manual
Photo Vac Micro FID	2 point check (fresh air and span gas)	At beginning of day with calibration checks throughout the day	Check against span gas	Within 10%	If out of range, check calibration again, recalibrate, then notify QA Manager	AMEC field personnel	Photo Vac User Manual
MiniRae 2000 PID	2 point check (fresh air and span gas)	At beginning of day with calibration checks throughout the day	Check against span gas	Within10%	If out of range, check calibration again, recalibrate, then notify QA Manager	AMEC field personnel	MiniRae User Manual

Notes:
FID = flame-ionization detector
PID = photo-ionization detector
SU = Standard Units
mg/L = milligrams per liter
*Of table value at temperature, altitude/barometric pressure, and salinity
QA = Quality Assurance

Prepared By/Date: PSJ 5/15/12
Checked By/Date: CHB 5/29/12

TABLE B-10

Laboratory Equipment Calibration
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Volatiles Mass Spec, EPA 8260B	Minimum of 5 calibration standards for all compounds	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	Volatiles Analyst	OA-11010
Semivolatiles Mass Spec, EPA 8270D	Minimum of 5 calibration standards for all compounds	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	SVOCs Analyst	OA-11011
Semivolatiles GC, EPA 8011	Minimum of 5 calibration standards for all compounds	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	SVOCs Analyst	OA-11007
Semivolatiles GC, EPA 8151A	Minimum of 5 calibration standards for all compounds	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	SVOCs Analyst	OA-11004
Semivolatiles GC, EPA 8015C	Minimum of 5 calibration standards for all compounds	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	SVOCs Analyst	OA-11002
ICP, EPA 6010C	Daily Calibration, 2 points; Linear Calibration Range studies conducted semiannually	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	Metals Analyst	IA-13002
FIMS, EPA 7470A	Minimum of 5 calibration standards for all compounds	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	Metals Analyst	IA-13037
FIMS, EPA 7471B	Minimum of 5 calibration standards for all compounds	When indicated by continuous calibration verification standard	Method Criteria	Detailed in SOP/QA Manual	Metals Analyst	IA-13033

Prepared By/Date: AC 5/31/12
Checked By/Date: PSJ 5/31/12

TABLE B-11

Supplies and Consumables
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Item	Vendor	Acceptance Criteria	Handling/Storage Conditions	Person Responsible for Inspection/Tracking
AMEC				
Gloves	Pine Environmental Services, Inc.	Undamaged and sealed packaging	Environmental storage unit	FOL
Tubing	Pine Environmental Services, Inc.	Undamaged and sealed packaging	Environmental storage unit	FOL
Color Tec Tubes	AMEC FOS	Undamaged and sealed packaging	Shipped from FOS, stored in field vehicle	FOL
Color Tec Supplies	AMEC FOS	Undamaged and sealed packaging	Shipped from FOS, stored in field vehicle	FOL
Calibration Standards	AMEC FOS	Undamaged, sealed, and within expiration date	Shipped with meters, stored in field vehicle	FOL
Alconox or Liquinox	Pine Environmental Services, Inc.	Undamaged and sealed packaging	Environmental storage unit	FOL
Deionized Water	Pine Environmental Services, Inc.	Undamaged and sealed packaging	Stored in field vehicle	FOL
Isopropanol	Fisher Scientific	Undamaged and sealed packaging	Stored in field vehicle	FOL
Aluminum foil	All	Undamaged and sealed packaging	Stored in field vehicle	FOL
Plastic sheeting	All	Undamaged and sealed packaging	Stored in field vehicle	FOL
Paper towels	All	Undamaged and sealed packaging	Stored in field vehicle	FOL
40 ml VOA	AES	Undamaged and sealed packaging	Stored in field vehicle	FOL
Batteries	All	Undamaged and sealed packaging	Stored in field vehicle	FOL
AES				
Laboratory chemicals	ERA, Accustandard, Absoulte Standards, VWR, Fisher Scientific, Restek	Certificates of analysis and laboratory testing	Laboratory storage	Receiving and laboratory personnel
Laboratory standards	ERA, Accustandard, Absoulte Standards, VWR, Fisher Scientific, Restek	Certificates of analysis and laboratory testing	Vendor-specific storage conditions	Laboratory analysts
Sample containers	QEC	Certificates of analysis and laboratory testing	Bottle storage area	Sample receiving personnel

Notes:
AMEC = AMEC Environment & Infrastructure, Inc.
AES = Analytical Environmental Services, Inc.
FOS = Field Operations Services
FOL = Field Operations Leader

Prepared By/Date: PSJ 5/15/12
Checked By/Date: AC 5/31/12

TABLE C-1

Assessment Activities
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Assessment	Frequency	Organization Responsible	Who Receives Report	Time Frame of Notification	Responsible for Corrective Action	Corrective Action Documentation	Receives Corrective Action Response
PT-full lab	1/year	Certified PT Vendor	Lab	Within 3 weeks of study close	Lab Manager	QA Form	QA Manager
Lab certification	Every 3 years	SCDHEC	Lab Manager	Within 90 days after audit	Lab Manager	Letter to SCDHEC	SCDHEC
Internal Assessment-Field	1/study	AMEC	FOL	Immediately	FOL	NCAR	AMEC PM
Internal Assessment-Lab	1/year	Lab	Lab QA Office	1 week of audit	Lab Manager	Memorandum	QA Manager
QAPP Assessment	1/study	AMEC	Lab Manager	Immediately	Lab Manager	NCAR	AMEC PM

Notes:
PT = Proficiency Test
QAPP = Quality Assurance Project Plan
SCDHEC = South Carolina Department of Health and Environmental Control
AMEC = AMEC Environment & Infrastructure, Inc.
FOL = Field Operations Leader
QA = Quality Assurance
NCAR = Nonconformance and Corrective Action Report

Prepared By/Date: PSJ 5/15/12
Checked By/Date: AC 5/31/12

TABLE D-1

Laboratory Data Qualifiers
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

FLAGS FOR DATA WITHIN ACCEPTABLE LIMITS (Usable as Reported)		
Flag	**Positive Results**	**Non-Detect Results**
No Flag	Use datum without qualification	Use datum without qualification
FLAGS FOR DATA WITHIN ACTION LIMITS (Usable with Qualification)		
Flag	**Positive Results**	**Non-Detect Results**
J	Estimated quantitation based upon QC data	Estimated quantitation based upon QC data
JB	Estimated quantitation: possibly biased high or	(Not applicable)
JH	Estimated quantitation - possibly biased high based	(Not applicable)
JL	Estimated quantitation - possibly biased low based	Possible false non-detect based upon QC data
JQ	Estimated quantitation; value is between the	(Not applicable)
UJ	(Not applicable)	Undetected; Reported detection limit is imprecise
UL	(Not applicable)	Undetected; Data biased low - Reported detection
FLAGS FOR DATA OUTSIDE ACTION LIMITS (Unsable)		
Flag	**Positive Results**	**Non-Detect Results**
R	Datum rejected based upon QC data: do not use	Datum rejected based upon QC data: do not use

Notes:
If the QC results suggest contradictory flags, the following hierarchy should be used to select the appropriate flag to assign:

R>JB>JH>JL> JQ
JH + JL = J
JQ > J

Prepared By/Date: PSJ 5/14/12
Checked By/Date: JAH 5/31/12

TABLE D-3

Data Validation and Verification Checklist
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Component	Activity
Data Deliverables and QAPP	Ensure that all required information on sampling and analysis was provided (including planning documents).
Analytes	Ensure that required lists of analytes were reported as specified.
Chain of Custody	Examine the traceability of the data from time of sample collection until reporting of data. Examine chain-of-custody records against contract, method, or procedural requirements.
Holding Times	Identify holding time criteria, and either confirm that they were met or document any deviations. Ensure that samples were analyzed within holding times specified in the method, procedure, or contract requirements. If holding times were not met, confirm that deviations were documented, that appropriate notifications were made (consistent with procedural requirements), and that approval to proceed was received prior to analyses.
Sample Handling	Ensure that required sample handling, receipt, and storage procedures were followed, and that any deviations were documented.
Sampling Methods and Procedures	Establish that required sampling methods were used and that any deviations were noted. Ensure that the sampling procedure and field measurements met performance criteria and that any deviations were documented.
Analytical Methods and Procedures	Establish that required analytical methods were used and that any deviations were noted. Ensure that the QC samples met performance criteria and that any deviations were documented.
Data Qualifiers	Determine that the laboratory data qualifiers were defined and applied as specified in methods, procedures, or contracts.
Deviations	Determine the impacts of any deviations from sampling or analytical methods and SOPs. Consider the effectiveness and appropriateness of any corrective action.
Sampling Plan	Determine whether the sampling plan was executed as specified (i.e., the number, location, and type of field samples were collected and analyzed as specified in the QAPP).
Sampling Procedures	Evaluate whether sampling procedures were followed with respect to equipment and proper sampling support (e.g., techniques, equipment, decontamination, volume, temperature, perservatives, etc.)
Co-Located Field Duplicates	Compare results of co-located field duplicates with criteria established in the QAPP.
Project Quantitation Limits	Determine that quantitation limits were achieved as outlined in the QAPP and that the laboratory successfully analyzed a standard at the quantitation limit.
Confirmatory Analyses	Evaluate agreement of laboratory results.
Performance Criteria	Evaluate QC data against project-specific performance criteria in the QAPP (i.e., evaluate quality parameters beyond those outlined in the method).
Data Qualifiers	Determine that the data qualifiers applied were those specified in the QAPP and that any deviations from specifications were justified.
Validation Report	Summarize deviations from methods, procedures, or contracts. Include qualified data and explanation of all data qualifiers.

Prepared By/Date: PSJ 5/14/12
Checked By/Date: JAH 5/31/12

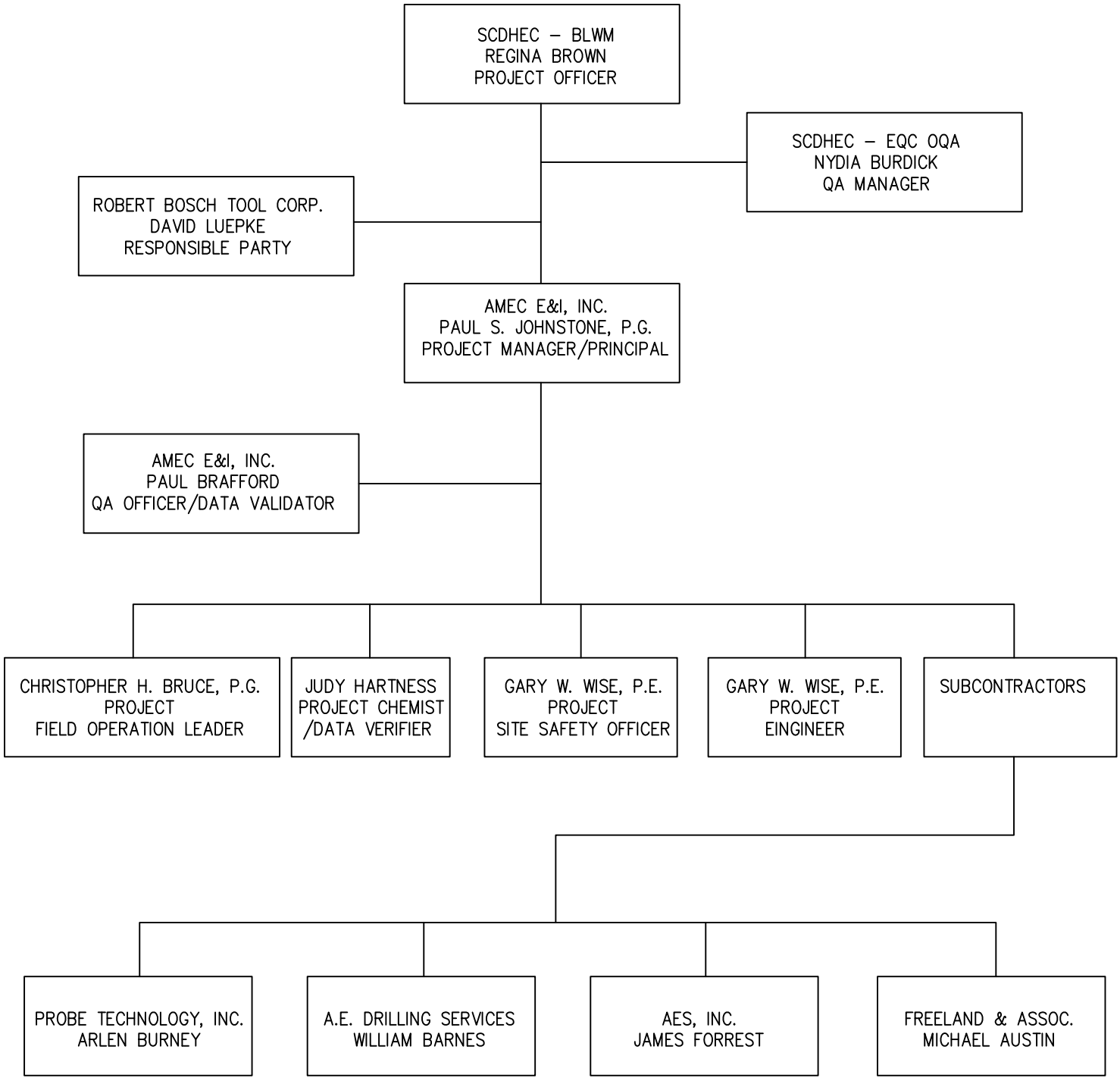
TABLE D-3

Data Usability Assessment Checklist
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Component	Assessment Activity
Data Deliverables and QAPP	Ensure that all necessary information was provided, including but not limited to validation results.
Deviations	Determine the impact of deviations on the usability of data.
Samping Locations, Deviation	Determine if alterations to sample locations continue to satisfy the project objectives.
Chain-of-Cusody, Deviation	Establish that any problems with documentation of custody procedures do not prevent the data from being used for the intended purpose.
Holding Times, Deviation	Determine the acceptability of data where holding times were exceeded.
Damaged Samples, Deviation	Determine whether the data from damaged samples are usable. If the data cannot be used, determine whether resampling is necessary.
PT Sample Results, Deviation	Determine the implications of any unacceptable analytes (as identified by the PT sample results) on the usability of the analytical results. Describe any limitations on the data.
SOPs and Methods, Deviation	Evaluate the impact of deviations from the SOPs and specified methods on data quality.
QC Samples	Evaluate the implications of unacceptable QC sample results on the data usability for the associated samples. For example, consider the effects of observed blank contamination.
Matrix	Evaluate matrix effects (interference or bias).
Meterological Data and Site Conditions	Evaluate the possible effects of meteorological (e.g., wind, rain, temperature) and site conditions on sample results. Review field reports to identify whether any unusual conditions were present and how the sampling plan was executed.
Comparability	Ensure that the results from different data collection activities achieve an acceptable level of agreement.
Completeness	Evaluate the impact of missing information. Ensure that enough information was obtained for the data to be usable (completeness as defined in the DQOs documented in the QAPP).
Background	Determine if background levels have been adequately established (if appropriate).
Critical Samples	Establish that critical samples and critical target analytes/COCs, as defined in the QAPP, were collected and analyzed. Determine if the results meet the criteria specified in the QAPP.
Data Restrictions	Describe the exact process for handling data that do not meet DQOs (i.e., when measurement performance criteria are not met). Depending on how those data will be used, specify the restrictions on use of those data for environmental decision making.
Usability Decision	Determine if the data can be used to make a specific decision considering the implications of all deviations and corrective actions.
Usability Report	Discuss and compare overall precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity for each matrix, analytical group, and concentration level. Describe limitations on the use of project data if criteria for data quality indicators are not met.

Prepared By/Date: PSJ 5/14/12
Checked By/Date: JAH 5/31/12

FIGURES

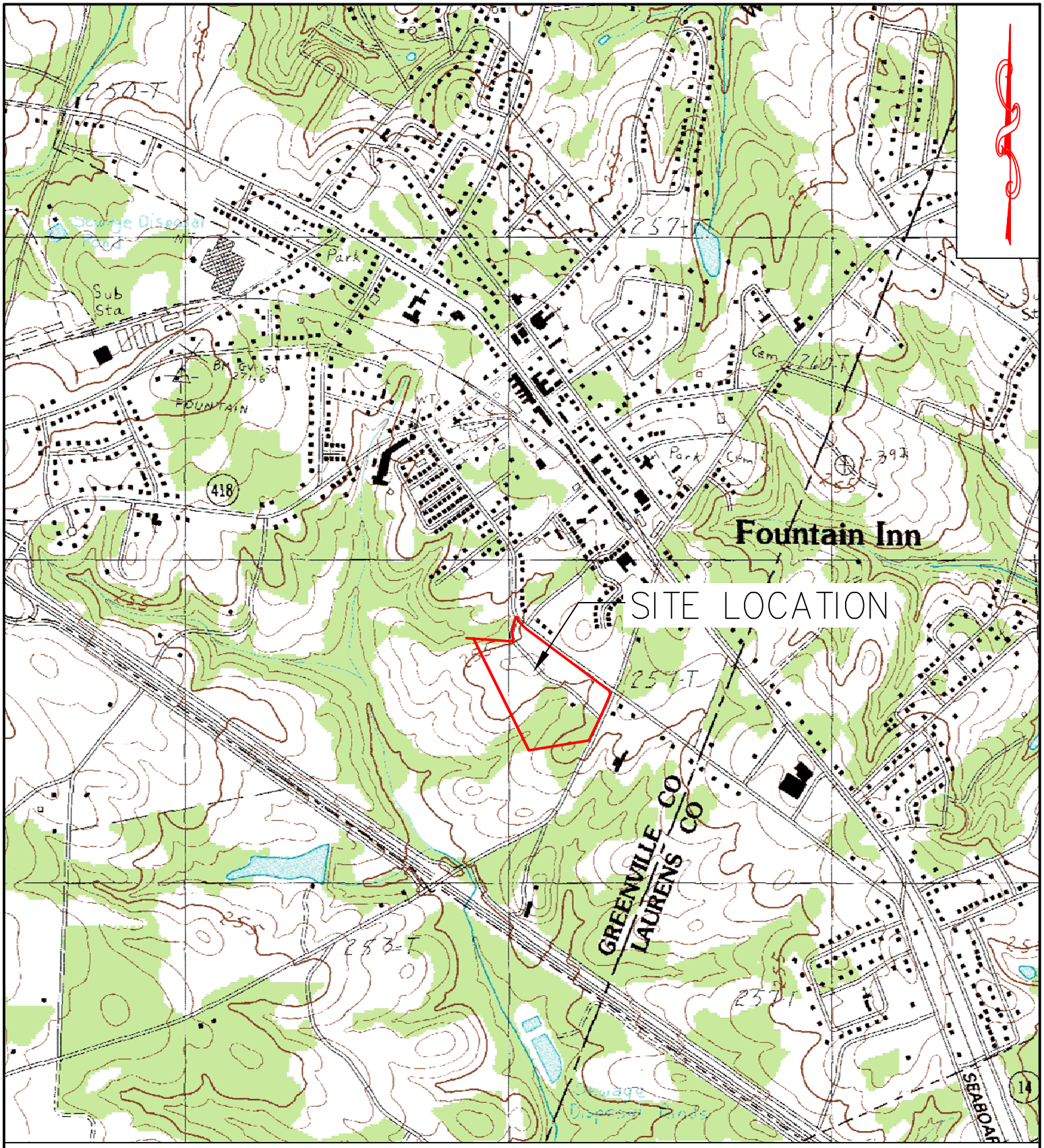


amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

PROJECT ORGANIZATION CHART
FORMER VERMONT BOSCH SITE
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A1

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE A-1.DWG	CHB	PSJ	PSJ	5/8/12	6251121007.01.01



REFERENCE:
2001 DELORME STREET ATLAS USA

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APPROXIMATE SCALE IN MILES

amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

SITE LOCATION MAP
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
A-2

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE A-2.DWG	CHB	PSJ	PSJ	5/8/12	6251121007.01.01

APPENDIX A

ANALYTICAL ENVIRONMENTAL SERVICES, INC.
QUALITY ASSURANCE MANUAL
AND
STANDARD OPERATING PROCEDURES

APPENDIX A-1

Laboratory Quality Assurance Manual

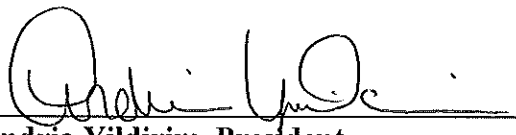
Standard Operating Procedure for the

Quality Assurance Manual

Analytical Environmental Services, Inc.
3785 Presidential Parkway

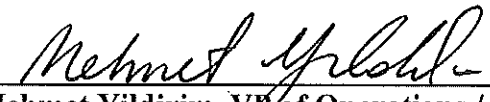
Atlanta, Georgia 30340-0370
(770) 457-8177
FAX (770) 457-8188

Effective Date of Revision 16: February 27, 2012
Portal Server Location:
http:// Home/AES/Quality Assurance/QA Manual/
2012_QA_Manual_Rev_16



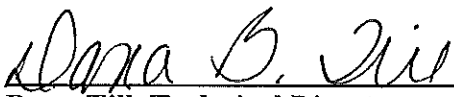
3/1/12

Andria Yildirim, President **Date**
24 River Park Drive, NW
Atlanta, Georgia 30328
(770) 913-9431 phone



2/27/12

Mehmet Yildirim, VP of Operations / Laboratory Manager **Date**

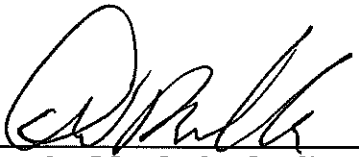


2/27/12

Dana Till, Technical Director **Date**

2/27/12

James Wallace, Technical Director Microbiology **Date**



2/27/12

Douglas Mendrala, Quality Assurance Manager **Date**

STANDARD OPERATING PROCEDURES FOR THE QUALITY ASSURANCE PROGRAM

2.0 TABLE OF CONTENTS

2.0 TABLE OF CONTENTS (cont.)

2.0 TABLE OF CONTENTS (cont.)

2.0 TABLE OF CONTENTS (cont.)

2.0 TABLE OF CONTENTS (cont.)

3.0 STATEMENT OF POLICY

3.1 Quality Policy

The objective of Analytical Environmental Services, Inc. is to generate high quality data in a cost-effective manner, which is accurate, impartial, reliable, and adequate for its intended use. The management of AES is committed to following accepted laboratory practices to achieve high quality of testing services, and strives to ensure both the analytical validity and legal defensibility of all reported data.

AES management is committed to compliance with The NELAC Institute (TNI) Standards, AIHA International Standard (ISO/IEC 17025) as well as North Carolina and South Carolina rules to establish, implement, and maintain a quality system appropriate to the scope of all laboratory activities, including the type, range, and volume of testing. Management shall document the policies, systems, programs, procedures, and instructions to the extent necessary to enable AES to assure the quality of the test results generated.

Quality system documentation is communicated to, understood by, and made available to personnel through AES management by means of training and educational instruction. All laboratory staff concerned with analytical testing activities must familiarize themselves with the quality documentation and implement the policies and principles in their work.

It is the policy of AES to continually improve quality systems and provide support to improvement efforts.

3.2 Purpose

The Quality Assurance Program (QAP) sets forth the management policy, organizational structure, and procedures for chemical analyses performed by AES. Management encourages the development and use of the best testing practices as dictated by each measurement situation. However, the procedures set forth herein must be followed to the greatest extent possible. All deviations must be documented in each individual case and maintained with the sample data. The QA Manual and all Standard Operating Procedures will be reviewed no less than annually.

Appropriate use of data generated under the varying conditions encountered in environmental analyses requires reliance on the quality control practices incorporated into the procedures. Although the EPA, state environmental protection departments, The NELAC Institute (TNI), AIHA, other regulatory agencies, and clients require the use of approved methods for sampling and analysis, the mere approval of these procedures does not guarantee adequate results. Inaccuracies can result from many causes, including matrix effect, equipment malfunction, and operator error. Therefore, the quality control component of each method is indispensable and cannot be compromised.

This manual delineates the elements of the QA Program that must be implemented by all analytical sections of the laboratory. The requirements outlined in this procedure are the minimum requirements. Method-specific procedures and project-specific Quality Assurance Project Plans (QAPP) may require more stringent QA requirements.

3.3 Definitions

3.3.1 Quality Assurance (QA) is the total program for assuring reliability of the monitoring and measurement of data. It comprises all those planned and systematic actions necessary to

provide adequate confidence that all aspects of laboratory service programs are performed in a manner satisfactory to AES management and to the needs of its customers.

3.3.2 Quality Control (QC) is the routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process. It encompasses the operational procedures, techniques, and activities that provide a means to measure, evaluate, and document the quality of data obtained in the laboratory. The QC Program specifies the minimum practices, which shall be implemented to assure that data is produced of a known and defensible quality and within acceptable limits.

3.4 Fields of Testing

This manual covers methods for the analysis of aqueous, solid, waste, and air matrices currently on AES scopes of accredited testing for AIHA, Florida DOH, The NELAC Institute (TNI), North Carolina DENR and South Carolina DHEC. A detailed list of test methods and analytes may be found in Section 5.0, which defines the minimum level of quality assurance/quality control needed to meet required specifications. All methods carried out by AES shall meet these stipulations as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs), or local regulations may require criteria other than those stated. In these cases, the laboratory will abide by the more stringent criteria, following a review and acceptance of the requirements by the Laboratory Manager and the Quality Assurance Manager.

3.5 Management of the Quality Assurance Manual

This manual was prepared in accordance with the current The NELAC Institute (TNI) standards and AIHA requirements. It also follows guidelines set by the U.S. Environmental Protection Agency, Florida DOH and ISO/IEC 17025.

Tests are always carried out in accordance with stated methods and customers' requirements.

3.5.1 The QA manual is reviewed annually by the Quality Assurance Manager and laboratory personnel to confirm that it reflects current in-house practices and meets all the requirements of both AES' clients and accrediting agencies. Modifications may be made in order to correct inconsistencies, implement improvements, encompass new concepts or procedures, adapt to new regulations, or update any changes in state or national policies or standards. The Quality Assurance Manager, Laboratory Manager, Technical Director, and relevant operational staff review the changes before they are integrated into the QA manual.

3.5.2 Policies or procedures in the manual which demand immediate attention are addressed through the use of temporary and permanent Interim Change Notices as described in Section 8.

3.6 Control of the Quality Assurance Manual

The Quality Assurance Manual is considered confidential within Analytical Environmental Services, Inc. It may not be altered in any manner by anyone other than the Quality Assurance Manager, the Laboratory Manager, or an employee duly appointed by either of the aforementioned. The manual shall be marked as an "Uncontrolled Copy" if provided to external users or regulators. It is intended for the exclusive purpose of the review of AES' quality systems and shall not be used in any other way without written permission of the President, Laboratory Manager, or Quality Assurance Manager.

3.7 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence shall be as follows:

1. Analytical Environmental Services, Inc., Interim Change Notice
2. Quality Assurance Manual
3. Standard Operating Procedures
4. Other (memos, charts, published methods, etc.)

4.0 ORGANIZATION AND RESPONSIBILITY

4.1 Organization

Analytical Environmental Services, Inc. is a locally owned, permanent laboratory facility that performs chemical and biological testing on a variety of environmental samples. These include solid waste matrices, soils, sediments, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, air sampling media, ground, surface and waste waters, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, and tars.

4.2 Organizational Structure

The relationship between management, technical operations, support services and quality system is as follows: Laboratory Operations, Quality Assurance Department, Technical Director, and Customer Service Department report to the Vice President of Operations, who in turn reports to the company President. The Vice President of Technical Operations (Support Services) also reports directly to the President. The organizational structure of AES provides for an independent Quality Assurance Department with the overall responsibility of developing and auditing for compliance to a comprehensive Quality Assurance Program. The QA Department has the authority and organizational freedom to ensure that QA activities are implemented and accomplished. The Quality Assurance Manager reports directly to the Vice-President of Operations of AES.

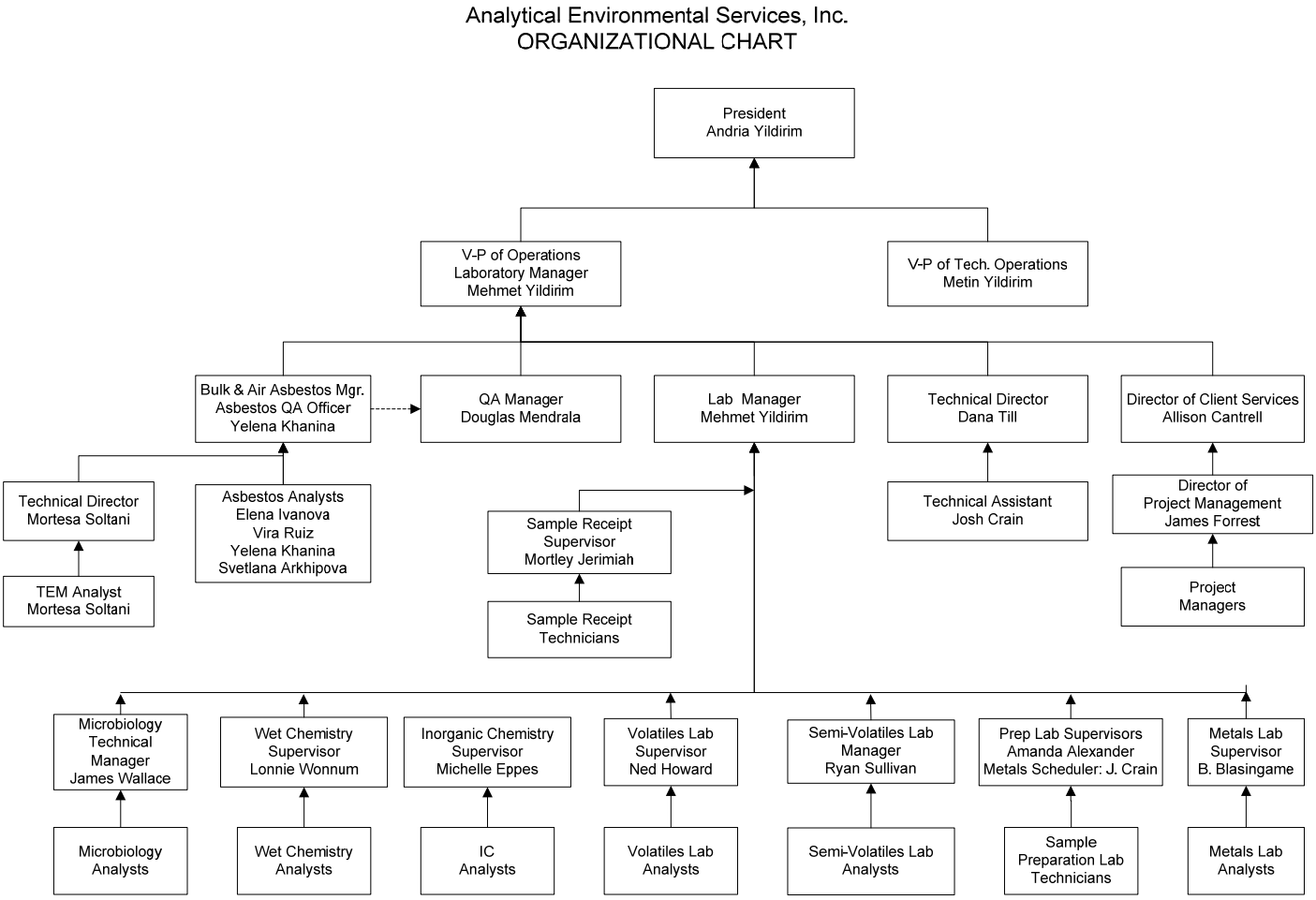
4.2.1 Because of the breadth of knowledge required to produce quality data, the cooperation of numerous individuals is required. All assigned personnel shall remain diligent to identify, report, and promptly rectify issues or events affecting data quality as they occur. To encourage the identification of these situations, management at all levels shall promote continuous quality improvement throughout the entire company. These events and their resolutions must be verified and substantiated as required by this document and any other applicable QA guidelines.

4.2.2 The establishment of a Quality Assurance Program requires the services of all the employees of AES in order to carry out the monitoring, record keeping, statistical techniques, and other functions required by the system. This total commitment of all personnel to the production and reporting of reliable data is dependent upon the conscientious effort of everyone involved. It is important, therefore, that each member of the organization have a clear understanding of his duties, responsibilities, and relationship to the total effort.

4.3 Organizational Chart

The organizational structure at AES is documented in the form of an organizational chart, Figure 4-1, which identifies the personnel involved in the production of quality data and depicts the lines of communication and responsibility throughout the entire organization.

Figure 4-1, ORGANIZATIONAL CHART



* The QA Manager will serve as deputy in the event of the Technical Director's absence.
** The Technical Director will serve as deputy in the event of the QA Manager's absence.

For AIHA accreditation: The Laboratory Technical Director will serve as deputy for the Microbiology Department Technical Manager. The Laboratory Quality Assurance Manager will serve as deputy for the Microbiology Department Quality Assurance Coordinator.

4.4 Responsibilities and Position Requirements

It is the responsibility of all AES employees to implement the Quality Assurance Program effectively. All chemists and technicians are responsible for understanding and following the measures of the QA program, and for reporting any quality failures to a Manager or Supervisor in a timely manner. Supervisors and Managers are responsible for ensuring that all laboratory personnel are familiar with the requirements of the Quality Assurance Program and that these requirements are implemented and maintained. It is the responsibility of the Supervisor to ensure that all laboratory personnel are trained to perform their assigned tasks. It is the responsibility of each Supervisor to ensure that any quality failures are reported to the Quality Assurance Department immediately.

The essential personnel involved in the implementation of and/or monitoring of the Quality Assurance Program are identified in the following sections.

4.4.1 President

The President is ultimately responsible for the quality of services provided by AES. The President is also responsible to establish and implement the procedures, policies, and findings of the QA program. The President is responsible for the commitment of delivering the appropriate tools and resources to the senior level staff and laboratory management to ensure that the overall QA program and clients needs can be met.

4.4.2 Vice-President of Operations

The Vice-President of Operations is responsible for the overall operation of the laboratory and reports directly to the President. The Vice-President of Operations ensures that all of the resources are available to implement and follow the procedures and policies as written in the AES QA Manual as well as management's commitment to compliance with The NELAC Institute (TNI) Standards. The Vice-President of Operations reviews and approves the Corporate Quality Assurance Manual.

Either the President or Vice-President of Operations will conduct the annual management review of laboratory operations to assess the effectiveness of policies and procedures in order to implement changes where deemed necessary. The agenda of the annual meeting will include reports from all department supervisors and cover such topics as quality assurance, accreditations, documentation, changes in the laboratory, equipment and maintenance needs, results of audits etc. The topics to be discussed will be determined by the President or Vice-President of Operations. A current list of topics is presented in Attachment 7.

4.4.3 Vice-President of Technical Services

The Vice-President of Technical Services reports directly to the President and is responsible for the selection and trouble-shooting of all equipment and instrumentation. The Vice-President of Technical Services is also responsible for the installation, maintenance, and data management associated with all computers, automated equipment, network systems, software, and Internet services, as well as the Laboratory Information Management System (LIMS). The Vice-President of Technical Services ensures that all computers and automated equipment used for acquiring, processing, manipulating, recording, reporting, retrieving, or storing test data meet all of AES' Quality Assurance objectives, and that all computer software is documented and adequate for use. This position provides for the protection of the integrity of all electronic data. All computers and automated equipment must be maintained to ensure proper functioning, which includes providing environmental and operating conditions necessary to maintain the integrity of the test data. The Vice-President of Technical Services establishes and implements appropriate procedures for ensuring electronic data security.

4.4.4 Laboratory Manager

The Laboratory Manager is responsible for the daily operations within the analytical sections of the laboratory. If the Laboratory Manager is absent for a period of time exceeding 15 consecutive calendar days, the Vice-President of Operations must designate another full-time staff member meeting the qualifications of the Laboratory Manager to temporarily perform this function. In case of a change of Laboratory Manager, all necessary, accrediting authorities must be notified in writing within thirty days. The following is the position description for Laboratory Manager:

Position Description and Requirements

Position Title: **Laboratory Manager**

Position Description: This function contributes to Analytical Environmental Services, Inc. (AES) by the following:

- Oversees the daily operations of the laboratory.
- Ensures that client specific reporting & quality control requirements are met.
- Works with the Project Managers and Group Team Leaders to ensure that project objectives are met in a timely manner.
- Sets goals and objectives for both the business and the laboratory employees.
- Provides direction to departmental managers to steer all departmental efforts toward the overall corporate production goals.
- Discusses and resolves disagreements, as necessary, with laboratory personnel.
- Coordinates any unresolved concerns between the project managers and the departmental supervisors.
- Ensures that all analysts and supervisors have the appropriate education & training to properly carry out the duties assigned to them, and ensures that this training has been documented.
- Ensures that a sufficient number of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that HR policies are adhered to and maintained.
- Ensures management's commitment to compliance with The NELAC Institute (TNI) Standards
- Hires key personnel and recruits professional talent.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Schedules analytical operations.
- Supervises the maintenance of instruments and the scheduling of repairs.
- Ensures that appropriate corrective actions are taken to address analyses as requiring such actions by internal & external performance or procedural audits.
- Ensures that personnel are free from any commercial, financial or other undue pressures that which adversely affect the quality of their work.
- Supervises the preparation & maintenance of laboratory records.
- Responsible for holding documented meetings as needed with the departmental supervisors.

Position Requirements: Must have a BA or BS in Chemistry, Microbiology, Biology, Environmental Science or any other related degree. Must have 2-5 years of experience carrying out the duties described above.

4.4.5 Project Manager

The Project Manager is responsible for directly ensuring that the individual client's needs are met on a project-by-project basis with respect to the laboratory's QA program and any project-specific QA programs. The Project Manager is responsible for disseminating any project-specific information to the Laboratory Manager and/or Laboratory Director. Non-routine QA requirements must be approved by the Laboratory Director and Laboratory Manager. The following is the position description for Project Manager:

Position Description and Requirements

Position Title: **Project Manager**

Position Description: This function contributes to Analytical Environmental Services, Inc. (AES) by the following:

- Ensures effective and accurate communication between the client and the laboratory.
- Handles all client requests and needs.
- Utilizes any corporate documents to consult with clients about client questions or concerns.
- Responsible for notifying the Director of Project Management of any client activities that entail services that are not currently performed by AES.
- Assesses client requests in light of current workload with consultation with the Director of Project Management.
- Develops and maintains client records and requirements.
- Ensures that the laboratory is aware of, and completes, all client requests and requirements.
- Responsible for meeting with the Marketing Manager, Director of Project Management, and President on a periodic basis for marketing purposes.
- Communicates proper sampling, shipping, and receiving procedures to clients.
- Documents all client interaction and maintains all client information in the Project Management System.
- Reviews and approves data reports prior to their release to the clients.
- Ensures client specific reporting and quality control requirements are met.

Position Requirements: A Degree or the necessary experience to achieve the position requirements outlined in the Position Description.

4.4.6 Quality Assurance Manager

The QA Manager is responsible for establishing a Quality Assurance Program that meets the quality assurance objectives of the company, and its clients. If the QA Manager is absent for a period of time exceeding 15 consecutive calendar days, the Vice-President of Operations must designate another full-time staff member meeting the qualifications of the QA Manager to temporarily perform this function. In case of a change of QA Manager, all necessary accrediting authorities must be notified in writing within thirty days. The following is the position description for Quality Assurance Manager:

Position Description and Requirements

Position Title: **Quality Assurance Manager**

Position Description: This function contributes to Analytical Environmental Services, Inc. (AES) by the following:

- Directs all corporate quality assurance (QA).
- Responsible for developing and maintaining all QA systems and documentation.
- Responsible for all aspects of the State and Federal Certification processes.
- Maintains records of acceptable performance of MDLs.
- Directs management to compliance with The NELAC Institute (TNI) Standards
- Maintains all quality control charts.

- Has direct access to the Technical Director and to the highest level of management where decisions are made on laboratory policy and resources.
- Serves as focal point for QA/QC and takes responsibility for the oversight and or review of quality control data.
- Functions independently from laboratory operations for which QA oversight is held.
- Evaluates data objectively and performs assessments without outside influence.
- Conducts internal audits on the entire laboratory technical operation annually.
- Notifies laboratory management of deficiencies in the quality system and monitors corrective action.
- Maintains currency of the QA manual.
- Responsible for preparing and submitting a quarterly status report to the President and Vice-President of Operations.
- Serves as deputy in the event of the Technical Director's absence.

Position Requirements: A Degree or the necessary experience to achieve the requirements outlined in the position description. Trained and qualified as an auditor.

4.4.7 QA Assistant

The QA Assistant is responsible for assisting the QA Manager in the review and preparation of various types of quality assurance and quality control documentation. The QA Assistant ensures that the data meets the quality assurance objectives of the company, and its clients. The QA Assistant is responsible for effective and accurate communication and data review with and between lab personnel and management as required.

Position Description and Requirements

Position Title: **Quality Assurance Assistant**

Position Description: This function contributes to Analytical Environmental Services, Inc. (AES) by the following:

- Responsible for assisting the QA Manager in maintaining all QA systems, documentation and day-to-day functions of the QA Department as needed.
- Notifies the QA Manager of deficiencies in the quality system and assists the QA Manager in addressing corrective actions and technical reprimands.
- Responsible for creating, maintaining, updating, modifying, issuing, collecting, tracking, retiring, scanning, and posting laboratory logbooks, checklists and assorted records.
- Responsible for tracking, monitoring checks, maintenance and calibrations of various laboratory controls and records such as but not limited to pipettors, balances, weights, thermometers, conductivity meters, and temperatures.
- Responsible for ordering, logging into LIMS, tracking, witnessing PT Sample preparation, and reporting Proficiency Test (PT) Studies after review by QA Manager.
- Conducts monthly, quarterly, and annual internal audits on the entire laboratory technical operation as needed.
- Performs QA Manual and Legal & Ethical training.
- Scans and posts correspondence and documentation to Portal Server for archiving.
- Performs data review and QA acceptance of certain tests as needed.

- Assists with updates to the QA Manual and intra-laboratory QC ranges and limits.
- Schedules, tracks and reviews data from required studies (e.g. Method Detection Limit Study).
- Performs other administrative and laboratory tasks as needed.

Position Requirements: A Degree or the necessary experience to achieve the requirements outlined in the position description. Trained and qualified as an auditor.

4.4.8 Technical Director

The Technical Director exercises daily supervision of laboratory procedures and the reporting of results. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Vice-President of Operations must designate another full-time staff member meeting the qualifications of the Technical Director to temporarily perform this function. In case of a change of Technical Director, all necessary accrediting authorities must be notified in writing within thirty days. The following is the position description for Technical Director:

Position Description and Requirements

Position Title: **Technical Director**

Position Description: This function contributes to Analytical Environmental Services, Inc. (AES) by the following:

- Updates SOPs as required.
- Maintains Test Codes
- Ensures that all employees are properly trained
- Reviews and approves revisions to the Quality Assurance Manual.
- Maintains records of employee training including acceptable performance of IDOCs.
- Provides technical assistance in the development of new methods.
- Responsible for following direction given by the Vice-President of Operations.
- Ensures management's commitment to compliance with The NELAC Institute (TNI) Standards
- Provides technical guidance to analytical staff.
- Assists with internal and external audits.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits.
- Oversees equipment maintenance and repair.
- Assists the Laboratory Manager in the investigation of new technologies and proposed equipment acquisitions by the laboratory.
- Serves as deputy in the Quality Manager's absence.

Position Requirements: A Bachelors Degree in chemical, environmental, biological, or physical sciences or engineering, with at least 24 college semester credit hours in chemistry and at least two years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A Masters or Doctoral Degree may be substituted for one year of experience.

4.4.9 Technical Assistant

The Technical Assistant reports directly to the Technical Director and assists with the implementation and maintenance of all programs assigned to the Technical Director.

Position Description and Requirements

Position Title: **Technical Assistant**

Position Description: This function contributes to Analytical Environmental Services, Inc. (AES) by the following:

- Schedules, tracks and provides preliminary document review for DOC studies.
- Performs SOP updates as instructed from Tech. Director.
- Maintains SOP document control system.
- Scans and publishes completed documents to Portal Server for archiving.
- Schedules and documents training sessions and staff meetings held by Tech. Director.
- Assists Tech. Director with development of training program content and media.
- Assists Tech. Director with day-to-day functions of the Tech. Direction Dept. as needed.

Position Requirements: A Bachelors Degree in the chemical, environmental, biological, or physical sciences or engineering.

4.4.10 Director of Project Management

The Director of Project Management serves as a liaison between the laboratory and its clients, and ensures delivery of data packages. The following is the position description for the Director of Project Management:

Position Description and Requirements

Position Title: **Director of Project Management**

Position Description: The Director of Project Management serves as a liaison between the laboratory and its clients, and ensures delivery of data packages. Responsibilities include:

- Meets client specifications by communicating project and quality assurance requirements to the laboratory.
- Assigns project managers.
- Notifies laboratory personnel of incoming projects and sample delivery schedules and requirements.
- Monitors the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Informs clients of data package related problems and resolves service issues.
- Coordinates requests for sample containers and other services such as data packages.
- Reviews and approves, with input from the Vice-President of Operations, proposals for marketing.
- Reviews laboratory data reports and quotes.

Position Requirements: High School diploma or equivalent, at least 2 years management or supervisory experience, strong computer and personnel skills, knowledge of the

environmental and chemical sciences, and previous project management experience.

4.4.11 Department Manager

Oversees daily operation of department(s), supervises all employees, and handles all issues in the department(s).

Position Description and Requirements

Position Title: **Department Manager**

Position Description:

Managerial duties:

- Supervise all employees to ensure they are working to full potential and are being productive at all times.
- Handle all personnel issues, i.e. conflict between workers, inappropriate behavior, schedule changes, time-off requests, etc…
- Write warnings if needed.
- Ensure employee's time sheets reflect actual work schedule.
- Make sure clock in-out times are accurate.
- Make sure employees are coming to work at the designated time.
- Monitor employee breaks.
- Assign tasks to personnel using the Task Management software.
- Grade task upon completion, this is to be included in the employee's Performance Evaluation.
- The use of this software will also be used in performing supervisor's Performance Evaluation.
- Perform Employee Performance Evaluations on all employees in department.

Production responsibilities:

- Maintain backlog to ensure all samples are completed within holding time, due date, and that all special requirements are met.
- Keep track of inventory and order supplies as needed.
- Sufficient amounts of reagents, solvents, standards, etc… must be kept at all times so production is not affected because of a shortage of supplies.
- Identify and solve problems within the department including, but not limited to equipment, tests performed, and any other issues resulting from the preparation/analysis of samples.
- A supervisor is required to stay until problems are solved or rush work is completed to within a reasonable amount of time or hour (this includes staying late and working weekends.)
- Delegate work to employees.
- Assign batches and/or tests.
- Assign new tests to employees so workload can be spread evenly among staff.
- Assign duties to employees, i.e. ordering of supplies, logging in new supplies, etc…

QA Responsibilities:

- Ensure all employees are properly trained and DOC's performed.
- Ensure all CDOC's are performed on a yearly basis for all employees and for all tests.

- Ensure MDL's are completed/prepped yearly, more often where applicable, or as needed due to instrument changes/maintenance.
- Complete PT samples in a timely manner and identify any issues with test as soon as possible.
- If necessary, coordinate preparing/running of Proficiency samples with associated departments to ensure their timely completion and enough samples remain for all tests.
- QA review any data generated within department.
- Review and revise SOP's when necessary.
- Ensure all batches, logbook pages, raw data, and associated paperwork are scanned and posted onto the Portal Server.

Position Requirements: A Bachelors Degree in chemical, environmental, biological, or physical sciences or engineering and at least two years of experience in the environmental analysis representative of that which will be overseen.

4.4.12 Supervisors

Supervisors are responsible for the operation of their respective section of the laboratory, and report to the managers.

Position Description and Requirements

Position Title: **Supervisors**

Position Description: Supervisors report to their respective Manager on all aspects of sample processing. If a section does not have a supervisor, the Manager of that section functions as the supervisor. The Supervisor's responsibilities include, when applicable:

- Training and qualification of personnel (under their supervision) on procedures.
- Monitors necessary protocols and standard operating procedures, including control charts.
- Maintains QC within their area of responsibility.
- Ensures that personnel (under their supervision) use approved procedures, and maintain all instrumental QC.
- Recommends and implements new or revised QC policies as approved by the QA Manager.
- Assists Technical Director in reviewing preventative maintenance as detailed in the QA manual or SOPs.
- Reviews all data and QC results, and reports non-conformances to the appropriate QA Manager, Technical Manager, and/or Vice-President of Operations.
- Provides guidance to analysts in resolving problems encountered daily during sample preparation and analysis, in conjunction with the Technical Director or Quality Assurance Manager.
- Ensures all logbooks are maintained and current.
- Maintains adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Assists Technical Director with MDLs and IDOCs.

Position Requirements: A Degree or the necessary experience to achieve the requirements outlined in the position description.

4.4.13 Analysts

Analysts are responsible for performing the various testing, digestive, and extractive procedures required in the laboratory.

Position Description and Requirements

Position Title: **Analysts**

Position Description: Each type of analyst position and the specific training required is described in detail in the Employee Training Files maintained by the Technical Director. In general, analysts are responsible for the following duties:

- Performs analyses by adhering to analytical and quality control protocols prescribed by SOPs, the QA manual, turn around times, rush analyses and short hold analyses, and project specific requirements (e.g. data packages).
- Documents standard and sample preparation, instrument calibration and maintenance, data calculations, and any observed non-conformances on work lists, bench sheets, or laboratory notebooks.
- Reports all out-of-control situations, instrument problems, matrix problems, and QC failures, which might affect the reliability of the data, to their respective supervisors or the QA Manager.
- Performs 100 percent review of the data generated prior to entering and submitting the data to the next level of review.
- Suggests method improvements to their supervisor, Technical Director, or the QA Manager for potential incorporation into SOPs.

Position Requirements: At a minimum, analysts must possess a high school diploma or equivalent. If the analyst operates ICP or GC/MS equipment, the analyst must satisfactorily complete a short course offered by an equipment manufacturer, professional organization, university, or other qualified training facility (formal in-house training is acceptable). The minimum experience requirement for the independent operation of AA, ICP, HPLC and GC is six months; 1 year for GC/MS equipment.

4.4.14 Project Manager Assistant

Project Manager Assistants are responsible for providing assistance to project managers with the production and completion of data packages.

Position Description and Requirements

Position Title: **Project Manager Assistant**

Position Description: Project manager assistants report to the project managers. This position is primarily responsible for assisting project managers with on time completion of all data packages and to ensure effective and accurate communication between lab and project managers with respect to data package status. In general project manager assistants are responsible for the following duties:

- Assigns data packages and completion dead lines to appropriate lab departments.
- Responsible for initial data package review after data package was completed by lab departments

- Responsible of notifying project managers or Director of Project Management of any internal problems or discrepancies that may affect data package on time completion.
- Responsible for formatting data package (inserting dividers, making table of contents, copying reports, COC and checklist, putting all data in appropriate order, etc);
- Responsible for setting bookmarks and creating CD ROM's, completing and updating data package status document (located on the AES Server) on the daily basis, and ensures that data package was scanned or copied after approved by the project manager

Position Requirements: A Degree or the necessary experience to achieve the requirements outlined in the position description.

4.5 Improper, Unethical, or Illegal Actions and Data Integrity System

4.5.1 It is recognized that the quality assurance program is an inherent function involving all of the organizational components and personnel. The achievement of quality objectives is attained by each individual performing assigned work in strict compliance with approved and applicable requirements and procedures.

4.5.2 For a quality assurance program to succeed, it is imperative that all employees adhere to procedures which detect and prevent improper, unethical, or illegal actions which could in any way compromise the reliability and data integrity. Training in legal, ethical, and data integrity responsibilities is mandatory. Records are maintained that document, through individual signatures, that every employee understands the consequences of improper, unethical, or illegal actions related to data integrity. Potential instances of improper, unethical, illegal actions or Data Integrity issues will be discussed and addressed in senior management meetings.

4.5.3 Improper actions are defined as deviations from method-specified or client-specified analytical or quality assurance practices. These events may be intentional or unintentional. Disciplinary measures may include verbal warnings, written warnings, and/or dismissal.

4.5.4 Unethical or illegal actions are defined as the deliberate falsification or alteration of analytical or quality assurance results where failed method, quality control, or client specifications are made to appear acceptable. These actions affect the integrity of the data. Also included as unethical or illegal actions is the falsification and reporting of data where analyses were never performed. Disciplinary measures may include verbal warnings, written warnings, and/or dismissal. Findings of fraud may be prosecuted to the fullest extent of the law.

4.5.5 Employee training of legal, ethical, and data integrity responsibilities establishes the program and procedures that prevent and detect improper, unethical, or illegal actions by employees. Deterrence begins with a position of zero tolerance established by management. Employee training supports and sustains the policy.

4.5.5.1 Training of laboratory employees with respect to their legal and ethical responsibilities is comprised of three basic components:

4.5.5.1.1 The definition of improper, unethical, or illegal actions.

4.5.5.1.2 The elements of the laboratory's prevention and detection program.

4.5.5.1.3 Some examples of inappropriate laboratory practices that affect data integrity.

4.5.5.2 Training courses in legal and ethical responsibilities also include the potential punishments and penalties for fraudulent conduct.

4.5.6 Laboratory management implements a variety of proactive measures to promote the prevention and detection of improper, unethical, or illegal activities. Minimum requirements are included in the quality program by means of the following:

4.5.6.1 An ethics and data integrity policy that is read and signed by all personnel.

4.5.6.2 Initial and annual ethics and data integrity training.

4.5.6.3 Internal audits.

4.5.6.4 Anti-fraud language in client contracts and project agreements, where applicable.

4.5.6.5 Analyst notation and signature on manual integration changes to data and/or calculations.

4.5.6.6 Mandatory use of electronic and computer software audit functions wherever possible.

4.5.6.7 A no-fault policy that encourages employees to come forward and report fraudulent activities.

4.5.7 Employees are provided routine communications in the form of training, lectures, and updates in policy that are intended to reduce illicit behavior.

4.5.8 Any of the following means may be used to monitor the quality and validity of test results:

4.5.8.1 Internal quality control samples.

4.5.8.2 Interlaboratory comparisons or proficiency test studies.

4.5.8.3 Certified reference materials or internal quality control using secondary reference materials.

4.5.8.4 Replicate tests using the same or different methods.

4.5.8.5 Re-testing of retained samples.

4.5.8.6 Correlation of results for different characteristics of a sample.

4.5.9 Examples of inappropriate practices include the following:

4.5.9.1 Failure to properly record and preserve data: Analysts must be able to clearly demonstrate how analytical values were obtained from the associated raw data. Such documentation shall be maintained by the laboratory and be available to data users or auditors at any time. This includes failure to document data in the original logbook or on the original company form. Transferring data from a scratch paper or

note paper to the logbook or company form is never allowed. The data must be recorded in the appropriate document at the time the test or preparation is being performed. Failure to comply with this will result in disciplinary measures up to and including dismissal.

4.5.9.2 Failure to properly document errors: All errors, mistakes, and justifications for manual integrations must be fully explained within the case narrative of the final report.

4.5.9.3 Failure to initiate corrective actions: Analysts having knowledge of any part of an analysis or procedure that requires corrective action must immediately notify management.

4.5.9.4 Failure to report a missed holding time: Samples analyzed outside of allowed holding times must not be reported without qualifying the data, and some results may be unusable due to lack of validity. Backdating an analysis to save a missed hold time is forbidden.

4.5.9.5 Failure to follow methods or SOPs as written: Methods and standard operating procedures must be followed without deviation. Analysts must immediately submit any changes to the Technical Director for revisions.

4.5.9.6 Signing another person's signature to documentation.

4.5.10 Improper, unethical, and illegal actions are considered fraudulent because they affect the integrity of the data. Gross deviations from specified procedures will be investigated for potential improper, unethical, illegal actions and data integrity issues. Findings of fraud may be prosecuted to the fullest extent of the law. The following are examples of improper, unethical, and illegal conduct that affect data integrity:

4.5.10.1 Improper use of manual integrations to meet calibration or method Quality Control criteria, such as peak shaving or peak enhancement, if performed solely to meet QC requirements.

4.5.10.2 Falsification of results to meet method requirements.

4.5.10.3 Reporting of results without analyses to support the data or reporting results from the analysis of one sample for those of another.

4.5.10.4 Selective exclusion of data to meet QC criteria, such as dropping calibration points without technical or statistical justification.

4.5.10.5 Misrepresentation of laboratory performance by falsifying calibration data or QC.

4.5.10.6 Reporting QC limits in data reports that are not linked to the data set reported or to historical data.

4.5.10.7 Citing matrix interference as a basis for exceeding acceptance limits, especially without initiating corrective actions, in interference-free matrices.

4.5.10.8 Unwarranted manipulation of computer software such as subtracting or not subtracting a blank or background, altering chromatographic baselines, or improper background subtraction for GC/MS to comply with ion abundance criteria in order to meet QC requirements.

4.5.10.9 Improper alteration of analytical conditions, such as modifying an EM voltage or changing a GC temperature program to induce a shorter analytical run time, which makes the standard analysis different from the sample analysis.

4.5.10.10 Misrepresentation of QC samples, such as adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over-spiking, or under-spiking.

4.6 Undue Internal and External Pressures

4.6.1 AES, Inc. strives for the highest caliber of laboratory performance in conjunction with accomplishing quality objectives. One component of realizing this goal is to protect laboratory personnel from undue internal and external pressures.

4.6.2 At AES, Inc. analysts and technicians are insulated from work-related undue pressures that would compromise the quality of their work. Management is aware and considerate of these internal pressures such as management burdens and project deadlines, and of external stresses such as customer complaints and priority requests for analysis.

4.6.3 Management policy is to remain supportive of laboratory personnel and aware of their workloads and the demands placed upon them. Precautions are taken to ensure that there are no conflicts of interest between staff and clients. For example, priority requests, complaints, or status of work inquiries are directed through supervisors, managers, or administrative personnel.

4.6.4 Internal complaints and concerns expressed by employees are handled by AES' policy of encouraging free communication with all levels of management. An "open door" approach promotes avenues of communication that could prevent improper conduct or data integrity issues resulting from undue external and internal pressures. Reducing workload for individual employees may include assigning additional personnel to assist in heavily backlogged areas, providing additional support, supplies, or equipment, or affording technical assistance and resources.

4.7 Responsibilities

4.7.1 It is the responsibility of all AES employees to implement the Quality Assurance Program effectively. All chemists and technicians are responsible for understanding and following the measures of the QA program, and for reporting any quality failures to a Manager or Supervisor in a timely manner.

4.7.2 Supervisors and Managers are responsible for ensuring that all laboratory personnel are familiar with the requirements of the Quality Assurance Program and that these requirements are implemented and maintained. It is the responsibility of each Supervisor to ensure that any quality failures are reported to the Project Manager and the Quality Assurance Department immediately.

4.7.3 It is the responsibility of the Technical Director to ensure that all laboratory personnel are trained to perform their assigned analyses.

4.7.4 The laboratory's approved signatories and designees of the Technical Manager are identified as follows:

Laboratory Manager
Director of Project Management
Project Managers

Individuals are authorized as project manager report signatories based on meeting the qualifications of project manager job description in the QA Manual as well as completion of the following training:

Quality Assurance Manual
Legal & Ethical Training
PCM Asbestos Reports Training

Individuals are authorized to act as project manager report signatories when these documents have been completed and signed by the individual(s) and referenced managers.

5.0 QUALITY ASSURANCE PROGRAM

5.1 The Quality Assurance Program (QAP) has been developed to provide a high-quality document that complies with the intent of testing regulations, standards, and established guidelines. The QAP takes into account requirements for special controls, processes, test equipment and skills to attain the required quality and the need for verification of quality by inspection and test. It also provides for the training of personnel to attain required proficiency levels and for regular assessments of the QAP to assure the adequacy of resources and the effectiveness of management controls established to achieve quality. The Quality Manual is maintained in a current condition.

5.2 Revisions to this QAP are made and controlled by the QA Manager, Technical Director, and Vice-President of Operations in accordance with AES' quality assurance practices. Such revisions and updates shall be performed as needed to improve the effectiveness of this program. Control of this QA manual is accomplished following the requirements of Section 8.2, "Document Control".

5.3 Definitions (NOT ALPHABETICAL)

5.3.1 Batch – A group of samples and QC samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

5.3.1.1 Preparation Batch - is composed of between 1 and 20 samples of the same matrix and meets the criteria for a batch as described in Section 5.3.1. Preparation batches consist of extractions, digestions, or concentrations. The maximum time between the start of processing of the first and last sample in a preparation batch is 24 hours. A preparation batch must have a spiked sample and a duplicate sample (or matrix spike duplicate).

5.3.1.2 Analytical Batch - is composed of prepared environmental samples (extracts, digestates, or concentrates) or non-prepared environmental samples which are analyzed together as a group. When the batch contains non-prepared samples as a group, the rules for preparation batches must be followed.

5.3.1.2.1 Test categories where samples do not have to be prepared prior to analysis include GRO, VOC, Ion Chromatography, direct injection SVOC, orthophosphorus, turbidity, pH, and Conductivity.

5.3.1.2.2 When soil VOC or GRO samples arrive in ENCORES or in jars, they considered prepared when placed into water or methanol. Rules for preparation batches apply.

5.3.1.2.3 The maximum length of time that an analytical batch can be left open is 24 hours. An analytical batch may have no more than 20 samples of similar matrix.

5.3.1.2.4 Test procedures take precedence over analytical batch considerations. For example, if the test procedure identifies a batch as occurring over a 12 or 24 hour period, then batches may not be left open for the time period stated in Section 5.3.1.2.1.

5.3.1.2.5 Methanol or water VOC or GRO samples prepared in the laboratory from ENCORES or jars cannot be combined into a sequence with samples that have not been prepared by the laboratory so as to create a batch that contains more than 20 samples or runs for longer than 24-hours.

5.3.1.2.6 An analytical batch must include the analysis of a spiked sample and a duplicate sample (or matrix spiked duplicate) every 20 samples in the batch. In addition, internal quality control dictates that a LCS sample is also included in the batch.

5.3.1.2.7 Always analyze the quality control samples at the beginning of the analytical batch. Quality control samples include the MS, MSD, LCS, LCSD, MB, CCB, and CCV.

5.3.1.2.8 Always verify batch completion date in LIMS.

5.3.2 Accuracy – The nearness of a result or the mean (average) of a set of results as compared to the true value. Accuracy is assessed by means of reference samples, laboratory control spikes, matrix spikes, etc, and is measured in percent recovery.

5.3.3 Blank – An analyte-free matrix, usually reagent water or clean sand, designed to monitor the introduction of contaminants either during the performance of a test or during sampling and transportation activities. Except for certain conditions listed below, all analytes associated with the blank must have concentrations less than the reporting limit.

5.3.3.1 The reporting limit may be raised above the level of contamination in the method blank and associated samples with documentation of client approval.

5.3.3.2 Sample results are 10 times the concentration of the method blank. The data may be reported with a flag indicating that low level contamination was detected in the method blank. Report data with a “B” qualifier.

5.3.3.3 There are several types of blanks. The various types are defined below.

5.3.3.3.1 Calibration Blank – specified in some analytical procedures, is an aliquot of analyte-free matrix used to establish a zero-concentration instrument response value.

5.3.3.3.2 Method Blank – an aliquot of analyte-free matrix, usually reagent water, to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document the absence of contamination resulting from the analytical process. A method blank may also be referred to as a reagent blank or preparation blank, depending on the procedure.

5.3.3.3.3 Field Blank – also called an equipment blank. A field blank is an aliquot of analyte–free water brought to the field in sealed containers, transferred to a sample container, and transported back to the laboratory with the samples to be analyzed. The field blank is used to evaluate any possible contamination introduced to the samples during the field collection process.

5.3.3.3.4 Trip Blank – an aliquot of analyte-free water which accompanies the empty containers to the field and the collected samples back to the laboratory. The trip blank is an indicator of possible sample contamination originating from site conditions and sample transportation.

5.3.4 Initial Calibration Verification (ICV) Standard. An ICV is a standard that has been prepared from a source that is not the same as the source used for the preparation of the calibration curve. A second source represents either, a different lot number of standard purchased from the same vendor, or the same standard purchased from a second vendor. ICV standards are not prepared using the same procedures as samples (e.g., digestions or extractions). The individual test methods describe the preparative procedures and suppliers for these standards. ICV standards are analyzed immediately after a successful calibration curve has been developed. Typically, the ICV standards are prepared so that their concentrations represent a midpoint of the calibration curve.

5.3.5 Continuing Calibration Verification (CCV) Standard. A CCV is a standard that has been prepared from the same source as the calibration standards. CCV standards are not prepared using the same procedures as samples are prepared (e.g. digestions or extractions). The individual test methods describe the preparative procedures and suppliers for these standards. CCV standards must be analyzed every 10 samples throughout the analytical batch, and at the beginning and end of the analytical batch.

5.3.6 Laboratory Control Sample (LCS) – Typically prepared by spiking an aliquot of reagent water or analyte–free soil with the analyte(s) of interest. The LCS is prepared and analyzed employing the same methodology as the associated samples. The LCS is used to monitor, assess, and control the laboratory's performance of the methods employed for sample preparation and analysis. The LCS must be performed once per analytical batch, extraction batch, or digestion batch. An extraction or digestion batch is defined as twenty or fewer samples of similar matrix analyzed in a 24-hour period using similar preparative and/or

extraction techniques. In many cases, a duplicate LCS sample (LCSD) will be analyzed along with the LCS.

5.3.7 Deionized Water (DI Water, DIW) – Reagent free water that is prepared by passage through various filters and membranes.

5.3.8 Environmental Sample – An environmental sample or field sample is a representative portion of any matrix (aqueous, non-aqueous, mixed waste, etc.) collected from any source for which the determination of the composition of the contamination is requested or required. For the purpose of this procedure, environmental samples are classified as follows:

5.3.8.1 Aqueous – Aqueous samples consist of surface water, ground water, drinking water (either treated or untreated), or wastewater. Wastewater consists of municipal and industrial influents and effluents.

5.3.8.2 Soils – Soil samples consist of sediments, soils, and sludges.

5.3.8.3 Non-Aqueous Liquids – Non-aqueous liquids consist of solvents, oils, and fuels. These sample types are not miscible with aqueous samples.

5.3.8.4 Non-Soil Solids – Non-soil solids consist of solid waste, precipitate waste, industrial sludges, concrete, wood, paint chips, ash, and wipes.

5.3.8.5 Bioassay – Bioassay samples consist of bio-solids and municipal waste treatment sludges.

5.3.8.6 Air – Air samples consist of filters, absorbent traps, activated carbon, and passive monitors used in the collection of air samples. Additionally, air samples can be collected in SUMMA canisters or Tedlar bags. In these two cases, the sample is the air itself.

5.3.9 External Quality Control – Those practices that monitor the quality of data from sources outside the control of the laboratory (e.g. multi-laboratory performance evaluation samples and external audits).

5.3.10 Instrument Detection Limits (IDL) – The minimum concentration limits of an analyte above the instrument noise level that can be detected and quantified with a high degree of confidence (>95%).

5.3.11 Internal Quality Control – Those practices implemented internally to monitor the quality of data and which are under the control of the laboratory (i.e. intra-laboratory performance samples, internal audits, single blind samples, etc.)

5.3.12 Matrix Spike / Matrix Spike Duplicate (MS/MSD) – An environmental sample to which predetermined quantities of specific analytes are added prior to sample preparation and analysis. Percent recoveries are calculated for each of the spiked analytes to assess the effect of the matrix on analyte recovery. In addition, a calculation of precision is made between the results of the MS/MSD to determine reproducibility of results in a specific matrix. This is measured by either the Relative Percent Difference (RPD) or Percent Relative Standard Deviation (%RSD). MS and MSD samples are analyzed with each analytical, extraction, or

digestion batch of up to 20 samples. MS and MSD precision and accuracy limits are developed from quality control data.

5.3.13 Method Detection Limits (MDL) – The minimum concentration of a substance that can be measured and reported, in a specific matrix, with 99% confidence that the analyte is present at a concentration greater than zero. The MDL is calculated by analyzing a minimum of seven replicates prepared in blank water or soil at concentrations that are 1 to 5 times higher than the estimated detection limit. The MDL study is performed on each matrix for each analyte that is analyzed in the laboratory. Samples prepared for the MDL study are made from a standard that has been prepared from the same source that was used for the preparation of the calibration curve. MDLs are laboratory derived from the MDL study data set. MDL studies are to be updated annually, or whenever instrument conditions change in a manner that will affect the established detection limits.

5.3.14 Precision – The agreement of a set of replicate results. Typically, the laboratory analyzes LCS and LCSD or MS and MSD samples and reports the results as RPD or %RSD.

5.3.15 Practical Quantitation Limit (PQL) – The lowest analyte concentration that can be reliably achieved, within specified limits of precision and accuracy, during routine laboratory operating conditions. The term PQL may be inter-changed with the term Reporting Limit (RL). Practical Quantitation – Is used synonymously with 'Reporting Limit'. The quantitation limits are tied to the detection limits in that the PQLs are never less that MDLs and a low level standard is analyzed at the PQL where applicable.

5.3.16 Qualifiers – A phrase or word group that limits or modifies the meaning. (See section 12.5.4)

5.3.17 RCRA – Resource Conservation Recovery Act

5.3.18 Relative Percent Difference (RPD) – A measure of agreement between two replicate results, expressed as follows:

$$\text{RPD} = 100 * \frac{X_1 - X_2}{\overline{X}}$$

where: X_1 and X_2 = the two results

$\overline{X}$ = mean value of the results

5.3.19 Relative Standard Deviation (RSD) – The variance from the mean or true value divided by the mean or true value, expressed as a percentage.

$$\% \text{ RSD} = 100 * S/\overline{X}$$

where:

$\overline{X}$ = arithmetic mean of the measurements

S = variance

5.3.20 Representativeness - The degree to which data represent a characteristic of a population or set of samples. It is a measurement of both analytical and field sampling precision.

5.3.21 Standard Curve - A curve, which plots known standard concentrations or amounts of an analyte versus the instrument response for the analyte. This curve is used to determine the concentration of the analyte in the unknown samples.

5.3.22 Surrogate - Organic compound(s) which is/are similar to analytes of interest in chemical composition, extraction efficiency, and chromatographic retention, but are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate to assess the effectiveness of the sample preparation and analysis and any potential matrix effects.

5.3.23 TNI – The NELAC Institute

5.3.24 AIHA – American Industrial Hygiene Association

5.3.25 Method of Standard Additions – The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.

5.3.26 **Estimation of Uncertainty** - is the parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurement. A reasonable 'Estimation of Uncertainty' shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data. It is monitored by the monthly checks, proficiency exam results and error rates. The estimate of day-to-day precision is determined by comparison of duplicate samples (or matrix spike duplicates). Results of the two analyses are compared by their relative percent difference, RPD: (A-B) / (Average of A and B)

Estimation of Uncertainty Limits may be method / program specified (e.g. AIHA ELLAP) or based on historical laboratory limits. Interim limits are used until enough data points have been generated to set representative limits. The following table identifies Estimation of Uncertainty used by the lab. The actual limits can be seen in LIMS test codes or posted on the portal server.

5.3.26.1 AIHA Uncertainty and Uncertainty Limits Determinations
All analytical values measured are affected by some degree of variability and bias. This uncertainty is the sum total of all errors introduced throughout the use of the procedure. The range of uncertainty (i.e. control limit) must, therefore, be determined for each parameter/procedure. These ranges or limits define the degree of variability between identical measurements as precision and the closeness to the true value as accuracy. Bias is included in the accuracy limits as ranges are centered around the mean recovery. Uncertainty or control limit determinations specific to each type of testing for AIHA programs is defined below.

5.3.26.1.1 Industrial Hygiene Chemical/Gravimetric Analysis. Ranges of uncertainty i.e. control limits, for IH chemical testing are determined for precision and accuracy. Generally, precision limits are determined using historical RPD values for each procedure/target analyte. Once at least 50 RPD values are

available, the mean and standard deviation of the data set are calculated. The data is evaluated for outliers using standard Grubbs Outlier calculations with all statistical outliers omitted. Control limits for precision are defined as "0"-3x the standard deviation of the RPD values. Generally, accuracy limits are determined using historical % recovery values for each procedure/target analyte. Once at least 50 % recovery values are available, the mean and standard deviation of the data set are calculated. The data is evaluated for outliers using standard Grubbs Outlier calculations with all statistical outliers omitted. Control limits for accuracy are defined as plus or minus 3x the standard deviation from the mean value of the % recovery values. Because accuracy limits are centered around the mean value, bias is included in these limits. Where target analyte spiking is not applicable such as for gravimetric testing, only precision limits are used for uncertainty determinations. If less than 50 points are available for calculation, interim limits are used. Specific information used for control limits for each individual IH test method are provided in Table 5.

5.3.26.1.2 Industrial Hygiene Asbestos by PCM Analysis. Ranges of uncertainty for IH asbestos by PCM testing are determined for precision only using daily reference slide and blind recount analyses as described below.

5.3.26.1.2.1 Daily reference slides are analyzed from two sources, AIHA PAT samples and representative client samples. For the PAT samples used as reference slides, the documented acceptance range for that PAT round is used as range of uncertainty to evaluate acceptability of the count for the individual analyst. For the representative client samples used as reference slides, uncertainty is evaluated using the Sr values as described in Method N7400.

5.3.26.1.2.2 Inter-analyst and intralaboratory precision acceptability ranges are calculated using Strontium values as described in Method N7400.

5.3.26.1.3 Environmental Lead Analysis for reporting under the ELLAP Program. Ranges for uncertainty, i.e. control limits, for ELLAP testing for precision and accuracy are as specified in Table 2C-1 in the AIHA LQAP Policy Module 2C. Specific information used for control limits for each individual ELLAP test method is provided in Table 5.

5.3.26.1.4 Quantifiable Fungal Analysis for reporting under the EMLAP Program. Ranges for uncertainty for quantifiable fungal testing are determined for precision only. Duplicate samples are counted for at least 5% of samples for inter-analyst precision monitoring and replicates samples are counted by different analysts for intra-analyst precision monitoring. Uncertainty ranges (control limits) are determined using the mean of the range of the logarithm of each count obtained from a minimum of 20 duplicate/replicate pairs. This mean value is multiplied by 3.27 to obtain the final control limit. Once the control limit is determined, the logarithmic range for each ongoing duplicate/replicate pair is determined and must be < control limit value.

Specific information used for control limits for each individual EMLAP test method are provided in Table 5.

5.3.26.1.5 Qualitative Fungal Analysis for reporting under the EMLAP Program. In order to monitor consistency with regard to genus/species identification, acceptability criteria for taxon identification and taxon abundance ranking are described below. These are laboratory determined; interim criteria as no regulatory guidance or method specified criteria are available.

5.3.26.1.5.1 Taxon identification acceptability: On the replicate and duplicate analyses, daily reference slide analyses, monthly reference culture analyses and round robin study analyses with at least 3 different organisms present, 60% of all genus/species of fungi and/or genus/group of fungi identified on the original sample at levels >10x LOD should also be identified on the recount.

5.3.26.1.5.2 Taxon abundance ranking acceptability: On the replicate and duplicate analyses, daily reference slide analyses and round robin study analyses, the top three genus/species of fungi and/or genus/group of fungi by abundance and >10x LOD will be ranked. The recount data should identify these same fungi for the identification to be considered acceptable.

5.3.26.1.5.3 Consistent fungal ID is also monitored through participation in the Direct Exam and Cultureble Fungal Analysis PT programs administered by EMLAP. Acceptability limits are currently set at 85% correct identification by AIHA.

5.3.26.1.5.4 It should also be recognized that other, non-quantifiable factors may also add additional uncertainty. These factors may include media selection, organism competition, etc. and are not directly measurable.

Estimation of Uncertainty Requirements

Method	Uncertainty Based On
NELAP METHODS	
E110.2 Color	NA
E120.1 Conductivity	Method Limits
E150.1 pH	NA
E160.1 TDS	NA
E160.2 TSS	NA
E160.3 TS	NA
E160.4 VS	NA
E160.5 Settleable Solids	NA
E1664 Oil and Grease_TPH	Method Limits
E180.1 Turbidity	Method Limits
E200.7 ICP AES Metals	Method Limits

E200.8 ICP MS Metals	Method Limits
E245.1 Mercury	Method Limits
E300 Anions by IC	Method Limits
E305.1 Acidity	NA
E310.1 Alkalinity	Method Limits
E310.2 Alkalinity	Method Limits
E325.2 Chloride	Method Limits
E330.5 Residual Chlorine	Method Limits
E335.1 Amenable Cyanide	Method Limits
E335.2 Total Cyanide	Method Limits
E335.4 Total Cyanide	Method Limits
E350.1 Ammonia	Method Limits
E351.2 TKN	Method Limits
E353.2 Nitrate_Nitrite	Method Limits
E354.1 Nitrite	Method Limits
E360.1 Dissolved Oxygen	NA
E365.1 Ortho Phosphorus	Method Limits
E365.1 Total Phosphorus	Method Limits
E365.3 Ortho Phosphorus	Method Limits
E370.1 Dissolved Silica	Method Limits
E375.4 Sulfate	Method Limits
E376.1 Sulfide	Method Limits
E377.1 Sulfite	NA
E405.1 BOD	Method Limits
E410.4 COD	Method Limits
E415.1 TOC	Method Limits
E420.1 Total Phenolics	Method Limits
E420.2 Total Phenolics	Method Limits
E425.1 MBAS Surfactants	Method Limits
E608 Pesticides PCBs	Method Limits
E608.2 Methoxychlor	Method Limits
E610 PAHs	Method Limits
E615 Herbicides	Historical Limits
E624 VOCs	Method Limits
E625 SVOCs	Method Limits
FL-PRO	Method Limits
RSK-175 Dissolved Methane, Ethane, Ethene	Method Limits
SM10200H Chlorophyll	Historical Limits
SM2340B Hardness	Method Limits
SM2540G Total, Fixed and Volatile Solids	NA
SM2710B SOUR	NA
SM3500-Cr D Hexavalent Chromium	Method Limits

SM3500-Fe D Ferrous Iron	Method Limits
SM5210B CBOD	Method Limits
SM9222B Total Coliforms	NA
SM9222D Fecal Coliforms	NA
SM9223B Total Coliforms and E. Coli	NA
SW1010 Flash Point	NA
SW1311 TCLP	Historical Limits
SW1312 TCLP	Historical Limits
SW6010 ICP AES Metals	Method Limits
SW6020 ICP MS Metals	Method Limits
SW7.3 Reactive Cyanide	Method Limits
SW7.3 Reactive Sulfide	Method Limits
SW7196 Hexavalent Chromium	Method Limits
SW7470 Mercury in Water	Method Limits
SW7471 Mercury in Soils	Method Limits
SW8011 EDB DBCP	Historical Limits
SW8015 DAI	Historical Limits
SW8015 DRO	Historical Limits
SW8015 GRO	Historical Limits
SW8081 Pesticides	Historical Limits
SW8082 PCBs	Historical Limits
SW8151 Herbicides	Historical Limits
SW8260 VOCs	Historical Limits
SW8270 SVOCs	Historical Limits
SW8310 PAHs	Historical Limits
SW8315 Formaldehyde	Historical Limits
SW9010_9012 Cyanide	Method Limits
SW9010_9014 Cyanide	Method Limits
SW9030_9034 Sulfide	Method Limits
SW9038 Sulfate	Method Limits
SW9040 pH in Water	NA
SW9041 pH by Paper	NA
SW9045 pH in Soil	NA
SW9050 Conductivity	Method Limits
SW9056 Anions by IC	Method Limits
SW9060 TOC	Method Limits
SW9065 Total Phenolics	Method Limits
SW9070 Oil and Grease_TPH in Water	Method Limits
SW9071 Oil and Grease_TPH in Soils	Method Limits
SW9081 Cation Exchange Capacity (Sodium)	NA
SW9095 Free Liquids by Paint Filter	NA

Method	Uncertainty Limits Based On*
AIHA METHODS	
3M3520/SKC575(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1003(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1300(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1400(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1450(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1457(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1500(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1501(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N1550(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N2000(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N2500(GC/FID)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N5506(HPLC)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N7300(ICP except lead)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N6009(AA Hg)	Precision: Historical RPD values for LCSD Accuracy: Historical %Rec values for LCS/LCSD
N7082(Lead Paint)	Accuracy: Table 2C-1 AIHA LQAP Policy Module 2C Precision: Table 2C-1 AIHA LQAP Policy Module 2C
SW3050B/7420(Lead in Soil)	Accuracy: Table 2C-1 AIHA LQAP Policy Module 2C Precision: Table 2C-1 AIHA LQAP Policy Module 2C
SW3050B/7000B(Lead in Soil)	Accuracy: Table 2C-1 AIHA LQAP Policy Module 2C Precision: Table 2C-1 AIHA LQAP Policy Module 2C
N9102(Lead in Dust Wipe)	Accuracy: Table 2C-1 AIHA LQAP Policy Module 2C Precision: Table 2C-1 AIHA LQAP Policy Module 2C
N7300(Lead in Air)	Accuracy: Table 2C-1 AIHA LQAP Policy Module 2C Precision: Table 2C-1 AIHA LQAP Policy Module 2C
N7400(Asbestos PCM)	Accuracy: NA Precision: PAT acceptance ranges and/or Sr values from Daily Reference Slides (both analyst specific and laboratory wide)
N0500/0600(Particulates)	Precision: Historical RPD values for LCSD Accuracy: NA

Fungal Air Culturable(Micro)	Accuracy: See Sec. 1.1.5 Precision: See Sec. 1.1.4
Fungal Bulk Culturable(Micro)	Accuracy: See Sec. 1.1.5 Precision: See Sec. 1.1.4
Fungal Surface Culturable(Micro)	Accuracy: See Sec. 1.1.5 Precision: See Sec. 1.1.4
Fungal Air Direct Exam(Micro)	Accuracy: See Sec. 1.1.5 Precision: See Sec. 1.1.4

Fungal Bulk Direct Exam(Micro)	Accuracy: See Sec. 1.1.5 Precision: See Sec. 1.1.4
Fungal Surface Direct Exam(Micro)	Accuracy: See Sec. 1.1.5 Precision: See Sec. 1.1.4

5.3.27 Interim Limits - are used to establish the level of uncertainty when limits are not available until enough laboratory data has been compiled to establish historical limits. Interim limits may be derived from published methods, those limits within similar analysis, LCS recovery ranges, or based on reasonable expectations from laboratory experience.

5.3.28 Measurand - Quantity intended to be measured or analyte concentration.

5.3.29 Type A evaluation of measurement of uncertainty - Evaluation of a component of measurement uncertainty by a statistical analysis of measured quantity values obtained under defined measurement conditions.

5.4 Data Quality Objectives

5.4.1 Precision. The laboratory objective for precision is to meet the performance criteria demonstrated for all analytical methods as published by the USEPA under SW-846 and 40 CFR Part 136. These criteria are met on similar samples and similar sample matrices. Precision is documented based on replicate analysis, usually duplicate or matrix spike duplicate samples.

5.4.2 Accuracy. The laboratory objective for accuracy is to meet the performance criteria demonstrated for these analytical methods as published by the USEPA under SW-846 and 40 CFR Part 136. These criteria are met on similar samples and similar sample matrices. Accuracy is documented based on recovery data; usually matrix spike samples.

5.4.3 Bias. Bias is incorporated into limits of uncertainty as control limits are centered around mean recovery.

5.4.3 Representativeness. The laboratory objective for representativeness is to provide data which is representative of the sampled medium. The representativeness of the analytical data is a function of the procedures used in processing the samples.

5.4.4 Comparability. The comparability objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar in quality to data generated by other laboratories for similar samples and to data compiled by AES over time. The comparability objective may be documented by any of the following:

5.4.4.1 Inter-laboratory studies carried out by regulatory agencies.

5.4.4.2 Inter-laboratory studies initiated for specific projects or contracts.

5.4.4.3 Comparison of periodically generated statements of accuracy, precision, and reporting limits to those of other laboratories.

5.4.4.4 Through approval from the US EPA or other regulatory agencies for any procedure to which significant modifications have been made.

5.4.5 Completeness. The completeness objective for data is can be set for a particular project and is expressed as the ratio of the valid data to the total data over the course of the project.

5.5 Criteria for Quality Indicators

5.5.1 The precision and accuracy acceptability limits for analyses performed at Analytical Environmental Services, Inc are located in the LIMS and posted on the portal server. The limits in the tables are either laboratory-generated or derived from USEPA methods.

5.5.2 Table 5-6 defines the criteria for data acceptability. Data may be accepted when QC falls outside these limits if probable cause can be attributed to the matrix, and laboratory control samples (LCS) show that the method is in control. Deviations are documented in the final report to the client.

In instances where an LCS limit is not available, a limit of 30-130% recovery may be used until in-house limits are available. (Note: Sometimes an alternative default limit may be found in a published method and substituted.) In some cases, lower default limits may be set with approval from the Quality Assurance Manager and Technical Director. The acceptable range of some compounds may be broader, based on prior knowledge of the analyte (e.g., phenols in EPA Method 8270C).

5.5.3 Statistically Derived Limits

5.5.3.1 Selected methods and programs require statistically derived accuracy and precision limits. Analytical Environmental Services, Inc. routinely uses statistically derived limits to evaluate method performance and to determine when corrective action is appropriate.

5.5.3.2 The laboratory periodically updates the limits as stated, but no less than annually. Analysts must use the current limits as found in LIMS.

5.5.3.3 The QA Manager maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used. If a method requires the generation of historical limits, they can be derived from recent data in the LIMS database or by viewing older limits within the database.

5.5.4 Development of new QC limits.

5.5.4.1 The QA Manager determines limits using the in-house LIMS system. This is accomplished by the statistical analysis of data for each test method where the method specifies that internal limits are developed. The table below indicates the methods that are updated annually.

QA Limit Update Requirements

Test Method	Required Update	Test Method	Required Update
Petroleum Hydrocarbons	Not required - Test specified limits	Wet Chemistry	Not required - Test specified limits
EPA 608	Determine spike limits (wastewater). Surrogates not required	EPA 610	Determine spike limits (wastewater). Surrogates not required
EPA 624	Determine spike limits (wastewater – specified limits for other aqueous matrix). Surrogates required, but no limits specified	EPA 625	Determine spike limits (wastewater – specified limits for other aqueous matrix). Surrogates required, but no limits specified
Organics by SW-846 8000 methods	Determine surrogate, spike, LCS Limits	Metals by 6000 methods	Determine spike, spike duplicate limits or use test limits
Metals by 7000 methods	Not required - Test specified limits	Metals by EPA methods	Not required - Test specified limits
CLP methods	Not required - Test specified limits	Inorganics by SW-846 9000 methods	

5.5.4.2 Data types within the methods that are reviewed include LCS, LCSD, MS, MSD, and surrogates in samples, control samples, and spikes. It is recommended that surrogates are evaluated on a separate basis for samples, LCS, and MS since recovery limits will be wider for client samples than for laboratory control samples.

5.5.4.3 QC limits are updated in LIMS through the Quality Control Section. To change limits, activate the tab called “control charting”. Enter the desired test code, analyte, and sample type. Enter the number of desired data points, and then “get data”.

5.5.4.4 The minimum number of data points chosen should be 20. For tests which data is generated more frequently, e.g. volatile surrogate recoveries in samples, a minimum of 40 data points should be chosen.

5.5.4.4.1 For tests in which there are less than 20 data points, use the interim limits specified by the method. If interim limits are not specified by the method, the QA Manager and Technical Director must choose interim limits that represent an estimation of the current laboratory performance. The data in the tables should be footnoted accordingly.

5.5.4.4.2 For tests in which data is generated more frequently, e.g. volatile surrogate recoveries in samples, a minimum of 40 data points should be chosen. The LIMS will pick data points in historical order beginning with the date the action is being performed. The LIMS will compile as many data points are available if the requested number exceeds the number of points in LIMS

The LIMS will pick data points in historical order beginning with the date the action is being performed. If the requested number exceeds the number of points in LIMS, then LIMS will compile as many data points as are available.

5.5.4.5 Data should be observed for outliers, and these samples de-selected using the "radio buttons". Once the data is reviewed, limits can be recalculated by choosing the "Re Calc Stats" tab. Outlying data points are determined by the following two methods:

5.5.4.5.1 Grubbs Test

5.5.4.5.2 Manual observation of data set to verify that the data points selected are within the calculated control limits. If they are not, then the data points must be "de-selected" and the limits recalculated until the data is within the calculated limits.

5.5.4.6 The lower limit determined from historical data shall not be set to a value less than 10. That is, if the calculated lower limit is < 10, a default value of 10 will be used for the lower limit unless specified by the published method.

5.5.4.7 When the data set is acceptable, choose the "Preview" tab to view data in a page format. Through the "Windows" application, print the data in "Adobe" format by selection of the proper network printer. The file should be saved in one of the following folders depending on which QC type:

TestMethod_Matrix_LCS_LCSD_REC
TestMethod_Matrix_LCS_LCSD_RPD
TestMethod_Matrix _MSD_REC
TestMethod_Matrix _MSD_RPD
TestMethod_Matrix_SURR_REC

5.5.5 Review of revised QC limits

5.5.5.1 After data has been revised for each test method and matrix, a copy of the QC Tables and charts is presented to the department managers, Technical Director, and Vice President of Operations for review. After a week comment period, the updated limits are entered into the laboratory LIMS system.

5.6 External Quality Assurance Objectives

5.6.1 External Quality Control is the process of employing outside sources to monitor the quality of the data produced by the laboratory. Included in the external quality control program are the analysis of performance evaluation samples and participation in performance evaluation audits.

5.6.1.1 AES, Inc. analyzes Proficiency Test (PT) samples for each PT field of testing as defined in The NELAC Institute (TNI) and AIHA Fields of Test tables according to matrix type, analyte, and regulatory or environmental program. Samples are obtained from NELAP-designated PTOB/PTPA-approved PT providers (such as Environmental Resource Associates) for NELAP compliance or directly from AIHA to meet their program requirements. The results of the analyses are submitted to the PT Provider for scoring. Study reports are maintained for a minimum of five consecutive years in the QA Office. The analyses of PT studies

are conducted in accordance with all TNI or AIHA. Where required (as with gravimetric analyses for AIHA), an internal PT will be used.

5.6.1.1.1 AES participates in a minimum of two single-blind, single-concentration PT studies per year for each PT field of testing for which accreditation is maintained. Studies are performed at least 15 calendar days apart. Successful completion of two of the last three successive proficiency rounds for a given PT field of testing must occur in order to maintain accreditation.

5.6.1.1.2 The blind water or soil PT samples contain amounts of specific constituents that are unknown to laboratory personnel. Upon arrival, PT samples are logged into the Laboratory Information Management System (LIMS) and tracked as routine environmental samples. PT samples provided by the vendor may be 'whole' samples or may have been provided in a concentrated form. PT vendor instructions are followed and dilutions performed on the concentrated vials to make them the 'whole' sample to be tested. Routine procedures for dilutions and analysis are followed per method specific SOPs. The laboratory results must be completed and reported within the required turn around time.

5.6.1.1.3 AES, Inc. maintains copies of all written, printed, and electronic records, including, but not limited to bench sheets, instrument strip charts and chromatograms or printouts, data calculations, and data reports resulting from the analysis of any PT sample. These records are maintained for five years or for as long as required by the applicable regulatory program, whichever is greater. These records include a copy of the PT study report forms used to report PT results. All laboratory records are available to assessors of the Primary Accrediting Authority during on-site audits.

5.6.1.1.4 Whenever a study is failed, AES determines the cause for the failure and takes the necessary corrective actions. The investigation and action taken are documented into QA records and provided, if required, to the Primary Accrediting Authority.

5.6.1.2 Performance evaluation samples are also obtained from the following list of suppliers.

5.6.1.2.1 ELPAT. This proficiency testing program is administered by the American Industrial Hygiene Association (AIHA). Once a quarter, the laboratory receives a set of proficiency samples from Research Triangle Institute for the analysis of lead content. The matrices are soils, wipes, and/or paint chips.

5.6.1.2.2 PAT. This proficiency testing program is administered by the American Industrial Hygiene Association (AIHA). Once a quarter, the laboratory receives a set of proficiency samples to be analyzed for metals, asbestos fibers, and organics. Sample matrices are 37mm filters for metals, 25mm filters for asbestos and charcoal, or silica tubes for organics. This program is required as part of the laboratory's certification to perform analyses on samples that measure indoor air quality.

5.6.1.2.3 EMPAT. This proficiency testing program is administered by the American Industrial Hygiene Association (AIHA). EMPAT fungal proficiency samples are available for both the 'Direct Examination' and the 'Laboratory Culture' categories. Once a quarter, the laboratory receives notification that the Fungal Direct Examination Proficiency Testing Program has opened on the AIHA website. The lab has access to the portal for 24 hours a day for 7 days at which time the study closes. This program requires the identification of selected slides within a set amount of time. In addition, the lab receives culturable samples and a blank three times a year. These samples are prepared according to instructions and cultured prior to examination.

5.6.1.2.4 North Carolina Department of Environmental, Health and Natural Resources. Once a year the laboratory receives performance samples for certification by North Carolina for all analyses not already submitted under other programs. These samples are critical for the continuation of certification by the state of North Carolina.

To renew certification each year, the lab must submit acceptable PT sample results to the NC WW/GW LC Program for each parameter, analyte, technology and matrix (where a method is matrix-specific) by October 31.

A laboratory that fails a PT sample for a parameter method technology must take steps to identify the root cause of the failure, take corrective action, report the corrective action taken to NCDENR, and participate in a second PT study meeting the criteria listed previously in this policy. The corrective action response must include the laboratory's root cause analysis and a copy of any objective evidence (e.g., calibration curves, revised procedures, records, training records, standard operating procedures, etc.) to indicate that the corrective actions have been implemented/completed. The results of the remedial PT must be received in this office within 60 days from the date the failed results are issued by the accredited proficiency testing provider. A laboratory failing the second (or remedial) PT study may be decertified for that parameter method technology (not necessarily for all technologies for that parameter).

For multi-analyte parameters (e.g., organic analyses), when greater than 80% of analytes are acceptable, but one or more individual analytes are graded unacceptable, acceptable performance has been demonstrated for the parameter method technology. The laboratory must, however, analyze a remedial PT for the individual analytes that were graded unacceptable. When a remedial PT is graded unacceptable for an individual analyte (constituting a second unacceptable result), the laboratory must qualify data for those individual analytes as "estimated" (whether detected or not) until acceptable results are obtained on two consecutive remedial PTs for the analyte in question.

5.6.1.3 Performance Audits

5.6.1.3.1 In order to maintain certification in many states, to comply with commercial contracts, and to satisfy many agency requirements, AES, Inc. must undergo initial and ongoing audits performed by external auditors. These audits may take the form of technical and/or evidentiary audits. Every section of the laboratory, both analytical and clerical, should be ready at all times to participate in these audits.

5.6.1.3.2 In the event that adverse findings or deficiencies are discovered, or observations and/or recommendations are made during an audit, QA and laboratory management shall review the comments and submit a response, including corrective actions, to the audit report.

5.6.1.4 State Audits

5.6.1.4.1 State Audits are performed in accordance with each individual state's certification program. These audits are generally performed to determine the laboratory's suitability to perform environmental analyses according to the parameters dictated by that state.

5.6.1.5 Commercial Audits

5.6.1.5.1 Audits performed by commercial clients may be scheduled on a pre-award basis for a contract. Once the contract is awarded, audits may be scheduled at the request of the client or at a pre-determined frequency. The client, as well as professional audit teams, may perform audits required by commercial clients.

5.7 Internal Quality Control

5.7.1 The internal quality control program serves two primary functions. One function is to monitor the reliability of the data (e.g., accuracy and precision). The other function is to control and maintain the quality of the data (e.g., the use of ACS grade reagents, traceable standards, etc.).

5.7.2 The following sections outline the specific actions and procedures employed to monitor the process for producing and reporting quality data that is consistent with the Quality Control Program. Processes such as, but not limited to, verification of operator competence, recovery of known spikes, analysis of reagent blanks, calibration with traceable standards, analysis of duplicates, and maintenance of quality control charts must be employed and continually monitored. The laboratory may also adopt additional quality assurance procedures; however, the minimum requirements are discussed below. The QA Manager and Technical Director, under restrictions by the methodology and in conjunction with the appropriate laboratory management staff, shall determine which requirements shall be implemented for each section.

5.7.3 Training and Certification of Operator Competence. Quality Control begins with the establishment of basic laboratory techniques and skills. It is imperative that analysts receive proper training before performing independent laboratory analyses. Each analyst must demonstrate proficiency of laboratory techniques and skills. Records to that effect are kept in the employee's personal training files.

5.7.4 Documentation. Regardless of which analytical procedures are used in the laboratory, the methodologies employed shall be carefully documented.

5.7.4.1 Standard Operating Procedures (SOPs) and approved methods may be periodically modified, updated, or replaced in their entirety due to advances in technology, regulatory protocols, or at the discretion of laboratory management. All proposed changes, however, are reviewed by the Technical Director to ensure compliance with all regulatory protocols.

5.7.4.2 If a client requests a change of procedure, the change must be pre-approved by the laboratory prior to use. The change must be documented in writing and kept on file as part of the laboratory project records.

5.7.4.3 If a method is modified such that it no longer complies with the provisions set forth by the accrediting agencies, the client will be informed.

5.7.4.4 Documentation of analytical procedures for generating laboratory data shall be clear, concise, adequately referenced, and reflect the actual steps employed by the analyst.

5.7.5 Standard Operating Procedures (SOP). Methodologies employed in the laboratory are documented in SOPs. See Chapter 8 gives detailed information on SOPs.

5.7.6 Initial Calibration Verification (ICV) Standard. Individual component recovery of the ICV standard is calculated using the following equation:

$$\text{ICV Standard Percent Recovery} = \frac{A}{T} x100$$

where:

A = concentration measured
T = true value of the spiking concentration

5.7.6.1 The ICV must be made from a different source than the calibration curve standards.

5.7.6.2 The acceptable recovery limits for the ICV standards vary based on the individual procedure and are specified in Table 5-6**.**

5.7.6.3 If the recoveries of any of the ICV standards are not within the limits specified in Table 5-6, the test method may not be performed. The analyst must follow the out-of-control procedures discussed in Section 5.8 before initiating any analyses.

5.7.7 Continuing Calibration Verification (CCV) Standard. Individual component recovery of the CCV standard is calculated using the following equation:

$$\text{CCV Standard Percent Recovery} = \frac{A}{T} x100$$

where:

A = concentration measured
T = true value of the spiking concentration

5.7.7.1 The acceptable recovery limits for the CCV standards are procedure dependent and are specified in Table 5-6.

5.7.7.2 If the recoveries of any of the CCV standards are not within the limits specified in Table 5-6, the testing must be discontinued. The analyst must follow the out-of-control procedures discussed in Section 5.8 before continuing any analyses.

5.7.8 The Laboratory Control Sample (LCS)

5.7.8.1 The individual test methods describe the preparative procedures and suppliers for the LCS and LCSD standards. The LCS and LCSD samples are prepared in either reagent grade water or sand in accordance with the procedural steps followed for the preparation of a matrix spike sample.

5.7.8.2 Individual component recovery of the LCS(D) is calculated using the following equation:

$$\text{LCS (LCSD) Spike Percent Recovery} = \frac{A}{T} x100$$

where:

A = concentration measured
T = true value of the spiking concentration

5.7.8.3 Precision between the LCS and LCSD recoveries is calculated using the following equation:

$$\% \text{ RPD} = \frac{\text{Difference between LCS and LCSD recoveries}}{\text{Average of LCS and LCSD recoveries}} \times 100$$

5.7.8.4 The acceptable recovery limits for the LCS standards vary based upon the individual procedure and are specified in Tables 5-6 through 5-8.

5.7.8.5 If recoveries of any of the LCS standards are not within the limits specified in the table, the testing must be stopped. If the precision between the two recoveries is not within the limits specified in the table, the testing must be stopped. The analyst must follow the out-of-control procedures discussed in Section 5.8 prior to continuing any analyses.

5.7.9 Matrix spike (MS) and matrix spike duplicate (MSD). Individual component recovery of the matrix spike is calculated using the following equation:

$$\text{Matrix Spike Percent Recovery} = \frac{(A-B)}{T} \text{x}100$$

where:

A = concentration measured after spiking
B = background concentration
T = true value of the spiking concentration

5.7.9.1 MS and MSD sample recovery limits are used to determine matrix affects on the recovery target analytes. The acceptable recovery limits for the MS and MSD standards are indicated in Tables 5-6 through 5-8**.**

5.7.9.2 It is the discretion of the department manager to have a batch re-processed or re-analyzed after assessment of the matrix spike recovery values and other available batch QC data. The analyst must follow the out-of-control procedures discussed in Section 5.8 prior to continuing any analyses.

5.7.9.3 In the event that insufficient sample is provided for MS/ MSD analysis, the narrative of the final report must be amended to indicate lack of sample for analysis of MS and/or MSD.

5.7.10 An Initial Demonstration of Capability (IDOC) study is performed to establish the ability of an analyst and/or analytical system to generate acceptable precision and accuracy data. An IDOC study is performed on each certified method and matrix analyzed in the laboratory where applicable. Samples prepared for the IDOC studies are made from a second source independent of the standard source used for the calibration determination. A second source standard may be a standard purchased from the same manufacturer but a different lot or batch. To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations:

5.7.10.1 Because of the nature of several test methods, IDOCs cannot be performed. These tests represent methods where samples of known concentrations cannot be prepared in the laboratory. Specific requirements for these test methods are described in TABLE 5-***.

5.7.10.2 Calculate the average recovery (x) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each analyte using the four results. Demonstration of Capability must be updated and documented annually or more frequently if required by method with a Continuing Demonstration of Capability (CDOC).

5.7.10.3 The Method Performance Section of the individual SOP provides laboratory recovery and precision data for the method. Similar results from spiked water should be expected. Results are considered comparable if the calculated standard deviation of the recovery does not exceed the single laboratory RSD or 10% (20% for some organic analytes), whichever is greater and the mean recovery lies within the interval indicated by the test method, or X ± 15%, whichever is greater. Specific requirements for each NELAP certified test method as well as those required by AIHA are described in TABLE 5-***.

TABLE 5 - Demonstration of Capability Acceptance Criteria

Certified Method	DOC Requirement	Control Limits/ Acceptance Criteria*
NELAP METHODS		
E110.2 Color	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E120.1 Conductivity	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E150.1 pH	4 LCS or PT	LCS Control Limits or PT acceptance Criteria

E160.1 TDS	PT	PT acceptance Criteria
E160.2 TSS	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E160.3 TS	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E160.4 VS	PT	PT acceptance Criteria
E160.5 Settleable Solids	PT	PT acceptance Criteria
E1664 Oil and Grease_TPH	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E180.1 Turbidity	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E200.7 ICP AES Metals	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E200.8 ICP MS Metals	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E245.1 Mercury	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E300 Anions by IC	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E305.1 Acidity	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E310.1 Alkalinity	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E310.2 Alkalinity	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E325.2 Chloride	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E330.5 Residual Chlorine	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E335.1 Amenable Cyanide	4 LCS	LCS Control Limits
E335.2 Total Cyanide	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E335.4 Total Cyanide	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E350.1 Ammonia	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E351.2 TKN	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E353.2 Nitrate_Nitrite	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E354.1 Nitrite	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E360.1 Dissolved Oxygen	4 LCS	LCS Control Limits
E365.1 Ortho Phosphorus	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E365.1 Total Phosphorus	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E365.3 Ortho Phosphorus	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E370.1 Dissolved Silica	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E375.4 Sulfate	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E376.1 Sulfide	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E377.1 Sulfite	Comparability Study	RSD Limit ≤ RPD Limits
E405.1 BOD	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E410.4 COD	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E415.1 TOC	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E420.1 Total Phenolics	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E420.2 Total Phenolics	4 LCS or PT; LCR / MDL	LCS Control Limits or PT acceptance Criteria
E425.1 MBAS Surfactants	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E608 Pesticides PCBs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E608.2 Methoxychlor	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E610 PAHs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E615 Herbicides	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
E624 VOCs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria

E625 SVOCs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
FL-PRO	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
RSK-175 Dissolved Methane, Ethane, Ethene	4 LCS	LCS Control Limits
SM10200H Chlorophyll	4 LCS	LCS Control Limits
SM2340B Hardness	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SM2540G Total, Fixed and Volatile Solids	PT	PT acceptance Criteria
SM2710B SOUR	SOP Sign-Off Only	N/A
SM3500-Cr D Hexavalent Chromium	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SM3500-Fe D Ferrous Iron	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SM5210B CBOD	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SM9222B Total Coliforms	PT	PT acceptance Criteria
SM9222D Fecal Coliforms	PT	PT acceptance Criteria
SM9223B Total Coliforms and E. Coli	PT	PT acceptance Criteria
SW1010 Flash Point	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW1311 TCLP	SOP Signoff/AES Training	N/A
SW1312 TCLP	SOP Signoff/AES Training	N/A
SW6010 ICP AES Metals	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW6020 ICP MS Metals	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW7.3 Reactive Cyanide	4 LCS	LCS Control Limits
SW7.3 Reactive Sulfide	4 LCS	LCS Control Limits
SW7196 Hexavalent Chromium	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW7470 Mercury in Water	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW7471 Mercury in Soils	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8011 EDB DBCP	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8015 DAI	4 LCS	LCS Control Limits
SW8015 DRO	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8015 GRO	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8081 Pesticides	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8082 PCBs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8151 Herbicides	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8260 VOCs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8270 SVOCs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8310 PAHs	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW8315 Formaldehyde	4 LCS	LCS Control Limits
SW9010_9012 Cyanide	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9010_9014 Cyanide	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9030_9034 Sulfide	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9038 Sulfate	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9040 pH in Water	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9041 pH by Paper	SOP Sign-Off Only	N/A

SW9045 pH in Soil	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9050 Conductivity	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9056 Anions by IC	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9060 TOC	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9065 Total Phenolics	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9070 Oil and Grease_TPH in Water	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9071 Oil and Grease_TPH in Soils	4 LCS or PT	LCS Control Limits or PT acceptance Criteria
SW9081 Cation Exchange Capacity (Sodium)	SOP Sign-Off Only	N/A
SW9095 Free Liquids by Paint Filter	SOP Sign-Off Only	N/A
AIHA METHODS		
3M3520/SKC575(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1003(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1300(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1400(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1450(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1457(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1500(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1501(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N1550(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N2000(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N2500(GC/FID)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N5506(HPLC)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N7300(ICP except lead)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N6009(AA Hg)	IDOC: PT or 4 LCSs CDOC: Batch QC or PT	PT Acceptance Criteria or LCS Control Limits
N7082(Lead Paint)	IDOC: 4 sets of 5 Ref Materials CDOC: Batch QC or PT	75% within 90-110%Rec LCS Control Limits or PT Acceptance Criteria
SW3050B/7420(Lead in Soil)	IDOC: 4 sets of 5 Ref Materials CDOC: Batch QC or PT	75% within 90-110%Rec LCS Control Limits or PT Acceptance Criteria
SW3050B/7000B(Lead in Soil)	IDOC: 4 sets of 5 Ref Materials CDOC: Batch QC or PT	75% within 90-110%Rec LCS Control Limits or PT Acceptance Criteria
N9102(Lead in Dust Wipe)	IDOC: 4 sets of 5 Ref Materials	75% within 90-110%Rec LCS Control Limits or PT Acceptance Criteria

	CDOC: Batch QC or PT	
N7300(Lead in Air)	IDOC: 4 sets of 5 Ref Materials CDOC: Batch QC or PT	75% within 90-110%Rec LCS Control Limits or PT Acceptance Criteria
N7400(Asbestos PCM)	PT Samples	PT Acceptance Criteria
N0500/0600(Particulates)	SOP Sign Offs	NA
Fungal Air Culturable(Micro)	PT Samples	PT Acceptance Criteria
Fungal Bulk Culturable(Micro)	PT Samples	PT Acceptance Criteria
Fungal Surface Culturable(Micro)	PT Samples	PT Acceptance Criteria
Fungal Air Direct Exam(Micro)	PT Samples	PT Acceptance Criteria
Fungal Bulk Direct Exam(Micro)	PT Samples	PT Acceptance Criteria
Fungal Surface Direct Exam(Micro)	PT Samples	PT Acceptance Criteria

*LCS Control Limits and RPD Limits as per Tables 5-7 and 5-8 and LIMS Test Code Limits

5.7.10.4 The large number of analytes in multi-element analyses presents a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined. Should this occur, re-analyze only the failed analytes, following the procedures discussed in this section.

5.7.10.5 When one or more of the analytes tested fails at least one of the acceptance criteria, the analyst must proceed according to the out-of-control procedures discussed in Section 5.8.

5.7.10.6 Because of the nature of several test methods, IDOCs cannot be performed. These tests represent methods where samples of known concentrations cannot be prepared in the laboratory. Tests that are included in this category are EPA 110.2, 160.3, 160.4, 160.5, 150.1, 9040, 9045, 1010, SM 2340B, SM2340G, SM9223, and SM9222. To complete IDOCs for these tests, the analyst(s) must satisfactorily pass available PE samples for all appropriate matrices.

5.7.10.7 Analyst Demonstration of Capability and training includes the following:

Quality Assurance Manual Training (annually)
Legal & Ethical Training (annually)
SOP Training (initially and as updated)
ICNs associated with the SOPs (initially and as updated)
Demonstration of Capability (program specific)
Procedure and Checklist Training (initially and as updated)

Individuals are authorized to perform analysis when these documents have been completed and signed by the individual(s) and referenced managers.

5.7.10.8 AIHA Training Requirements

AIHA Technician/Analyst Training Requirements. All technicians and analysts must complete training and demonstrate proficiency prior to analysis of any ELLAP or IHLAP program samples. The training and proficiency demonstrations must

meet the requirements specified in the AIHA LQAP Policy Document, Modules 2A, 2B and 2C and are described in Section 1.2 and 1.3 below.

5.7.10.8.1 ELLAP Specific Technician/Analyst Training Requirements:

5.7.10.8.1.1 Initial demonstration of capability.

Each technician/analyst must complete at least 20 days of work/training in the prep and/or metals analysis lab using technologies/instrumentation similar to that to be used for ELLAP samples under the direct supervision of an ELLAP trained technician/analyst prior to unsupervised prep and/or analysis of ELLAP regulated client samples.

Each analyst/technician must read, understand and agree to follow the laboratory SOP as documented using the SOP Acknowledgement sign-off form.

Each technician/analyst must prep and/or analyze as appropriate at least 2 blind reference material test samples (concentration unknown to the technician/analyst). These samples may be AIHA provided PT samples or laboratory prepared Certified Reference Material of the appropriate matrix, i.e. soil, paint, wipe(spiked with baghouse dust) or air filter. Results must fall within the PT acceptance range or laboratory LCS range as appropriate.

Each technician/analyst must complete a minimum of 4 independent test runs of sample preparation/analysis prior to prepping/analyzing actual samples. This test is performed through the digestion/analysis of four separate groups of 5 replicate, matrix specific Certified Reference Material samples, with each group separated by at least one day. To be deemed acceptable per ELLAP requirements, 75% of the replicates in each group must recover within 90-110% of the true value. Any individual group that fails to meet the ELLAP criteria must be repeated in its entirety (all 5 replicates repeated).

Once all requirements in 5.7.10.8.1.1 have been met, the technician/analyst will be approved to begin unsupervised prep/analysis of client samples. Documentation of approval to begin work is defined as the date signed by the Technical Director (or designee) on the Demonstration of Capability Certification form.

5.7.10.8.1.2 Continuing Demonstration of Capability (CDOC). Each technician/analyst must demonstrate continued capability at least every 6 months through the analysis of AIHA provided PT samples or in house laboratory QC samples, i.e. LCS samples. Results must fall within the AIHA PT acceptance criteria or Policy Module 2C, Table 2C-1 LCS control limits per samples used. CDOCs are documented via AIHA PT reports or LIMS LCS data per samples used.

5.7.10.8.1.3 All IDOC and CDOC documentation for ELLAP related procedures is maintained and available for review for at least 10 years.

5.7.10.8.2 IHLAP Chemistry Specific Technician/Analyst Training Requirements:

5.7.10.8.2.1 Initial demonstration of capability.
Each technician/analyst must complete at least 20 days of work/training in the prep and/or metals analysis lab using technologies/instrumentation similar to that to be used for IH samples under the direct supervision of an IH trained technician/analyst prior to unsupervised prep and/or analysis of IH regulated client samples.

Each analyst/technician must read, understand and agree to follow the laboratory SOP as documented using the SOP Acknowledgement sign-off form.
Each technician/analyst must prep and/or analyze as appropriate at least 2 blind reference material test samples (concentration unknown to the technician/analyst). These samples may be an AIHA provided PT samples or laboratory prepared Certified Reference Material added to the method specific media used for client samples. Results must fall within the PT acceptance range or laboratory LCS range as appropriate.

Once all requirements in 5.7.10.8.2.1 have been met, the technician/analyst will be approved to begin unsupervised prep/analysis of client samples. Documentation of formal approval to begin work is defined as the date signed by the Technical Director on the Demonstration of Capability Certification form.

5.7.10.8.2.2 Continuing Demonstration of Capability (CDOC). Each technician/analyst must demonstrate continued proficiency at least every 6 months through the analysis of AIHA provided PT samples or in house laboratory QC samples, i.e. LCS samples. Results must fall within the AIHA PT acceptance criteria or laboratory established LCS control limits as appropriate. CDOCs are documented via AIHA PT reports or LIMS LCS data as appropriate.

5.7.10.8.2.3 All IDOC and CDOC documentation for IHLAP related procedures is maintained and available for review for at least 10 years.

5.7.10.8.3 IHLAP Asbestos by PCM Specific Technician/Analyst Training Requirements:

5.7.10.8.3.1 All PCM technicians/analysts must complete a NIOSH 582 equivalent training course and successfully pass the course examination during their training period and prior to beginning unsupervised work on client samples.

5.7.10.8.3.2 Initial demonstration of capability.

Each technician/analyst must complete at least 20 days of work/training in the PCM analysis lab using technologies/instrumentation similar to that to be used for IH/PCM samples under the direct supervision of an IH/PCM trained technician/analyst prior to unsupervised prep and/or analysis of IH/PCM regulated client samples.

Each analyst/technician must read, understand and agree to follow the laboratory SOP as documented using the SOP Acknowledgement sign-off form.

Each technician/analyst must prep and/or analyze as appropriate at least 2 blind reference material test samples (concentration unknown to the technician/analyst). These samples may be an AIHA provided PT samples or laboratory prepared Reference Slides. Results must fall within the PT acceptance range or laboratory reference slide counting acceptance ranges as appropriate.

Once all requirements in 5.7.10.8.3.2 have been met, the technician/analyst will be approved to begin unsupervised prep/analysis of client samples. Documentation of formal approval to begin work is defined as the date signed by the Technical Director on the Demonstration of Capability Certification form.

5.7.10.8.3.3 Continuing Demonstration of Capability (CDOC).
Each technician/analyst must demonstrate continued proficiency at least every 6 months through the analysis of AIHA provided PT samples or laboratory prepared Reference Slides. Results must fall within the AIHA PT acceptance criteria or laboratory reference slide counting acceptance ranges as appropriate. CDOCs are documented via AIHA PT reports or in the QC data log books maintained in the PCM laboratory as appropriate.

5.7.10.8.3.4 All IDOC and CDOC documentation for IHLAP related procedures is maintained and available for review for at least 5 years.

5.7.10.8.4 EMLAP Specific Technician Training Requirements:

5.7.10.8.4.1 EMLAP laboratory technicians must meet minimum educational requirements of a high school diploma or GED.

5.7.10.8.4.2 Initial demonstration of capability.
Each technician must complete at least 6 months documented training for Air-Direct Exam (spore trap) and work/training in the EMLAP microbiology laboratory under the direct supervision of an EMLAP trained technician/analyst prior to performing unsupervised technician level work on EMLAP regulated client samples.

Each technician must read, understand and agree to follow the laboratory SOP as documented using the SOP Acknowledgement sign-off form.

Technician level personnel are limited to preparatory operations and assistance in all steps leading to the identification of microorganisms and may not perform analyses or be responsible for the final decisions related to the identity of microorganisms, except as described below:

"Technicians may function as analysts for Air-Direct Examination (spore traps) analysis after completion of 12 months documented on the job training and demonstrated proficiency. During the 12 month analyst training period, the trainee may perform work under the direct supervision of another qualified analyst. All work must be reviewed by another qualified analyst prior to release of data."

Demonstrated proficiency for technicians functioning as analysts shall be documented through successful analysis of EMLAP PT samples or laboratory reference slides and document the ability to identify genus/groups of fungi reported. The technician must also complete and pass the laboratory Fungal Identification Examination/Quiz as administered by the microbiology department supervisor.

Once all requirements in 5.7.10.8.5.2 have been met, the technician will be approved to begin unsupervised prep/analysis of client samples. Documentation of formal approval to begin work is defined as the date signed by the Technical Director on the Demonstration of Capability Certification form.

5.7.10.8.4.3 Continuing Demonstration of Capability (CDOC).
Each technician must demonstrate continued proficiency at least every 6 months through the analysis of AIHA provided PT samples or laboratory prepared Reference Slides. Results must fall within the AIHA PT acceptance criteria or laboratory reference slide counting acceptance ranges as appropriate. CDOCs are documented via AIHA PT reports or in the QC data log books maintained in the microbiology laboratory as appropriate.

5.7.10.8.4.4 All IDOC and CDOC documentation for EMLAP related procedures is maintained and available for review for at least 5 years

5.7.10.8.5 EMLAP Specific Analyst Training Requirements:

5.7.10.8.5.1 EMLAP laboratory analysts must meet minimum educational requirements of a baccalaureate degree in microbiology, biology or related life science.

5.7.10.8.5.2 Initial demonstration of capability.
Each analyst must complete at least 3 months of documented training fro Air-Direct Exam (spore trap) and at least 6 months of work/training in the EMLAP microbiology laboratory prior to performing unsupervised work on EMLAP regulated client samples.
Each analyst must read, understand and agree to follow the laboratory SOP as documented using the SOP Acknowledgement sign-off form

Each analyst must prep and/or analyze as appropriate at least 2 blind reference material test samples. These samples may be an AIHA provided PT samples or laboratory prepared Reference Slides. Results must fall within the PT acceptance range or laboratory reference slide counting acceptance ranges as appropriate and document proper identification of genus/species and genus/groups of fungi reported.

Once all requirements in 5.7.10.8.5.2 have been met, the technician/analyst will be approved to begin unsupervised prep/analysis of client samples. Documentation of formal approval to begin work is defined as the date signed by the Technical Director on the Demonstration of Capability Certification form.

5.7.10.8.5.3 Continuing Demonstration of Capability (CDOC). Each technician/analyst must demonstrate continued proficiency at least every 6 months through the analysis of AIHA provided PT samples or laboratory prepared Reference Slides. Results must fall within the AIHA PT acceptance criteria or laboratory reference slide counting acceptance ranges as appropriate. CDOCs are documented via AIHA PT reports or in the QC data log books maintained in the microbiology laboratory as appropriate.

5.7.10.8.5.4 All IDOC and CDOC documentation for EMLAP related procedures is maintained and available for review for at least 5 years.

5.7.11 The Method Detection Limit (MDL). MDL studies are to be updated annually, or whenever instrument conditions change in a manner that will affect the established detection limits. Method detection limits are determined in the following manner:

5.7.11.1 Prepare a minimum of 7 replicates of a standard containing all the analytes in the method by spiking test-related volumes and weights of reagent water and soil, respectively. Add internal standards and/or surrogates if required for the test method.

5.7.11.2 Review MDL studies from the past year to ascertain the appropriate spike concentration. To be valid, the MDL must be less than or equal to the spike concentration. (For AIHA ELLAP, the reporting limit has to be at least twice the MDL or the MDL must be one half the PQL or less. Also, AIHA requires annually the analysis of a media spike at or near the laboratory reporting limit. This can be

satisfied by an annual MDL study.) In addition, the calculated MDL will be evaluated to see if it is greater than 10% of the spike concentration. This determination will be used as a guide to establish that an appropriate concentration was used based on the laboratory practical quantitation limit (PQL) unless required by regulatory rule (E.g. EPA Title 40 CFR Part 136, Appendix B, as listed in Appendix V.) The QA Manager, Technical Director or Laboratory Manager may approve an MDL study with a calculated MDL that is greater than 10% of the spike concentration if the PQL is substantially above the MDL.

5.7.11.3 Analyze the replicates by the test method. Include any necessary preparative procedures such as digestions and extractions. Using all the results not disqualified through the Grubbs outlier test, calculate the MDL of all analytes in the test method using the appropriate student t value for the number of replicates used.

5.7.11.4 The quantitation limits are tied to the detection limits in that the PQLs are never less that MDLs and a low level standard is analyzed at the PQL where applicable.

5.7.11.5 Table 5-1 below summarizes the calculations used to determine the MDL. The calculations represent commands in Excel format.

Table 5-1 MDL Calculation Form/Spreadsheet

Column Heading	Calculation
Analyte	
Rep 1 – Rep 7 (Individually)	
Mean	AVERAGE (Replicate1...Replicate7)
SDn-1 (Standard Deviation)	STDEV (Replicate1...Replicate7)
Spike Amount	Final Concentration Including Prep
MDL	t Value * STDEV
% Recovery	(Mean/Spike Amount) * 100
Low Spike Check	IF(Spike Conc.>10*MDL,”OK”,”NOT OK”

5.7.12 Method Blank (MB). For each method, the analyst must daily analyze reagent water blank to demonstrate that interferences from the analytical system are under control. The method blank is treated in the same manner as any sample, including any sample preparations such as digestions and extractions.

5.7.12.1 In the method blank, the concentration of any analyte of interest should not exceed the laboratory established practical quantitation limit (PQL). If contamination is detected in the blank, one of the following conditions must be met, or re-analysis of all associated samples is required (Section 5.8, Out of Control Procedures).

5.7.12.1.1 With documentation of client approval, the PQL may be increased above the level of contamination in the method blank and associated samples. Report data with a “B” qualifier.

5.7.12.1.2 For sample results greater than or equal to 10 times the concentration of the method blank, the data may be reported with a flag indicating that low level contamination was detected in the method blank. Report data with a “B” qualifier.

5.7.13 Surrogates and Surrogate Recovery measured during the analysis of organic compounds. In order to monitor sample extraction efficiency, all client samples, blanks, and QC samples are fortified with surrogate spiking compounds before extraction and injection into the instrument.

5.7.13.1 Acceptance Criteria: Acceptable surrogate recoveries are contained in LIMS.

5.7.13.2 At a minimum, the laboratory annually updates surrogate recovery limits on a matrix-by-matrix basis for each test method.

5.7.13.3 If the surrogate recovery fails the above stated acceptance criteria, the analyst must proceed according to the out-of-control procedures discussed in Section 5.8.

5.7.13.4 Calibration curves. At a minimum, a 5 point calibration curve must be developed for each surrogate that is used in a particular test method.

5.7.14 Additional Quality Control Parameters Required for GC/MS Analysis Methods.

5.7.14.1 BFB or DFTPP tune. Inject or introduce 50ng of the 4-Bromofluorobenzene (BFB) standard into the GC/MS system prior to VOC analysis, or 50ng of decafluorotriphenylphosphine (DFTPP) prior to SVOC analysis. The resultant mass spectra for the BFB must meet the criteria given in Table 5-2 before VOC sample analysis begins, or the criteria given in Table 5-3 before SVOC sample analysis begins. This criterion must be demonstrated each 24-hour shift for 624 and 625 or each 12 hour shift for 8260 and 8270 during which samples are analyzed. If these criteria cannot be achieved, analyses may not be performed.

5.7.14.1.1 Perform the tune by acquiring three scans (the peak apex scan and the scans immediately preceding and following the apex) and subsequently averaging them. Background subtraction is required and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of either the DFTPP or the BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP or BFB peak.

5.7.14.1.2 Use the DFTPP or BFB mass intensity criteria in Tables 5-2 or 5-3 as tuning acceptance criteria

5.7.14.1.3 If the DFTPP or BFB tune cannot be achieved, analysis may not start. If subsequent tunes performed during an analytical run fail the criteria, proceed according to the out-of-control procedures discussed in Section 5.8.

Table 5-2
BFB Tuning Criteria:

m/z	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 1% of mass 95
174	greater than 50% of mass 95

175	5 to 9% of mass 174
176	between 95 and 101% of mass 174
177	5 to 9% of mass 176

Table 5-3
DFTPP Tuning Criteria:

m/z	Ion Abundance Criteria
51	30 to 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 to 60% of mass 198
197	<1% of mass 198
198	base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17 to 23% of mass 442

5.7.14.2 Internal standard retention time – The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during GC or GC/MS data acquisition.

5.7.14.2.1 If the retention time for any internal standard changes by more than 30 seconds from the retention time of the mid-point standard in the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. Proceed according to the out-of-control procedures discussed in Section 5.8.

5.7.14.2.2 Internal standard response - If the area for any of the internal standards in the ICV or CCV changes by more than a factor of two (-50% to +100%) from that of the mid-point standard level in the most recent initial calibration sequence, the mass spectrometer or GC system must be inspected for malfunctions and corrections must be made unless the exceedance is caused by matrix interference. Proceed according to the out-of-control procedures discussed in Section 5.8.

5.7.14.3 Determination of Retention Time Window. Before establishing windows, be certain that the GC, GC/MS, or HPLC system is within optimum operating conditions. To determine the retention time window, make three injections of the sought for standard(s) or analyte(s) throughout the course of a 72 hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

5.7.14.3.1 Calculate the standard deviation of the three absolute retention times for the standard(s) in question.

5.7.14.3.2 The retention time window for individual peaks is defined as plus-or-minus (+/-) three (3) times the standard deviation of the absolute retention time.

5.7.14.3.3 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use +/- 0.05 minutes as a retention time window.

5.7.14.3.4 The laboratory must calculate retention time windows for each standard on every existing GC column and on each new GC column when it is installed. The data is be retained by the laboratory for a period of 5 years.

5.7.14.4 Calibration check compounds (CCCs). These compounds are part of the CCV.

5.7.14.4.1 The purpose of the CCCs is to evaluate the calibration based on the integrity of the GC/MS instrument system. High variability of these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes in the test methods.

5.7.14.4.2 Acceptance criteria are presented in Table 5-6.

5.7.14.4.3 Table 5-4 indicates the CCC compounds used in GC/MS analysis.

Table 5-4
GC/MS CCC Compound List

8260 Volatile Organic Compounds

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

8270 Semi-Volatile Organic Compounds

Base Neutral Fraction	*Acid Fraction*
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

5.7.14.5 System performance check compounds (SPCCs). Table 5-5 indicates the SPCC compounds used in GC/MS analysis.

5.7.14.5.1 A system performance check is performed after the initial calibration curve is run and every 12 hours thereafter.

5.7.14.5.2 Five compounds are checked for a minimum average response factor. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

5.7.14.5.3 Acceptance criteria are described in Table 5-6

Table 5-5
GC/MS SPCC Compounds and Required Response Factors

Volatile Organic Compounds

Compound	*Response Factor*
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

Semi-Volatile Organic Compounds (Minimum RF = 0.05)

Base/Neutral Fraction	*Acid Fraction*
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

5.7.14.6 For TCLP analysis, a matrix spike should be prepared and analyzed for each waste type (e.g., oil, solid) associated with a batch of 20 or fewer samples of similar matrix.

5.7.15 Additional Quality Control Parameters Required for Metals Analysis by 7000 Series Methods.

5.7.15.1 Dilution test. For each analytical batch, select one typical sample for serial dilution to determine whether interferences are present. The concentration of the analyte should be at least 25 times the estimated detection limit.

5.7.15.1.1 Determine the apparent concentration in the undiluted sample. Dilute the sample by a minimum of five fold (1 + 4) and reanalyze.

5.7.15.1.2 If all of the samples in the batch are below 10 times the detection limit(s), perform the spike recovery analysis.

5.7.15.1.3 Agreement within 10% between the concentration of the undiluted sample and five times the concentration of the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the method of standard additions.

5.7.15.2 Spike Recovery Test. If results from the dilution test do not agree (or if none of the samples in the batch are at a concentration level that is 10 times the MDL) the spike recovery test must be performed.

5.7.15.2.1 Withdraw another aliquot of the test sample and add a known amount of analyte to bring the concentration of the analyte to 2 to 5 times the original concentration.

5.7.15.2.2 If all of the samples in the batch have analyte concentrations below the detection limit, spike the selected sample at 20 times the detection limit.

5.7.15.2.3 Analyze the spiked sample and calculate the spike recovery. If the recovery is less than 85% or greater than 115%, the method of standard additions

shall be used for all samples in the batch or data qualified and narrated with client report.

5.7.15.3 Method of Standard Additions (MSA).

5.7.15.3.1 Add, to equal volumes of the sample, a series of standard solutions containing different known quantities of the analyte. Typically, add known concentrations that are 50%, 100%, and 150% of the original measured concentration.

5.7.15.3.2 Dilute all solutions to the same volume with DI water.

5.7.15.3.3 Plot the absorbance of all of the solutions, including the original, on the vertical axis of a graph, versus the added concentrations of the analyte, on the horizontal axis of the graph.

5.7.15.3.4 Extrapolate the line through zero absorbance. The point of interception of to the left of the vertical axis gives the determined concentration of the analyte in the sample. Note that the scaling on both sides of the horizontal axis must be the same.

5.7.15.3.5 For this technique to be valid, the following limitations must be taken into consideration:

5.7.15.3.5.1 The apparent concentrations from the calibration curve must be linear over the concentration range of interest. For the best results, the slope of the MSA plot should be similar to the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised since the variant spike recovery may be due to instrument problems rather than a matrix effect.

5.7.15.3.5.2 The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner to the analyte.

5.7.15.3.5.3 The determination must be free of spectral interference and corrected for nonspecific background interference.

5.7.16 Additional Quality Control Parameters Required for Metals Analysis by ICP Methods.

5.7.16.1 The upper limit of the linear dynamic range must be established for each wavelength utilized. This is accomplished by measuring the signal response of a standard that is 10% higher than the upper range of the calibration curve.

5.7.16.2 The laboratory must establish and verify every six months an inter-element spectral interference correction routine to be used during sample analysis. See the individual ICP method SOPs for instructions on performing this test.

5.7.16.3 Duplicate or matrix spike duplicate samples. For all target metals, one sample per analytical batch is digested and analyzed in duplicate or as matrix spike duplicate. The results are compared and should meet the precision control limits established.

The results are only considered valid if the concentration of the sample is at least two times the MDL.

5.7.16.4 An instrument blank should be run after any sample giving a response that exceeds the calibration range of the instrument. This is done to show that there is no carry-over to the next analysis. The instrument blank shall consist of a high purity solvent (e.g., hexane for pesticide analysis by GC/ECD, methylene chloride for semi-volatiles analysis by GC/MS).

5.7.17 Additional Quality Control Parameters Required for Microbiological Test Methods.

5.7.17.1 Laboratory water quality must be checked and documented at the frequency indicated below.

Requirement	Criteria	Frequency
pH	5.5-7.5	Each day test is performed
Residual Chorine	<1.0 mg/L	Each day test is performed
Conductivity	<2.0 µmho/cm @25°C	Each day test is performed
Heterotrophic Plate Count	<500 colony forming units/ml	Monthly
Bacteriological Ratio	0.8-3.0	Annually
Cd, Cr, Cu, Ni, Pb, Zn	<0.05 mg/L each, total <1.0 mg/L	Annually
NH_3, Organic Nitrogen	<0.1 mg/L	Monthly
TOC	<1.0 mg/L	Monthly
Student's t value	<2.78 (Annual use test)	Annually

5.7.17.2 The laboratory maintains records of monthly checks on sterile water and membrane filters as evidence of trends in contamination levels for microbiology through Heterotrophic Plate Count measurements. If the contamination level exceeds 1000 CFU/ml, all equipment should be checked for sterility and re-sterilized as necessary. In addition, if additional testing indicates that the problem is still present, then the room used for bacteriological testing should be cleaned with a disinfectant soap and plate counts measured again. Repeat the process as necessary.

5.7.18 Internal Proficiency Testing

5.7.18.1 The laboratory has an internal proficiency testing program that is used to show competency as required by certain regulatory agencies for fields of testing not covered by an external proficiency program.

5.7.18.2 For fields of testing not covered by AIHA proficiency samples, the laboratory shall demonstrate competency for a minimum of one method per field of testing by the implementation of one of the following three alternatives.

5.7.18.2.1 The laboratory will annually participate in a third party proficiency testing program at least twice per year. Upon completion of the study, results will be evaluated and if results are unacceptable, a corrective action will be initiated to determine the cause of the failure.

5.7.18.2.2 The laboratory will annually participate in a round robin program.

5.7.18.2.2.1 The Round Robin Program will include participation of at least three laboratories with two rounds per year. Each round will be completed within a six month timeframe. On a rotating basis, each lab will act as the designated provider of samples.

5.7.18.2.2.2 Each round will include a minimum of four samples of varying concentrations that will be independently analyzed and reported by all the individual analysts.

5.7.18.2.2.3 Samples will resemble real world samples as much as possible.

5.7.18.2.2.4 Upon completion of the study, results will be submitted to the laboratory that provided the samples and all the results will be distributed for evaluation.

5.7.18.2.3 The laboratory will annually participate in an internal proficiency program at least twice per year for at least one method in the field of testing.

5.7.18.2.3.1 The Quality Assurance Manager will initiate the internal proficiency testing during the peak periods of proficiency testing in the lab, generally in the spring and autumn.

5.7.18.2.3.2 A minimum of four samples will be logged into the laboratory's LIMS system by a member of the Quality Assurance Department.

5.7.18.2.3.3 The department supervisor will be notified to prepare the blind spikes at varying concentrations. Sample media will be spiked and the documented target concentrations will be forwarded to the Quality Assurance Manager, who will prepare a reporting form for each method.

5.7.18.2.3.4 The blind proficiency samples will be analyzed as routine samples. Results will be entered into the laboratory's LIMS system and processed as routine samples. All method and batch QC criteria must be acceptable before sample results are reported.

5.7.18.2.3.5 The results will be checked against the established acceptance criteria. Department Supervisor and Analyst will be notified as to whether the proficiency samples passed or failed.

5.7.18.2.3.6 The upper and lower acceptance limits will be established using ± 3 standard deviations ($3\sigma_{n-1}$) from the mean LCS % recovery based on historical data.

5.7.18.2.3.7 If the results are unacceptable, a corrective action will be initiated to determine the cause of the failure. Upon completion of the corrective action, the study will be repeated to ensure the analyst can pass this study.

5.7.18.2.3.8 The current programs with fields of testing for which Internal Proficiency testing will be performed include:

For the AIHA Program

Field of Testing	Method
HPLC	N5506
Gravimetric	N0500
AA	N6009

5.8 Procedures for Assessing and Treating Out-of-Control Situations.

5.8.1 Quality control analyte samples consist of the following: MB, LCS, LCSD, MS, MSD, ICV, CCV, BFB and DFTPP tunes, internal standards, surrogates, post digestion spikes, and dilution tests.

5.8.2 If any of the quality control analyte recovery values are outside either the laboratory or method-established control limit(s), they are considered to be out-of-control.

5.8.3 The resolution of an out-of-control situation, with identification and correction of the root cause, must be documented prior to initiating subsequent analyses. Documented corrective action (which may or may not require re-analysis) must also be performed if any of the recovery values in the LCS exhibit any "out-of- control" patterns.

5.8.4 Out-of-control conditions that are not addressed in Table 5-6 include the following special situations:

5.8.4.1 When the acceptance criteria for the continuing calibration verification have a high bias, and there are associated samples that are non-detects, then the non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be re-analyzed after the source of the problem has been corrected.

5.8.4.2 When the acceptance criteria for the continuing calibration verification have a low bias, those sample results may be reported if they exceed a maximum regulatory limit or decision level. Otherwise, the samples affected by the unacceptable verification shall be re-analyzed after the source of the problem has been corrected.

5.8.4.3 The root cause of such failures must be investigated and documented in a Non-Conformance Report (NCR). Any corrective actions identified as a result of the investigation must be implemented and documented in a Corrective Action Report (CAR) prior to reprocessing the affected sample batch.

5.8.4.4 The quality control requirements associated with each test method are listed LIMS test codes. They are also posted as charts and tables on the portal server. Unless otherwise indicated, if tables and charts have been produced, the precision and accuracy limits were determined from laboratory data.

5.9 Inter-laboratory QA and QC

5.9.1 Each section of the laboratory may be given blind and double blind samples to analyze for requested parameters. Blind samples may be assigned in containers to be diluted, digested, and/or extracted and analyzed by the appropriate laboratory section. Double-blind samples

may arrive on a pre-scheduled basis from a "client" as real samples to be analyzed by designated analytical sections for specific analytes.

5.9.2 Blind QC samples may be used as a test of proficiency for analysts needing certification and/or qualification for performing an analysis. The Section Supervisor should obtain the QC sample from, either, the Quality Assurance Department or from a source independent of the source of standards for the analysis.

5.9.3 Double blind samples represent quality control samples whose analyte concentrations are known to, either, an outside source, such as a client, or an inside source, such as the Quality Control Manager, Project Managers, or the Technical Director.

5.9.3.1 Double blind samples will arrive in the laboratory as real samples and their identity will not be known to anyone as quality control samples except for Quality Assurance and Project Management.

5.9.3.2 The results of these double-blind samples will be sent to the "client" to be compared to the true value of the samples. The laboratory's performance on these samples may be compared to other laboratories in the program (if applicable). These results will be mailed to the Quality Assurance Department.

5.9.3.3 When the double blind samples are created within the laboratory, a report will be generated by the Quality Assurance Manager or the Technical Director that indicates the true value of the analyte. These values will be compared to the reported value by the laboratory. The analysis of double blind samples is used as an aid to improve quality control within the laboratory.

5.9.3.4 Double blind samples are submitted to the various laboratory departments on a semi-annual for TNI Accreditation per the annual schedule for AIHA Certification.

5.10 Sample Dilution

5.10.1 All instruments are periodically calibrated with calibration curves. The calibrations typically are developed by comparison of area or intensity against sample concentration. Per the requirements of the various accreditation agencies, the calibrations are verified initially and periodically, usually every day or every 12 hours.

5.10.2 Various test methods additionally require that the linear range of the instrument is determined on a specified frequency.

5.10.3 In the event that a measured sample concentration exceeds the concentration of the highest calibration standard or the linear range of the instrument (where determined), the sample must be diluted per the following procedure.

5.10.3.1 The analyst should attempt to dilute the sample so that the measured concentration of the diluted sample is approximately 60% that of the highest standard in the calibration curve.

5.10.3.2 The sample must be diluted with the same matrix as the undiluted sample as indicated below.

5.10.3.2.1 Aqueous samples are diluted with reagent grade distilled water.

5.10.3.2.2 Extracts in solvents are diluted with the same solvent of the same purity.

5.10.3.2.3 ICP digestates are diluted with nitric acid or hydrochloric acid-water mixtures that emulate the original matrix.

5.10.3.3 The sample dilution is reported in the LIMS and on the data sheet. The results are reported to the client and the reporting limits are automatically adjusted by the LIMS system to account for the sample dilution.

Table 5-6
Summary of Calibration and QC Procedures for Various Tests

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action (3,4)
SW-8081B	Five-point initial calibration	Initial calibration prior to sample	RF = 20%	Correct problem then repeat initial
Pesticides	for all analytes	analysis		calibration
SW-8082A				
PCB			Linear - least squares regression	
SW-8151A			r>0.995	
Herbicides	Second source calibration	Once per five point initial	All analytes within 15% of	Correct problem then repeat initial
SW-8015C	verification standard (ICV)	calibration - from second	expected value	calibration
Organics		source.	GRO/DRO = 15% PRO = 20%	
GRO	Retention time window	System set-up	3 times standard deviation for	Correct problem then re-analyze
DRO	calculated for each analyte		each analyte retention time from	all samples analyzed since
FL-PRO			72 hour study	retention time check
SW-8315	Continuing calibration	Before sample analysis, after	All analytes within 15% of	Correct problem then repeat initial
Carbonyls	verification	every 10 samples, and at the end	expected value	continuing calibration verification
TO-10A		the analysis sequence - varying	GRO/DRO = 20% PRO = 25%	and re-analyze all samples since
		concentrations	8081B/8082A = 20%	last successful CCV
		GRO/DRO Every 12 hours before		
		sample analysis, after every 10		
		samples , and at the end of the		
		analytical sequence		
		GRO/DRO = RT window		
		required analyzed at same		
		frequency as CCV		
	Breakdown check (Endrin	Daily prior to analysis of samples	Degradation <15%	Inlet column maintenance; repeat
	and DDT)(1)			breakdown check
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See @ LIMS Test codes or	Re-prep and analyze the LCS/LCSD
			QC Charts	and all samples in the affected
				analytical batch
	Surrogate Spike	Every sample, spiked sample,	See @ LIMS Test codes or	Check system, re-inject, re-extract
		standard, and method blank	QC Charts	
	MS/MSD	One per prep batch	See @ LIMS Test codes or	None - Narrate the results in LIMS
			QC Charts	
	IDOC	Every time a new analyst	See @ LIMS Test codes or	Analyst cannot perform the test
		performs the test method for the		method until the IDOC passes
		first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed		of different concentration
	Second column	100% for all positive results	Same results as primary	Only report the results that match.
	confirmation (2)	(not for 8015B)	column analysis	Use the highest results
SW-8260B	Tune GC/MS	Prior to initial calibration and	See individual method for	Analyst cannot perform the test
SW-8270D	BFB for 8260B	continuing calibration verification	tune criteria.	method until the tune passes
	DFTPP for 8270D	every 12 hours		method criteria

SW-8260B	Five-point initial calibration	Initial calibration prior to sample	SPCCs average RF>0.30 for	Correct problem then repeat initial
VOC	for all analytes	analysis.	for 1,1,2,2-Tetrachloroethane and	calibration
			Chlorobenzene. RF>0.10 for	
			Chloromethane, Bromoform, and	
			1,1-Dichloroethane.	
			% RSD for RFs for CCCs <30%	
			and mean RSD for all analytes	
			average <15%.	
SW-8270D			Linear RSD≤20%	
SVOC			Non-linear CC≤0.99	
	Second source calibration	Once per five point initial	All analytes within 30% of	Correct problem then repeat initial
	verification standard (ICV)	calibration -second source.	expected value	calibration
	Retention time window	Each Sample	Relative retention time (RRT) of	Correct problem then re-analyze
	calculated for each analyte		the analyte within 0.06 RRT units	all samples analyzed since
			of the RRT	retention time check
SW-8260B	Continuing calibration	Daily prior to analysis of samples	SPCCs average RF≥0.30 and	Correct problem then repeat initial
	verification	and every 12 hours of analysis	%RSD for RFs for CCCs <20%	continuing calibration verification
		time.	and mean RSD for all	and re-analyze all samples since
			Analytes average <20%.	last successful CCV
SW-8270D			Each of the most common target	If not met the system should be
			analytes in the calibration	evaluated, and corrective action
			verification standard should meet	should be taken before analysis
			the minimum response factors	begins.
			In Table 4 of the method.	If criterion is not met for more than
				20% of the compounds included
				In the initial calibration, then corrective
				action must be taken prior to analysis
				of samples.
			Target compounds ≤20%	
	Internal Standards	Every sample/standard	Retention time +/-30 seconds	Inspect GC/MS for malfunctions;
			from retention time of the mid-	mandatory re-analysis of samples
			point	analyzed while system was
			in the CCV/ICAL	malfunctioning.
			(sample/standard)	
			EICP area within -50% to +100%	
			of ICAL mid-point standard	
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See @ LIMS Test codes or	Re-prep and analyze the LCS/LCSD
			QC Charts	and all samples in the affected
				analytical batch
	Surrogate Spike	Every sample, spiked sample,	See @ LIMS Test codes or	Check system, re-inject, re-extract
		standard, and method blank	QC Charts	
	MS/MSD	One per prep batch	See @ LIMS Test codes or	None - Narrate the results in LIMS
			QC Charts	
	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
		for the first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed		of different concentration
SW-8310	Five-point initial calibration	Initial calibration prior to sample	Linear - RSD of average RF	Correct problem then repeat initial
PAH	for all analytes	analysis	of all analytes <20% or	calibration
			mean RSD for all analytes <20%	
			with no individual analyte RSD>	
			30%	
	Second source calibration	Once per five point initial	All analytes within 20% of	Correct problem then repeat initial
	verification standard (ICV)	calibration - second source.	expected value	calibration
	Retention time window	Each initial calibration and	3 times standard deviation for	Correct problem then re-analyze
	calculated for each analyte	continuing calibration	each analyte retention time from	all samples analyzed since
		verification	72 hour study	retention time check
	Continuing calibration	Before sample analysis, after	All analytes within 20% of	Correct problem then repeat initial
	verification	every 10 samples, and at the end	expected value	continuing calibration verification

		the analysis sequence - varying		and re-analyze all samples since
		concentrations		last successful CCV
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See table in Section 5	Re-prep and analyze the LCS/LCSD
				and all samples in the affected
				analytical batch
	Surrogate Spike	Every sample, spiked sample,	See table in Section 5	Check system, re-inject, re-extract
		standard, and method blank		
	MS/MSD	One per prep batch	See table in Section 5	None - Narrate the results in LIMS
	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
		for the first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed		of different concentration
	Confirmation	100% for all positive results	Same results as primary	Only report the results that match.
		(not for 8015B)	column analysis	Use the highest results
SW-7000	3-point initial calibration	Daily initial calibration prior to	Correlation coefficient >0.995 for	Correct problem then repeat initial
Metals	(minimum 3 standards	sample analysis	linear regression	calibration
	and a blank)			
	Second source calibration	Once per initial daily calibration	All analytes within 10% of	Correct problem then repeat initial
	verification standard (ICV)	second source.	expected value	calibration
	Continuing calibration	Before sample analysis, after	All analytes within 20% of	Correct problem then repeat initial
	verification	every 10 samples, and at the end	expected value	continuing calibration verification
		the analysis sequence		and re-analyze all samples since
				last successful CCV
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See table in Section 5	Re-prep and analyze the LCS/LCSD
				and all samples in the affected
				analytical batch
	MS/MSD	One per prep batch	See table in Section 5	None - Narrate the results in LIMS
	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
		for the first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed		of different concentration
	Dilution test: 1:4 dilution	Each preparatory batch	Five times dilution sample result	Perform post digestion spike
		Sample concentration must be	must be within 10% of the	addition
		20X MDL	undiluted sample result	
	Recovery Test	When dilution test fails or	Recovery within 15% of expected	Perform method of standard
		sample concentration < 20X MDL	results	additions
SW-9010B	Initial calibration (six	Daily initial calibration prior to	Correlation coefficient >0.995 for	Correct problem then repeat initial
CN Distil	standards and a calibration	sample analysis	linear regression	calibration
SW-9012A	blank)			
Cyanide	Distilled standards (one	Once per initial daily calibration	All analytes within 10% of	Correct problem then repeat initial
	high and one low)		expected value	calibration
	Second source calibration	Once per initial daily calibration	All analytes within 15% of	Correct problem then repeat initial
	verification standard (ICV)	second source.	expected value	calibration
	Continuing calibration	Before sample analysis, after	All analytes within 15% of	Correct problem then repeat initial
	verification	every 10 samples, and at the end	expected value	continuing calibration verification
		the analysis sequence - varying		and re-analyze all samples since
		concentrations		last successful CCV
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See table in Section 5	Re-prep and analyze the LCS/LCSD
				and all samples in the affected

				analytical batch
	MS/MSD	One per prep batch (9010B)	See table in Section 5	None - Narrate the results in LIMS
		Every 10 samples (9012A)		
	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
		for the first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed		of different concentration
EPA-624	Tune GC/MS	Prior to initial calibration and	See individual method for	Analyst cannot perform the test
VOC	BFB for 8260B	continuing calibration verification	tune criteria.	method until the tune passes
EPA-625	DFTPP for 8270C	every 24 hours		method criteria
SVOC	3-point initial calibration	Initial calibration prior to sample	%RSD<30%	Correct problem then repeat initial
	for all analytes	analysis		calibration
	Second source calibration	Once per 3 point initial	All analytes within 25% of	Correct problem then repeat initial
	verification standard (ICV)	calibration	expected value	calibration
	Retention time window	Each Sample	Relative retention time (RRT) of	Correct problem then re-analyze
	calculated for each analyte		the analyte within 30 seconds	all samples analyzed since
			of the RT	retention time check
	Continuing calibration	Daily prior to analysis of	All calibration analytes within	Correct problem then repeat initial
	verification	samples - varying concentration.	20% of expected value	continuing calibration verification
				and re-analyze all samples since
				last successful CCV
	Internal Standards	Every sample/standard	Retention time +/-30 seconds	Inspect GC/MS for malfunctions;
			from retention time of the mid-	mandatory re-analysis of samples
			point	analyzed while system was
			in the CCV/ICAL	malfunctioning.
			(sample/standard)	
			EICP area within -50% to +100%	
			of ICAL mid-point standard	
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See table in Section 5	Re-prep and analyze the LCS/LCSD
				and all samples in the affected
				analytical batch
	Surrogate Spike	Every sample, spiked sample,	See table in Section 5	Check system, re-inject, re-extract
		standard, and method blank		
	MS/MSD	One per prep batch	See table in Section 5	None - Narrate the results in LIMS
	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
		for the first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed		of different concentration
EPA-245.1	Initial calibration (minimum	Daily initial calibration prior to	Correlation coefficient >0.995 for	Correct problem then repeat initial
EPA-245.2	5 standards and a blank).	sample analysis.	linear regression.	calibration.
Mercury	Linear Dynamic Range	Once Annually	Analyte within 10% of expected	Calibration range lowered to meet
			value (not necessary if diluting	LDR results.
			within calibration curve).	
	Proficiency Testing	Once Annually	All analytes within EPA control	Correct problem then repeat initial
	Sample		limits.	calibration.
	Second source calibration	Once per five point initial	All analytes within 5% of	Correct problem then repeat initial
	verification standard (ICV)	Calibration - second source.	expected value	calibration
	Continuing calibration	Before sample analysis, after	All calibration analytes within 10%	Correct problem then repeat initial
	verification	every 10 samples, and at the end	of expected value before	continuing calibration verification
		of the analysis sequence -	sample analysis	and re-analyze all samples since
				last successful CCV
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See table in Section 5	Re-prep and analyze the LCS/LCSD
				and all samples in the affected
				analytical batch
	MS/MSD	One per prep batch	See table in Section 5	None - Narrate the results in LIMS

	IDOC	Every time a new analyst performs the test method for the first time - second source.	See LIMS Test code or QC Charts LCS Accuracy for Limits	Analyst cannot perform the test method until the IDOC passes method criteria
	MDL	Annually or whenever instrument or conditions changed	MDL < Spike Level < 10X MDL	Repeat MDL with new standard of different concentration
EPA 200.7 SW-6010C ICP Metals	Initial calibration (minimum 1 standards and a blank)	Initial calibration prior to sample analysis	Not applicable	Correct problem then repeat initial calibration.
	CRI /LLICV/LLCCV	Set to PQL	Result must be greater than calibration blank, <PQL	Correct problem the repeat initial calibration
			±30% for all analytes	
	Check Standard	Calibration verification	All analytes within 5% of true value	Correct problem, then reanalyze the calibration standard and check std.
	Second source calibration verification standard (ICV)	Once per initial calibration - second source.	Mean value of all analytes within 5% of expected value for 200.7 within 10% for 6010C	Correct problem then repeat initial calibration
	ICSA	Interference analytes Ca, Fe, Mg, Al Beginning, end & periodic intervals	Concentrations of analytes within 20% of true value	Terminate analysis; correct problem reanalyze ICS; reanalyze all affected samples.
	ICSAB	Interference analytes Ca, Fe, Mg, Al All others at 0.5 mg/L Beginning, end & periodic intervals	Concentrations of analytes within 20% of true value	Terminate analysis; correct problem reanalyze ICS; reanalyze all affected samples.
	Linear dynamic range	Every six months	All analytes within 10% of expected value.	Calibration range adjusted to meet calibration results.
	Calibration blank	After every calibration verification	No analytes detected within +/- one MDL	Correct problem then repeat initial continuing calibration verification and re-analyze all samples since last successful calibration blank
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence -	Analytes within 10% of expected value for method 200.7, within 10% for method 6010C	Repeat calibration and re-analyze all samples since last successful calibration verification.
	Method Blank	Once per analytical batch	No analytes detected within +/- one MDL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Duplicate	One per batch Sample concentration must be 4X MDL or greater for valid results.	%RSD must be 20% for water %RSD must be 30% for soil	Reanalyze duplicate sample. Check system, re-prep, re-analyze as needed
	LCS/LCSD	One per prep batch	All samples within 20% of expected value	Re-prep and analyze the LCS/LCSD and all samples in the affected analytical batch
	Dilution test: 1:4 dilution	Each preparatory batch Sample concentration must be 20X MDL	Five times dilution sample result must be within 10% of the undiluted sample result for 6010C and 10% for 200.7	Perform post digestion spike addition
	Recovery Test	When dilution test fails or sample concentration < 20X MDL	Recovery within 25% of expected results	Perform method of standard additions
	MS/MSD	One per prep batch	All analytes within 20% RPD MS-(200.7 70-130%) (6010 C 75-125%) PDS-(200.7 85-115%) (6010 C 80-120%)	Check system, re-prep, re-analyze as needed Sample Conc. > 10X spike Conc., if not cannot validate MS
	IDOC	Every time a new analyst performs the test method for the first time - second source.	See LIMS Test code or QC Charts LCS Accuracy for Limits	Analyst cannot perform the test method until the IDOC passes method criteria
	MDL	Annually or whenever instrument or conditions changed	MDL < Spike Level < 10X MDL 70-130% acceptable for 6010C	Repeat MDL with new standard of different concentration
EPA 200.8	Initial calibration (minimum	Initial calibration prior to sample	Not applicable	Correct problem then repeat initial

SW-6020A	1 standards and a blank)	analysis		calibration.
Metals				
	CRI /LLICV/LLCCV	Set to PQL	Result must be greater than	Correct problem the repeat initial
			calibration blank, <PQL	calibration
			±30% for all analytes	
	Check Standard	Calibration verification	All analytes within 5% of	Correct problem, then reanalyze the
			true value	calibration standard and check std.
	Second source calibration	Once per initial calibration -	Mean value of all analytes	Correct problem then repeat initial
	verification standard (ICV)	second source.	within 5% of expected value for	calibration
			200.8	
			within 10% for 6020A	
	ICSA	Interference analytes	Concentrations of analytes	Terminate analysis; correct problem
		Ca, Fe, Mg, Al	within 20% of true value	reanalyze ICS; reanalyze all affected
		Beginning, end & periodic		samples.
		Intervals(every 12 hours)		
	ICSAB	Interference analytes	Concentrations of analytes	Terminate analysis; correct problem
		Ca, Fe, Mg, Al	within 20% of true value	reanalyze ICS; reanalyze all affected
		All others at 0.5 mg/L		samples.
		Beginning, end & periodic		
		Intervals (every 12 hours)		
	Linear dynamic range	Every six months	All analytes within 10% of	Calibration range adjusted to meet
			expected value.	calibration results.
	Calibration blank	After every calibration verification	No analytes detected within +/-	Correct problem then repeat initial
			one MDL	continuing calibration verification
				and re-analyze all samples since
				last successful calibration blank
	Continuing calibration	Before sample analysis, after	Analytes within 10% of expected	Repeat calibration and re-analyze
	verification (CCV)	every 10 samples, and at the	value for method 200.8,	all samples since last successful
		end of the analysis sequence -	within 10% for method 6020A	calibration verification.
	Method Blank	Once per analytical batch	No analytes detected within +/-	Correct problem then re-prep and
			one MDL	analyze method blank and all samples
				processed with the contaminated blank
	Duplicate	One per batch	%RSD must be 20% for water	Reanalyze duplicate sample.
		Sample concentration must be	%RSD must be 30% for soil	Check system, re-prep, re-analyze
		4X MDL or greater for valid		as needed
		results.		
	LCS/LCSD	One per prep batch	All samples within 20% of	Re-prep and analyze the LCS/LCSD
			expected value	and all samples in the affected
				analytical batch
	Dilution test: 1:4 dilution	Each preparatory batch	Five times dilution sample result	Perform post digestion spike
		Sample concentration must be	must be within 10% of the	addition
		20X MDL	undiluted sample result for 6020A	
			and 10% for 200.8	
	Recovery Test	When dilution test fails or	Recovery within 25% of expected	Perform method of standard
		sample concentration < 20X MDL	results	additions
	MS/MSD	One per prep batch	All analytes within 20% RPD	Check system, re-prep, re-analyze
			MS-(200.8 70-130%) (6020A 75-	as needed
			125%)	
			PDS-(200.8 85-115%) (6020A 80-120%)	Sample Conc. > 10X spike Conc., if not
				cannot validate MS
	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
		for the first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed	70-130% acceptable for 6010C	of different concentration
EPA-608	Five-point initial calibration	Initial calibration prior to sample	RF = 10%	Correct problem then repeat initial
Pest/PCB	for all analytes	analysis		calibration
			Linear - least squares regression	
			r>0.99	
	Second source calibration	Once per five point initial	All analytes within 10% of	Correct problem then repeat initial
	verification standard (ICV)	calibration - from second	expected value	calibration
		source.		
	Retention time window	Each day test is performed.	3 times standard deviation for	Correct problem then re-analyze

	calculated for each analyte		each analyte retention time from	all samples analyzed since
			72 hour study	retention time check
	Continuing calibration	Before sample analysis, after	All analytes within 10% of	Correct problem then repeat initial
	verification	every 10 samples, and at the end	expected value	continuing calibration verification
		the analysis sequence - varying		and re-analyze all samples since
		concentrations		last successful CCV
	Breakdown check (Endrin	Daily prior to analysis of samples	Degradation <15%	Inlet column maintenance; repeat
	and DDT)(1)			breakdown check
	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
				analyze method blank and all samples
				processed with the contaminated
				blank.
	LCS/LCSD	One per prep batch	See table in Section 5	Re-prep and analyze the LCS/LCSD
				and all samples in the affected
				analytical batch
	Surrogate Spike	Every sample, spiked sample,	See table in Section 5	Check system, re-inject, re-extract
		standard, and method blank		
	MS/MSD	Every 10 samples	See table in Section 5	None - Narrate the results in LIMS
	IDOC	Every time a new analyst	See table in Section 5	Analyst cannot perform the test
		performs the test method		method until the IDOC passes
		for the first time - second source.		method criteria
	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
		or conditions changed		of different concentration
	Second column	100% for all positive results	Same results as primary	Only report the results that match.
	confirmation (2)		column analysis	Use the highest results
EPA-160.1	Verification standard	Each batch	All analytes within 10% of	Repeat test. If results are still not
TDS	Single standard		expected value	within 10%, report result and narrate
EPA-160.2	(if available)		Flashpoint result 77-82°F	in LIMS.
TSS	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
EPA-160.3				analyze method blank and all samples
T. Residue				processed with the contaminated
EPA-160.4				blank.
VSS	Duplicate	One per batch	%RSD must be 20% for water	Reanalyze duplicate sample. If results
EPA-160.5		Sample concentration must be	and 30% for soil.	not within RSD limits, report QC
Sett Solids		2X MDL or greater for valid		failure in LIMS or flag as
SW-1110		results.		non-homogenous for soils.
Corrosivity	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
SM-2540E		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
VSS		for the first time - second source.		method criteria
SW-1010				
Flashpoint				
EPA-305.1	Verification standard	Each batch	All analytes within 10% of	Repeat test. If results are still not
Acidity	Single standard		expected value	within 10%, report result and narrate
EPA-310.1	(if available)			in LIMS.
Alkalinity	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
SM-9222D				analyze method blank and all samples
F. Coliform				processed with the contaminated
SM-2223				blank.
T. Coliform	Duplicate	One per batch	%RSD must be 20% for water	Reanalyze duplicate sample. If results
		Sample concentration must be	and 30% for soil.	not within RSD limits, report QC
		2X MDL or greater for valid		failure in LIMS or flag as
		results.		non-homogenous for soils.
	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
		for the first time - second source.		method criteria
EPA-350.1	Five-point initial calibration	Initial calibration prior to sample	RF = 10%	Correct problem then repeat initial
EPA-350.3	for all analytes	analysis		calibration
Ammonia	(Excludes BOD, CBOD)		Linear - least squares regression	
EPA-351.2			r>0.99; $\geq$0.995 for 9056A	
EPA-351.4				
EPA 335.4				
EPA-353.2				
EPA-353.3	Second source calibration	Once per five point initial	All analytes within 10% of	Correct problem then repeat initial
NO3/NO2	verification standard (ICV)	calibration - from second	expected value	calibration
EPA-365.1		source.		
EPA-365.3				

Phosphorus				
EPA 375.4	Continuing calibration	Before sample analysis, after	All analytes within 10% of	Correct problem then repeat initial
SW-9038	verification	every 10 samples, and at the end	expected value	continuing calibration verification
Sulfate		the analysis sequence - varying		and re-analyze all samples since
EPA-376.1		concentrations		last successful CCV
SW-9034	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
Sulfide				analyze method blank and all samples
EPA-377.1				processed with the contaminated
Sulfite				blank.
EPA-410.4	LCS/LCSD	One per prep batch	See table in Section 5	Re-prep and analyze the LCS/LCSD
COD				and all samples in the affected
EPA-415.1				analytical batch
SW-9060				
TOC				
EPA-420.1				
EPA-420.2				
SW-9065				
Phenolics				
EPA-425.1	MS/MSD	Every 10 samples (9038)	See table in Section 5	None - Narrate the results in LIMS
MBAS		One per prep batch (remainder)		
EPA-300.1	IDOC	Every time a new analyst	See LIMS Test code or QC Charts	Analyst cannot perform the test
SW-9056A		performs the test method	LCS Accuracy for Limits	method until the IDOC passes
Ion Chrom		for the first time - second source.		method criteria
SW-1664				
SW-9071A				
OAG				
EPA-405.1	MDL	Annually or whenever instrument	MDL < Spike Level < 10X MDL	Repeat MDL with new standard
BOD	(Excludes BOD, CBOD)	or conditions changed		of different concentration
SM-5210B				
CBOD				
EPA-120.1	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
Conductivity				analyze method blank and all samples
EPA-110.2				processed with the contaminated blank.
Color				
EPA-140.1	Single Standard	Once per analytical batch	All analytes within 10% of	Correct problem then repeat initial
Odor			expected value	calibration
EPA-150.1			Conductance and color standard	
pH			within 5% of expected value.	
EPA-180.1				
Turbidity				
SM-2710B				
SOUR	Duplicate	One per batch	%RSD must be 20%	Reanalyze duplicate sample. If results
EPA-330.5				not within RSD limits, report QC
Cl Residual				failure in LIMS
SW-9095A				
Paint Filter				
PEA-360.1				
DO				
SW-1311	Method Blank	Once per analytical batch	No analytes detected > PQL	Correct problem then re-prep and
TCLP				analyze method blank and all samples
SW-1312				processed with the contaminated
SPLP				blank.
	Post extraction duplicate	One per batch	%RSD must be 20%	Reanalyze duplicate sample. If results
				not within RSD limits, report QC
				failure in LIMS
	Post extraction spike	Once per analytical batch	See individual test methods.	See individual test methods.
	1. Endrin/DDT breakdown check for 8081B only.			
	2. Excludes chlordane, toxaphene, and PCB.			
	3. Sample data associated with QC non-conformances resulting in high bias may be reported if all target analytes are below reporting			
	limits.			
	4. In the event that reanalysis is not possible, I.e. no remaining sample, holding times expired, etc., data may be reported with non-			
	Conformance and its potential affect on the data described in a Case Narrative.			

6.0 SAMPLE BOTTLE AND PRESERVATIVE PREPARATION

6.1 Analytical Environmental Services, Inc. does not provide sampling services and, therefore, has no sampling plan or procedures. If requested by the client, AES does provide appropriate pre-cleaned sample containers. The laboratory assumes responsibility for supplying the proper containers and preservatives.

6.2 Sample Container Preparation. Table 6-1 provides a guideline for the correct containers needed for each analysis.

6.2.1 A laboratory label and proper preservative are added to the sample bottle prior to shipment or pick-up by the client. Some clients may request several cases of bottles, preservative in separate containers, and separate labels. Should this occur, the client would be responsible for label attachment and the addition of preservatives in the field. If the client performs these duties, this is indicated on the bottle label and the chain of custody.

6.2.2 If contamination is observed in trip blanks, a representative from each "lot" of sample containers may be analyzed for the detected parameter(s) to ascertain the cause.

6.2.3 Bottle contamination checks are typically accomplished by filling the bottle with DI water and analyzing for the analytes in question. If any results are above the reporting level, contamination is present and the source must be found.

6.2.3.1 If the analysis of the bottles for the detected test parameter(s) indicates the absence of the parameter(s), then the original cause of the observed contamination was not the bottle or the laboratory.

6.2.3.2 If the analysis of the bottles for the detected test parameter(s) indicates the presence of the parameter(s), then the original cause of the observed contamination is either the bottle or the laboratory.

6.2.3.3 A typical method of laboratory contamination is the introduction of volatile compounds into VOC vials by the use of extraction chemicals such as methylene chloride. Another means of laboratory contamination is the cross contamination of analytes into reagent bottles through poor analytical techniques. An example would be returning aliquots of reagents to their original containers after use. In this instance, contaminants in the reagents are measured as part of the sample result when the reagent is used in the test. Finally, cross contamination can occur during analysis when glassware that is used for the test is not been properly cleaned between samples.

6.2.3.4 If the analysis indicates that the contamination source is the bottle manufacturer, the vendor or manufacturer must be informed immediately. Use of the affected bottles must stop immediately and another lot of bottles used instead.

6.2.3.5 Methods of eliminating sample contamination are discussed in the individual analyte SOPs.

6.2.3.6 Procedures for checking sample bottles for sterility and metals contamination are outlined in the Sample Receiving SOP (Sec. 3.1.2.3).

6.3 When the addition of preservatives is performed by laboratory personnel, the preservation type and amount used is marked on the label. This procedure informs the sample collection agent that the sample bottle has pre-measured preservative in it. Additionally, it provides important safety information for the sample collection agent.

6.4 Preservatives prepared by the laboratory are documented in a Preparation Standard logbook. The logbook contains the preservative preparation information including the preservative lot number and if the chemical was used "as is" from the manufacturer or if it was prepared in the laboratory. See Section 6.7 Preservatives.

6.5 Proper packing of bottles is essential to prevent breakage during shipping. All bottles should be wrapped in bubble wrap and the container, usually a cooler, filled with packing material.

6.6 Certain biological analyses require a sterile bottle for sampling. This includes plate counts, E-coli, and Total Coliform analyses. The laboratory purchases sterilized bottles for these analyses. Never break the seal on these bottles or open them as this can contaminate the bottles.

6.7 Preservatives and removal of interferences.

6.7.1 There are several preservatives used to increase the holding time for an analysis. In most cases, these preservatives are required by the test method, and are added to alter the sample pH or to remove possible interferences. The preservatives used at AES include the following:

6.7.1.1 HCl: Concentrated Hydrochloric Acid (1 ml) is added to VOC vials and other sample bottles to lower the resultant pH to ≤ 2 after the addition of sample to the bottle.

6.7.1.2 H_2SO_4: Concentrated Sulfuric Acid (1 ml) is added to sample bottles to lower the resultant pH to ≤ 2 after the addition of sample to the bottle.

6.7.1.3 NaOH: Solid Sodium Hydroxide (one pellet) is added to sample bottles to raise the resultant pH to ≥ 12 after the addition of sample to the bottle.

6.7.1.4 HNO_3: Six ml per liter of sample of a 1:1 Nitric Acid (1 part concentrated Nitric Acid mixed with 1 part DI water) is added to sample bottles to lower the resultant pH to ≤ 2 after the addition of sample to the bottle.

6.7.1.5 EDTA: One ml per 100ml sample of a 2.5% EDTA solution (2.5g dissolved in 100 ml of DI water) is added to various types of sample bottles to remove any metal interferences.

6.7.2 Low results can be expected when analyzing for BOD, Volatile Organics, and Pesticides in the presence of chlorine. These samples must be tested for the presence of chlorine. This procedure is performed by placing a drop of sample on a starch-potassium iodide paper strip. If the strip turns blue, chlorine is present and treatment is needed. Chlorine removal is accomplished through the addition of sodium thiosulfate (usually 2 – 4 ml of a 0.008% or a 1 N solution). Following the addition of this compound, the destruction of chlorine is verified through a subsequent chlorine check.

6.7.3 Low results can also be expected when analyzing for BOD in the presence of cyanides. Testing for the presence of cyanide is performed by placing a drop of sample on a lead acetate paper strip. If the strip turns black, cyanide is present and treatment is needed. Cyanide removal is accomplished through the addition of ascorbic acid, a few grains at a time, until the paper does not turn black. A few more grains can be added to the sample to ensure cyanide removal.

6.8 Bottle Kit Preparation

6.8.1 The number of bottles required per test, the type of preservatives, and the bottle type are method specific.

6.8.2 Table 6-1 indicates the preservation, holding times, and containers required for the types of tests and matrices analyzed in the laboratory.

TABLE 6-1
Preservation, Holding Time and Containers

Analysis	Matrix*	Holding Time	Container	Preservative
Acidity	Water	14 days	P, G	4°C
Alkalinity	Water	14 days	P, G	4°C
Ammonia	Water	28 days	P, G	1:1 H_2SO_4(pH<2), 4°C
Bicarbonate	Water	14 days	P, G	4°C
BNA	Water	7 days (Ext)	G	4°C
BOD	Water	48 hours	P, G	4°C, check for Cl
BTEX	Water	14 days	G	1:1 HCl (pH<2), 4°C check for Cl^-
BTEX	Soil	48 hours to preserve, 14 days	Encore*	Sodium Bisulfate, 4°C or Methanol, 4°C
TPH-DRO	Water	14 days (ext)	G	4°C
TPH-DRO	Soil	14 days (ext)	G	4°C
TPH-GRO	Water	14 days	G	4°C
TPH-GRO	Soil	14 days	G	4°C
Carbonate	Water	14 days	P, G	4°C
Cation Exchange Capacity	Soil	6 months	G	4°C
Chloride, Total	Water	28 days	P, G	None
Chloride, Total	Soil	28 days	P, G	None
Chlorine, Tot. Residual	Water	Immediately	P, G	None

Analysis	Matrix*	Holding Time	Container	Preservative
Chlorophyll a	Water	Filtration: 2 days Analysis: 21 days	G (amber)	4°C
COD	Water	28 days	P, G	1:1 H_2SO_4, 4°C
Coliform, Fecal	Drinking Water	8 hours	P, sterilized	Sodium Thiosulfate, 4°C
Coliform, Fecal	Non-potable Water	8 hours	P, sterilized	Sodium Thiosulfate, 4°C
Coliform, Fecal	Soil / Sludge	24 hours	P, sterilized	Sodium Thiosulfate, 4°C
E.Coli	Drinking Water	30 hours	P, sterilized	Sodium Thiosulfate, 4°C
Coliform, Total	Non-potable Water	8 hours	P, sterilized	Sodium Thiosulfate, 4°C
Coliform, Total	Drinking Water	30 hours	P, sterilized	Sodium Thiosulfate, 4°C
Coliform, Total	Soil Sludge	24 hours	P, sterilized	4°C
Color	Water	48 hours	P, G	4°C
Conductivity	Water	28 days	P, G	4°C
Corrosivity	Waste	14 days	G	None
Cobalt thiocyaniate active substances (CTAS)	Water	48 hours	P, G	4°C
Cyanide, Amenable	Water	14 days	P, G	NaOH (pH>12), 4°C
Cyanide, Amenable	Soil	14 days	P, G	4°C
Cyanide, Reactive	Waste	7 days	P, G (amber)	4°C
Cyanide, Total	Water	14 days	P, G	NaOH (pH>12), 4°C
Cyanide, Total	Soil	14 days	P, G	4°C
Density / Specific Gravity	Water	7 days	P, G	4°C
Density / Specific Gravity	Soil / Sludge	6 months	P, G	4°C
EDB, DCBP	Water	14 days (Ext)	40 mL VOA	4°C
Ferrous Iron	Water	24 hours	P, G	None, 4°C
Flash Point/Ignitability	Liquid	14 days	P, G	None
Flash Point/Ignitability	Solid	14 days	G	None

Analysis	Matrix	Holding Time	Container	Preservative
Fluoride	Water	28 days	P, G	None
FOC/FOM	Solid	28 days	G	4°C
Formaldehyde	Water	72 hours	G (amber)	4°C
Formaldehyde	Soil	72 hours	G	4°C
Hardness	Water	6 months	P, G	1:1 HNO_3 (pH<2)
Herbicides	Water	7 days (Ext)	G (amber)	4°C
Herbicides	Soil	14 days (Ext)	G	4°C
Hexavalent Chromium	Water	24 hours	P, G	4°C
Hexavalent Chromium	Soil	7 days (Ext)	P, G	4°C
Lead	Air	6 months	**	None
Lead	Wipe	6 months	***	None
Lead	Paint chips	6 months	N/A	None
MBAS (Surfactants)	Water	48 hours	P, G	4°C
Mercury	Water	28 days	P, G	1:1 HNO_3 (pH<2)
Mercury	Soil	28 days	P, G	**4°C**
Metals	Water	6 months	P, G	1:1 HNO_3 (pH<2)
Metals	Soil	6 months	P, G	None
Nitrate	Water	48 hours	P, G	4°C
Nitrate	Soil	28 days	P, G	4°C
Nitrate-Nitrite	Water	28 days	P, G	1:1 H_2SO_4 (pH<2), 4°C
Nitrate-Nitrite	Soil	28 days	P, G	4°C
Nitrite	Water	48 hours	P, G	4°C
Nitrite	Soil	28 days	P, G	4°C
Oil and Grease	Water	28 days	G	1:1 H_2SO_4 (pH<2), 4°C
Oil and Grease	Soil	28 days	G	4°C
PAH	Water	7 days (Ext)	G (amber)	4°C
PAH	Soil	14 days (Ext)	G	4°C
PCB	Water	360 days	G (amber)	4°C

Analysis	Matrix	Holding Time	Container	Preservative
PCB	Soil	360 days	G	4°C
Pesticides, Chlorinated	Water	7 days (Ext)	G (amber)	4°C
Pesticides, Chlorinated	Soil	14 days (Ext)	G	pH 5-9, 4°C
Pesticides, Special	Water	7 days (Ext)	G (amber)	4°C
Pesticides, Special	Soil	14 days (Ext)	G	4°C
pH	Water	Immediately, 15 minutes	P, G	None
pH	Soil	Immediately, 15 minutes	P, G	None
Phenolics	Water	28 days	G (amber)	1:1 H_2SO_4 (pH<2), 4°C
Phosphorus, Ortho	Water	48 hours	P, G	4°C
Phosphorus, Total	Water	28 days	P, G	1:1 H_2SO_4 (pH<2), 4°C
Phosphorus, Total	Soil	28 days	P, G	4°C
Potassium Permanganate	Water	48 hours	40 mL	4°C
Semi-Volatiles	Water	7 days (Ext)	G (amber)	4°C
Semi-Volatiles	Soil	14 days (Ext)	G	4°C
Salinity	Water	28 days	P	4°C
Silica	Water	28 days	P	4°C
Solids, Settleable	Water	48 hours	G	4°C
Solids, Total	Water	7 days	P, G	4°C
Solids, Total Dissolved	Water	7 days	P, G	4°C
Solids, Total Suspended	Water	7 days	P, G	4°C
Field to SPLP Extraction (Tumble)	Liquid / Solid	14 days	G	4ºC
FOR FLORIDA Field to SPLP ZHE Extraction (Tumble)	Liquid / Solid	**48** hours, 14 days after Freezing	G	4ºC, **freeze upon receipt**
OTHER STATES Field to SPLP ZHE Extraction (Tumble)	Liquid / Solid	14 Days	G	4ºC
Sulfate	Water	28 days	P, G	4°C

Analysis	Matrix	Holding Time	Container	Preservative
Sulfide	Water	7 days	P, G	NaOH to pH>9 / Zinc Acetate, 4°C
Sulfide	Soil	28 days	P, G	4°C
Sulfite	Water	Immediately	P, G	4°C
Sulfide, Reactive	Waste	7 days	P (opaque), G (amber)	4°C
Field to TCLP Extraction (Tumble)	Liquid / Solid	14 days	G	4ºC
Field to TCLP ZHE Extraction (Tumble)	Liquid / Solid	14 days	G	4ºC
Temperature	Water	Immediately	P, G	None
TKN	Water	28 days	P, G	1:1 H_2SO_4 (pH<2), 4°C
TKN	Soil	28 days	P, G	4°C
Total Inorganic Carbon	Water	28 days	P, G	4°C
Total Organic Carbon	Soil	28 days	P, G	4°C
Total Organic Carbon	Water	28 days	P, G	1:1 H_2SO_4 (pH<2), 4°C
TOX	Waste	7 days	P, G	1:1 H_2SO_4 (pH<2), 4°C
TPH	Water	14 days	G	1:1 H_2SO_4 (pH<2), 4°C
TPH	Soil	14 days	G	4°C
Turbidity	Water	48 hours	P, G	4°C
Volatile Solids	Water / Soil	7 days	P, G	4°C
Volatile Organics	Water	14 days	G	1:1 HCl (pH<2), 4°C
Volatile Organics	Soil	48 hours, 14 days after preservation	Pre-weighed vials or Encore*	Sodium Bisulfate, methanol, 4°C

* Encore™ Samplers are approved by EPA and allow Volatile soil organics to be transported to the lab without preservative. If an Encore sampler is not used, the soil samples must be weighed in the field and preserved with sodium bisulfate or Methanol (5 ml). This will raise the detection limits considerably. See EPA SW-846 method 5035 for further information.

** Lead in air is usually sampled with a cartridge device that attaches to an air pump that samples an area for a given amount of time. There are several types of cartridges approved by NIOSH, but all are self-contained and require no special treatment.

*** Lead wipes are usually a 1 to 2 inch square piece of material that is free of lead at the time of sampling. A specified area, usually 1 square foot, is “wiped” with this material. The wipe is placed in a of non-contaminating (non-metal) container for shipment to the lab.

P Plastic container

G Glass container

7.0 CUSTODY OF SAMPLES, EQUIPMENT, AND SUPPLIES

7.1 Review of New Work

7.1.1 The Laboratory Manager is primarily responsible for determining the capacity of the facility and its resources to handle new work, although other senior members of management may be called upon to provide expertise and input as needed. This determination consists of a comprehensive appraisal of the client's projected needs. Factors assessed are the ability of the laboratory to comply with the requirements of its accreditations while maintaining the expected level of legal defensibility and analytical validity of all reported data.

7.1.2 Prior to the acceptance of any new requests, tenders, or contracts by Analytical Environmental Services, Inc., the appropriateness of facilities and resources is considered utilizing the information in the following sections. If the facility and/or resources are inadequate to perform the work, the Laboratory Manager may exercise his discretion to refuse to perform all or part of a particular project. The Client Services Manager will be informed of this decision and the Project Managers will inform the client. The laboratory affords clients cooperation to clarify requests and to monitor the laboratory's performance in relation to the work performed (while ensuring confidentiality to other clients).

7.1.2.1 Facilities

7.1.2.1.1 The facility must be suitable for the proper receipt and storage of the number and type of samples proposed to be accepted.

7.1.2.2 Resources

7.1.2.2.1 Stipulated methods, sample preparations, final reports, data packages, and deliverables are reviewed to determine the availability of suitable instrumentation and personnel.

7.1.2.2.2 The laboratory must be must be capable of meeting all analytical requirements for the selected test methods. The specified requirements and methods must be adequately defined, documented, and understood.

7.1.2.2.3 The laboratory shall advise and obtain approval from the client before subcontracting work to another laboratory.

7.1.3 Technical and Management Capability

7.1.3.1 The review of capability must establish that the laboratory possesses the necessary physical personnel, information, and resources to perform the tests in question. Additionally, the laboratory personnel must have the skills and expertise required for performing these tests.

7.1.3.2 The laboratory shall have adequate personnel at all times during the performance of analytical testing to ensure that clients receive data which meets the terms and conditions of the work agreement.

7.1.3.3 The review may consider the results of previous work of a similar nature or, where new testing is being implemented, the results of interlaboratory testing, trial tests, proficiency samples, MDL studies, etc.

7.1.4 Discrepancies

7.1.4.1 Any differences between the request or tender and the capability of the laboratory to fulfill the proposed work are resolved before any testing begins.

7.1.4.2 Modifications are allowed upon consent of the client. Changes are documented in the contract prior to acceptance. Each contract shall be acceptable to both the laboratory and the client.

7.1.4.3 Problems encountered during any stage of reviewing the testing are addressed and resolved to the satisfaction of both the laboratory and the client.

7.1.5 Records

7.1.5.1 The laboratory maintains any records for the initial review of new work entering the laboratory, including any significant changes in the proposed work plan.

7.1.5.2 Communication logs (telephone calls, on-site visits, meetings, e-mails, etc.) are used to record all pertinent discussions concerning the client's requirements. Logs must include the date, time, brief details of the exchange, resolution of any complaints, and identification of the parties involved.

7.1.5.3 Subcontracted work is fully described and documented in advance of receipt of the work from the client.

7.1.6 Once work has been accepted, the Director of Project Management is responsible for setting up the client in the LIMS system, setting up an account with the client, and monitoring the project to ensure that all of the client's requirements are met.

7.2 Sample Receipt

7.2.1 The laboratory has defined protocols for receiving samples and for the "logging in" process. These protocols provide information to the analysts regarding requested analyses, holding times, types of preservation, matrices, etc.

7.2.2 Sample Acceptance Policy - The laboratory will accept or reject samples for analytical testing based on presence, absence, or resolution of the required criteria specified for documentation, labeling, identification, preservation, hold time, container type, or volume. If this information is missing or comes into question, a corrective action report will be started to address any nonconformances. Upon completion of the corrective action, it will be determined if the laboratory accepts the samples. Samples will be considered accepted upon final login review. Unaccepted samples will be noted in the project narrative if other samples received meet the requirements.

7.2.2.1 The laboratory sample acceptance policy outlines the circumstances under which samples are accepted and rejected. This policy is available to sample collection personnel and includes the following:

7.2.2.1.1 Documentation shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any comments concerning the samples.

7.2.2.1.2 Client samples should be properly labeled with unique identification. Indelible ink should be used along with water resistant labels.

7.2.2.1.3 Sample containers should be suitable for the requested test and the analysis hold time must be adhered to. (See table 6-1 for Preservation, Hold Time, and Containers required.)

7.2.2.1.4 Sufficient sample volume must be available for the necessary tests. If the client does not provide enough sample for all the requested tests, it will be noted on the sample receipt checklist. The project manager will contact the client to determine which tests the lab is to perform on the sample and whether or not the client will provide additional sample for the other tests.

7.2.2.1.5 If samples show signs of damage, contamination or inadequate preservation, or any other concern, a corrective action must be initiated to determine if samples are acceptable for the requested analysis. Project managers with the assistance of the Director of Project Management, Technical Director, Quality Assurance Manager, or the Laboratory Manager address and close the corrective action by either accepting or rejecting the samples. (Corrective Actions and Nonconformances section 13.0)

7.2.3 Upon receipt, each sample is identified by a laboratory-issued project number and a unique individual sample number. Properly followed, the preceding procedures provide court defensible documentation related to sample release to the lab, proper preservation and handling, and traceability throughout the analytical and reporting process.

7.2.4 Samples usually arrive at the laboratory in one of three ways: 1) delivered by carrier (UPS, Federal Express, and Mail), 2) delivered by courier, or 3) delivered by client personnel. In all cases, a document called a "Chain of Custody" (COC) must accompany the samples. This document, supplied by the laboratory to clients, is designed to provide to the laboratory all the necessary information about the client, samples, and which analyses are required. In addition, this document provides evidentiary information indicating who had the samples in their possession at any time and when possession was changed. In some instances, the client provides their own chain of custody.

7.2.5 Once samples have been relinquished to the laboratory, they are checked for condition including the type(s) of preservation employed (temperature, pH, etc.), correctness of containers, and if the COC has been properly completed and signed.

7.2.5.1 Almost all soil and water matrix samples require a transport temperature of 4 ± 2° C. The samples should be packed in ice in a thermal container. Typically, an insulated ice cooler is used for sample transportation. The cooler should have a temperature blank included for use as a sample temperature check. The temperature blank is a plastic bottle filled with water.

7.2.5.1.1 Temperature is measured with a calibrated thermometer. The thermometer is individually identified and labeled with its calibration expiration date. The temperature of the blank must always be recorded during the login procedure. If the temperature is outside the 4 ± 2°C range, this should be annotated so that the project managers can notify the client.

7.2.5.1.2 Samples that are hand-delivered to the laboratory immediately after collection may not meet these temperature criteria. In these cases, the

samples shall be considered acceptable there is evidence that the chilling process has begun (such as arrival on ice).

7.2.5.2 Before placement in the storage area, samples must be checked for integrity. If any bottles are broken or have leaked, the client must immediately be contacted. This is particularly important if there are no duplicates of the sample in order to obtain instructions from the client on how to handle the situation. It may be necessary to re-sample for the incomplete tests.

7.2.5.3 Sample labels are checked against the Chain of Custody for accuracy and discrepancies. Custody seals must be intact if used. This procedure is best accomplished by sorting samples by their location rather than by their testing requirements. For example, all samples labeled "MW-1A" are combined and may include VOCs, metals, SVOCs, etc. Make sure that all sample labels match the COC for number of analyses, sample ID, matrix, etc. If a discrepancy is found, the variance is noted on the Sample Receipt checklist and the client is contacted to clarify the problem.

7.2.5.4 Samples are checked for type and proper degree of preservation. This only applies to aqueous samples and never to volatile organic samples (VOC samples are checked after the vial has been opened and the sample analyzed). There are several types of preservation required for the different analyses. Most involve either a high or low pH.

7.2.5.4.1 To check the sample for pH, take a clean disposable Pasteur pipette and touch its tip to the top of the aqueous surface. Sample should be drawn by capillary action up the tube. Remove the pipette, recap the sample and touch the Pasteur pipette to some pH paper. Read the paper to the nearest pH unit.

7.2.5.4.2 Check the preservation chart (Section 6) to see if the pH is in the range required for the sample. If not, notify the Project Manager immediately. The Project Manager may require the addition of proper preservative to the sample. If the holding time is affected by inappropriate preservation, this should also be communicated to the client and analysts through the Project Manager.

7.2.5.5 Samples are checked for holding time. Holding times begin the moment the sample is taken, not when it is received. While most analyses have a holding time of several days, holding times vary widely from as little as 15 minutes to as long as 6 months. The time involved in shipment of a sample to the laboratory can greatly reduce the amount of time the analyst has to perform the procedure. It is therefore critical that holding times be noted accurately and the appropriate analyst or manager notified immediately if holding time is running out (less than 24 hours left).

7.2.5.6 The results of all observations are noted on a "Sample Receipt Check List" during the logging in procedure.

7.2.5.7 If the COC matches the samples it represents, the sample custodian, through LIMS, will issue individual numbers for each sample received. These numbers are the

project number followed by a single digit assigned to each bottle. This indicates that the samples are from the same site. For example, a group of samples is logged in as project "C8855". Each sample within the project is given a sequential number starting with the number "1". Thus, "C8855-1" is the first sample of this group.

7.2.5.7.1 The sample bottles are also given a letter designator beginning with the letter "A" that corresponds to each sample "fraction" received at the laboratory. For example, samples collected for metals and SVOC in two bottles would be designated as "A" and "B". The two sample bottles in the above example would be designated as "C8855-1A" and C8855-1B".

7.2.5.7.2 The assigned alphanumeric sample names are written on the COC, usually in the far right column, and on the sample label or top.

7.2.5.7.3 To ensure that the sample identifiers remain intact, use an indelible ink pin, such as a Sharpie™, when marking the samples. Wet or oily sample containers will preclude the use of pen ink.

7.2.6 All samples are properly logged into the computer with all pertinent information, including any comments about improper preservation or holding times. This information is compiled into a spreadsheet called the "daily" and the information is distributed to the analysts. A folder is prepared with a cover sheet that gives the project number and lists the analyses needed. All information pertaining to the project is placed inside the folder including the COC, client contact information, and any special documentation.

7.2.7 Samples are then placed in the sample holding area, either in the appropriate cooler or on the correct shelf. If the project requires a continuous Chain of Custody, they must be logged out of the area by the analyst and logged back in when analysis is completed using the logbook provided. If the sample is completely exhausted, this must be noted in the logbook.

7.2.8 Any deviations must be brought to the attention of the client and/or the Project Manager so the client may be contacted for directions on how to proceed. For example, some samples may be unsuitable for testing if the temperature has not been maintained.

7.2.9 After all sample information is logged into the computer, a printout of the entered data is made. A second individual must verify the accuracy of the sample information entered. If the log-in, COC, and all sample information are approved, the checking individual initials the work and the project folder is given to the Project Manager.

7.2.10 Occasionally, samples require special storage times after the analyses are complete. This should be noted when these samples arrive at the laboratory to avoid them being prematurely discarded. To apprise all affected personnel, annotate this information into LIMS. These samples are to be stored in the special holding area designated by the Sample Receiving Department. A Project Manager will notify the Sample Receiving Department which samples are required to be placed in this area.

7.2.11 Sample bottles are segregated according to their required analyses. Samples to be analyzed for volatile organics are placed in a separate cooler/refrigerator from semi-volatile organics or inorganics because of the high probability of cross-contamination from inorganic and waste

samples. Samples for metal analyses do not require cooling. These samples may be placed on the shelf at room temperature.

7.2.12 Once samples have been removed from a cooler, the cooler must be cleaned before reuse. Typically, rinsing and air drying of the cooler will be sufficient. Make sure the cooler is returned if the client requests it.

7.3 Review of Sample Login

7.3.1 When samples (a project) arrive at the laboratory, the project is created in the laboratory information management system (LIMS) and reviewed by a project manager as discussed in the Section 7.3.3.

7.3.1.1 A "Review of Sample Login" report is filled out by the sample custodian and this report is turned in to the project manager. The project manager reviews the information to ensure that all analyses, sample IDs, etc. are correct.

7.3.1.2 If any problems were found, they are corrected. A copy of the problem and its resolution is transmitted to the Sample Receiving Manager.

7.3.2 Sample Receipt Checklist (SRCL)

7.3.2.1 The sample receipt checklist is a list of all information pertaining to the arrival of a project at the laboratory. If any problems are found, such as errors on the chain-of-custody (COC), or any situation does not comply with the procedure or method, such as problems with sample preservation or holding time, the project manager is notified immediately in order to contact the client. The following list represents the questions asked on the SRCL:

7.3.2.1.1 Was the shipping container/cooler in good condition?

7.3.2.1.2 If there were custody seals on the shipping container/cooler, were they intact?

7.3.2.1.3 If there were custody seals on the samples, were they intact?

7.3.2.1.4 Was the container/temperature blank in compliance?

7.3.2.1.5 Was the chain-of-custody present?

7.3.2.1.6 Was the chain-of-custody signed when relinquished and received?

7.3.2.1.7 Did the chain-of-custody agree with sample labels?

7.3.2.1.8 Were samples received in the appropriate containers to perform the requested analysis? If VOA vials were received, were all vials void of headspace?

7.3.2.1.9 Were all sample containers received intact?

7.3.2.1.10 Was sufficient sample volume received to perform requested analysis?

7.3.2.1.11 Were all samples received within the EPA recommended holding times and within the recommended temperature ranges?

7.3.2.1.12 Was turnaround time marked on the chain-of-custody?

7.3.2.1.13 If samples were submitted for volatiles analysis, did they have zero headspace?

7.3.2.1.14 Was the pH acceptable for water samples upon receipt?

7.3.2.1.15 Were samples in good condition?

7.3.2.1.16 Is a known blank included for diffusive samples or AIHA lead analysis?

7.3.2.2 All information at the top of the SRCL, such as client name, date/time received, and carrier name, must also be checked for accuracy. All out-of-compliance and non-conforming events are documented on the SRCL as well as in the PM non-conformance corrective action logbook. The client is contacted to discuss the issue or conflict. Resolution, as agreed upon by the client, is documented in the PM corrective action logbook and SRCL. Either the Director of Project Management or the Laboratory Manager closes out all corrective actions.

7.3.3 Procedure for Creating and Reviewing Projects in LIMS

7.3.3.1 Open the project in LIMS and verify that the client name listed on the COC is the client selected in the LIMS. Check for any client related notes, such as "report samples on a dry-weight basis" or "provide final report in duplicate" in the "Client ID" field. Verify that this information has been passed on in the work order.

7.3.3.2 Check the project name and check for any project specific requirements in the project notes. Ensure that this information has been carried over into the work order.

7.3.3.3 Go to the "ReportOptions" screen and, if known, enter the state where the samples were collected. In the field "Rpt Name", select the proper reporting format. The preferred format is "AES base report". However, try to select the format that will reduce the overall size of the report, such as "base report consolidated". Unless the client has requested "J" flags, turn off the "Qualifiers" selection key.

7.3.3.4 Go to the "InvoiceInfo" screen. Enter the P.O. number if the client has provided one. If the client has pre-paid, enter this information into the "PrePaid" field and ensure that the accounting department has been informed. Enter any markups for rush fees. Enter into the "MiscCharges" field any sample media charges, courier fees, shipping charges or other expenses. Enter appropriate comments into the "MiscComments" field. The following information is entered into LIMS using the following format:

7.3.3.4.1 Rush Fees: Rush fees applied for same business day TAT.

7.3.3.4.2 Rush fees applied for next business day TAT.

7.3.3.4.3 Rush fees applied for two business day TAT.

7.3.3.4.4 Sample Media Charges: These charges are as follows.

7.3.3.4.4.1 Sample media charges for syringe vial combinations, 2 @ $6.50 each.

7.3.3.4.4.2 Sample media charges for Encore samplers, 3 @ $8.50 each.

7.3.3.4.5 Shipping Fees: FED-EX shipping fees included.

7.3.3.4.6 Courier Fees: Courier fees included.

7.3.3.4.7 Sampling Fees: Sampling fees included.

7.3.3.5 Select the "LOGIN" key. Remove the COC from the folder. Systematically check each sample and fraction in the following order:

7.3.3.5.1 Verify the date and time received.

7.3.3.5.2 Verify the sample I.D. on the COC against the information that is entered into the LIMS.

7.3.3.5.3 Verify the sample description against what is entered for the tag number. If no sample description is given put "N/A" in the field.

7.3.3.5.4 Verify the date and time collected.

7.3.3.5.5 Verify the sample matrix.

7.3.3.5.6 Verify container type. Container type should match the type of container requested for the analysis. Any discrepancies should be noted on the SRCL.

7.3.3.5.7 Verify the number of containers.

7.3.3.5.8 Verify the storage area. This should be consistent with AES' storage policy. Volatile samples are stored in R8, Wet chemistry samples are stored in R1, extractable organic samples are stored in R10, and metal samples are stored on S1.

7.3.3.5.9 Enter any essential sample or analytical information into the sample comments field, such as "expect high concentrations", "perform 5x concentration", or "perform library search".

7.3.3.5.10 Verify that each sample and fraction is logged in for the requested analysis. This must be done independently of the above steps. Attempting to perform both tasks at the same time will only increase the probability of errors.

7.3.3.5.11 Expand the test field column so that the entire code for every analysis is visible.

7.3.3.5.12 Verify, one sample fraction at a time, that each of the appropriate test codes and their corresponding prep code are entered. If there is a test code that is missing a prep code, return to the project and pull the appropriate prep code. Add the prep code to the test field column. If the test code does not appear to have a prep code, inform the Director of Project Management.

7.3.3.5.13 For each test, check the selection list and ensure that the appropriate compounds have been chosen. Check every test.

7.3.3.5.14 If you are aware that a project requires specific detection limits, verify them at this time.

7.3.3.5.15 If air samples are received, click on the "Air Data" screen and verify the air volume in liters against what is listed on the COC.

7.3.3.5.15.1 If no sample volume is given, enter the sample flow rate in liters, change the units to L/min and enter the time sampled in hours and minutes. Hit the "Calc Air Unknowns" to calculate the volume.

7.3.3.5.16 Enter the media type and size. This information is found in the "tests" screen in the media field. If the media provided by the client is not the same media as listed in the method, inform the Director of Project Management immediately.

7.3.3.6 After you have completed the above information for all samples, the work order is ready for approval.

7.3.3.7 Return to the main window of the work order.

7.3.3.8 Enter the date and time that you are approving the login review.

7.3.3.9 You will be prompted to enter your password. Enter your password and the work order will now appear in the "work to be completed" list.

7.4 A Corrective Action Report is generated in LIMS for any sample receiving non-conformance. Section 13 of this Manual describes the Corrective Action Process in detail.

7.5 Health and Safety

7.5.1 All samples should be considered to be hazardous. Until a sample is analyzed, it is impossible to determine what type of contamination is involved. With this in mind, always wear the following safety equipment when handling samples.

7.5.1.1 Safety Glasses: OSHA approved safety glasses must be worn when working with samples. Safety glasses prevent an invasion of the sample into the eye and protect the eyes in case of a sample explosion.

7.5.1.2 Latex Gloves: Latex Gloves must be used when handling samples. Latex gloves protect the hands from the effects of corrosive materials, such as strong acids or bases. In addition, gloves prevent the introduction of hazardous materials into the body by absorption through the skin.

7.5.1.3 Sensible Clothing: Long pants and close-toed shoes (no sandals) must be worn at all times while working in the sample receiving area. Many of the samples received by the laboratory are 1 liter or greater in size. A liter of water weighs slightly more than 2 pounds. Dropping a liter of water on an unprotected toe from waist height can fracture the toe. Never wear any clothing that you are not afraid to ruin. Many of the preservatives used in the laboratory are acidic and will eat a hole in most natural materials. If the fiber is man-made, such as nylon, any strong solvent will melt it.

7.5.1.4 Lab Coat: Required when in the laboratory or handling samples or chemicals. Not only does it protect your clothing, but it also provides an additional cloth barrier against splashes and spills.

7.6 Sample Custody

7.6.1 AES has implemented sample chain-of-custody procedures to provide accurate, verified, and traceable records of sample possession and handling, from sample container shipment through laboratory receipt and sample disposition.

7.6.2 Documentation of sample collection, shipment, laboratory receipt and custody is accomplished utilizing a chain-of-custody record. A sample is considered in custody if the following conditions have been met.

7.6.2.1 The sample(s) are in the physical possession of the sampler or courier.

7.6.2.2 The sample(s) are in view after being in the physical possession of the sampler or courier.

7.6.2.3 The cooler(s) or sample bottle(s) are sealed, so that sample integrity is maintained, while in the possession of the sampler or transferee.

7.6.2.4 The cooler(s) or sample bottle(s) are in a secured area restricted to authorized personnel.

7.6.3 Custody Record Maintenance

7.6.3.1 Laboratory records, including copies of the chain-of-custody forms and any associated documentation, are maintained in a secure area with any associated project records.

7.6.3.2 Laboratory data are recorded in bound notebooks and entries are made in waterproof ink.

7.6.3.3 Laboratory data entry errors are deleted with a single-line through the error. The correction is initialed and dated by the analytical staff member making the change.

7.6.3.3.1 Correction tape or other substances designed to obliterate documentation are strictly prohibited in the laboratory and custody areas.

7.6.3.4 Laboratory information is documented on pre-prepared forms. All forms for recording laboratory data include a space for the date and for initials that must be

completed by the data recorder. Laboratory documentation not recorded on pre-prepared forms is also dated and initialed.

7.6.4 The sample custodian, under either routine or special legal chain-of-custody procedures, receives all samples. Legal custody is a special type of sample custody in which all events associated with a specific sample are documented in writing.

7.6.5 Laboratory Provided Sample Containers

7.6.5.1 Sample containers provided by AES are manufactured from EPA-designated materials, contain EPA-prescribed preservatives, and are affixed with an AES identification label.

7.6.5.2 Pre-cleaned sample containers are purchased by AES. When deemed necessary by the Technical Director, containers from each lot are pre-certified in house prior to use. A lot number is affixed to each container for purpose of traceability.

7.6.6 Chain of Custody Documentation, Traceability, and Sample Integrity

7.6.6.1 Formal chain-of-custody procedures are initiated by a sample custodian responsible for the organization and relinquishing of sample containers to the client or field personnel.

7.6.6.2 Properly record all fields of information on the chain-of-custody form. Proper completion of the form is the responsibility of the client's field sampling manager and is required prior to relinquishing the samples.

7.6.6.3 If the site location is different from the client address, the site location is recorded in the "Project Name" space on the chain-of-custody form, or on the right hand side of the form if additional space is required. The sample identifications assigned in the field are recorded in the "Sample Identification" column.

7.6.6.4 Common carriers may identify themselves by signing the "Relinquished By" space on the chain-of-custody form.

7.6.6.5 Maintain chain-of-custody for samples transported from the field to the laboratory by common carrier. Completed custody forms must accompany each sealed cooler by placing them in a plastic bag taped to the inside lid of the cooler.

7.6.6.6 Maintain a copy of each air bill package tracking form associated with a shipment of samples in the appropriate client files.

7.6.6.7 The custody-technician is responsible for the inspection of shipping containers upon laboratory receipt for overall integrity to ensure that the contents have not been altered or tampered with during transit. If tampering is apparent, the sample custodian immediately contacts the assigned project manager who is responsible for notifying the client.

7.6.6.7.1 The cooler inspection form, filed by the sample custodian, describes the deficiency and annotates any corrective action required by the client. Document

any appropriate changes on the accompanying project chain-of-custody form, which is dated and signed by the sample custodian or project manager.

7.6.6.8 If shipping containers arrive intact, the sample custodian in the receiving area immediately opens them. The chain-of-custody form and temperature bottle are removed for inspection. Upon receipt, the container temperature is documented in a sample registry or, if requested by the client, documented on the chain-of-custody form.

7.7 Continuous Chains of Custody

7.7.1 A "Continuous Chain of Custody" sets protocols for keeping an unbroken, or continuous, chain of custody. The intent of this procedure is to enable AES employees to track samples from the time and date of receipt to the time and date of disposal, particularly where legal cases are involved. In doing this, a constant record is kept of when and by whom samples are removed from the Sample Receiving Department.

7.7.2 Project Managers will notify the Sample Receiving Department when jobs require this unbroken Chain of Custody.

7.7.3 A sequential laboratory identification number is assigned to the project and recorded on the chain-of-custody form, on each sample container submitted with the project, and in the Sample Registry.

7.7.3.1 Accurate and complete sample documentation must be provided on the chain-of-custody form in order to log samples into the sample registry. The sample registry includes all information necessary to maintain chain-of-custody including laboratory ID, client (field) ID, and initials of the sample receipt custodian.

7.7.3.2 Ancillary information, such as sample collection date and requested analyses, is transferred directly from the chain-of-custody form into the LIMS and appears on the client project-specific acknowledgement.

7.7.4 Once the chain of custody is verified, the project is logged into the LIMS to transfer the desired work order request to the laboratory.

7.7.4.1 The sample custodian checks the information on each sample's label against that on the chain-of-custody form for discrepancies.

7.7.4.2 The sample custodian also inspects all samples for leakage or obvious seal (if provided) tampering. All samples are unpacked in a well-ventilated sample receipt area.

7.7.4.3 Samples received in plastic containers, or those that appear to be accumulating or evolving gas, are treated cautiously and inspected under a chemical hood since they may contain toxic fumes or be of an explosive nature.

7.7.4.4 A "Cooler Receipt Form" is completed to document custodial concerns at sample login.

7.7.5 Custody discrepancies noted by the sample custodian are transmitted to the project and sample manager and are resolved with the client prior to laboratory work assignment. Discrepancies are documented on the Anomaly Report.

7.7.5.1 The Project Manager and the Sample Custodian attempt to resolve custody discrepancies expeditiously to avoid holding time compromises. After a decision

concerning a sample has been made, the Project Manager or Sample Custodian makes an initialed note on the in the work order narrative. The person, who was notified, time, date, and resolution, if applicable, is documented. This information is also documented on the Sample Custody Excursion form.

7.7.5.2 A faxed or hard copy of custodial resolutions or project order alterations is secured from the client prior to work initiation. Copies of this documentation are mailed to the client and maintained in the client file.

7.7.6 After addition of the project sequential identification number, the samples are distributed to the appropriate sample storage areas. Sample storage temperature logs are maintained for all sample storage refrigerators to assure proper temperature maintenance throughout the analytical process.

7.7.7 As soon as possible, all samples received by AES are checked, by the appropriate preparation or analytical department, for proper pH adjustment. The pH of each sample is measured, documented, and adjusted if necessary. To avoid compromising sample integrity, volatile samples are checked for proper pH adjustment only at the time of analysis. The pH of volatile samples is not adjusted.

7.7.8 Only authorized personnel are permitted within the laboratory areas where sample access is possible. Sample storage areas are designed to segregate volatile and non-volatile samples. Standards and extracts are also departmentally controlled and stored separately.

7.7.9 The set of analyses required for a group of samples is project-dependent. After sample registry login and verification, samples are transferred from the receiving area to the appropriate sample preparation area. Those samples not requiring preparation are immediately sent to the sample analysis storage area. Using LIMS-generated sample preparation worksheets for guidance, samples are extracted, digested, or distilled as appropriate. The extracts, digestates, or distillates are then transferred to the appropriate analysis section, where analysis is performed.

7.7.10 For projects where the client requires in-laboratory custody records, the AES project manager informs the sample custodian that they need to coordinate custody activities prior to sample receipt. For these samples, staff complete department-specific in-laboratory sample tracking forms. Samples and sample preparations are stored in approved sample storage areas.

7.7.11 Sample holding times are tracked via the LIMS. Sample collection dates are routinely entered into the LIMS with all sample logins. This information allows holding times specific to each departmental analysis to be tracked by department managers, supervisors, chemists, and analysts through the use of daily status sheets, reference sheets, and preparation worksheets.

7.7.11.1 Date analyzed is recorded via instrument outputs or analysis forms as an integral part of the raw data.

7.7.11.2 The date of analysis is entered into the LIMS and compared to the date sampled to validate that holding times were not compromised.

7.7.12 Upon completion of analytical work, custody of unused sample portions, extracts, or digests is relinquished to a central secured storage area. Here the samples, digests, or extracts await

disposal, which is performed with assistance of the LIMS. The LIMS stores client specific disposal instructions, compiles results from the analyses of composite samples, prepares sample disposal lists, invoices for disposal and sample return costs, and provides a disposal record for all excess samples.

7.7.13 By careful assignment of user passwords and file access/lock codes, AES maintains a high level of data security in the LIMS. Thus, only authorized AES personnel can access client files to view data. In addition, data entry and editing is restricted to highly trained data management personnel.

7.7.13.1 Data may be downloaded in a variety of standard formats including ASCII, spreadsheet, database, and text files, such as *.ASC, *.WK1, *.DBF, *.TXT, etc.

7.7.13.2 Additionally, laboratory data may be formatted to match client-specific requirements. These requirements are defined and agreed upon prior to project commencement.

7.7.13.3 Laboratory-generated data is thoroughly reviewed prior to preparation of electronic or diskette deliverables. The download process includes both electronic and logical error check routines to confirm that the data files delivered are consistent with the client's format and data content request.

7.7.13.4 A signed hardcopy report is provided with all electronic or diskette deliverables and an electronic and documentation audit trail of each download event are maintained.

7.8 Data Security

7.8.1 In order to ensure data integrity and security, all files selected for data downloads are transferred from the LIMS to an isolated PC computer system. Access to downloaded files is then controlled via required matches of log-on sequences and confidential passwords. The entire download process is regularly reviewed and maintained by the computer department for system performance.

7.8.2 The LIMS manager maintains internal documentation for all LIMS programs. This documentation includes descriptions of any program additions, deletions, or modifications, the dates of revisions, and the initials of the responsible programmer. To verify proper functioning of the program hardware and software, a simulation account is maintained. When hardware or software is modified, the LIMS uses actual data in the simulation account to verify that the modifications are functioning as anticipated. Anti-virus software serves as an additional protective measure.

7.8.3 Data is entered into the LIMS through direct instrument interfaces and manual entry of data from the chemists' worksheets. Immediately following data entry, approval sheets are printed with the entered data as it appears in the LIMS. Assistant project managers compare all data on the approval sheets against the chemists' worksheets for data transcription errors.

7.8.4 Data worksheets, data approval forms, and final reports are routinely printed for verification and signatures. Hard copies of final reports, field data, chain-of-custody forms, and any ancillary documentation pertinent to the project are kept in a secured storage area and placed chronologically within alphabetically arranged client files.

7.8.5 AES maintains a security policy. Under this policy, all external doors are either visually monitored by AES staff or kept locked. Visitors are required to sign in. They are accompanied at all times by an AES staff member.

7.9 Container Receipt

7.9.1 When the laboratory receives a container, it is entered into the Received Container Logbook. This program issues an AES ID Container number unique to that case of containers. It also checks for contamination from containers that do not provide a Certificate of Quality Environmental Compliance.

7.9.2 The following is a step-by-step guide for entering all information associated with the container:

7.9.2.1 A unique AES ID # is given to each box of containers. This number is given in numerical sequence by adding one to the previous number.

7.9.2.2 Under "Container Description", enter a brief description of the bottle type. Include: bottle size, plastic or glass, clear or amber, preservatives, and pre-cleaned, if noted.

7.9.2.3 Enter the date that the containers were received at the laboratory in the "Date Received" box.

7.9.2.4 Under "Vendor Name", enter the name of the vendor that the containers were ordered from. The sample-receiving manager has this information.

7.9.2.5 Enter the vendor lot number under the "Vendor Lot #" box. This number is found on a vendor provided label on the outside of each case of bottles.

7.9.2.6 Under "Date Expires", enter the date that the containers will expire. This date will be one year after the containers were received at the laboratory, unless otherwise stated by the manufacturer.

7.9.2.7 Enter the number of containers in each case under the "No. of Containers in Lot" box. This information is found on a vendor provided label on the outside of each case of bottles.

7.9.2.8 A Certificate of Quality Environmental Compliance is found inside of each box of glass containers. This information is filed in the sample-receiving department. All plastic containers will be checked for contamination in each new lot that is received by the laboratory. The AES lab number will be written in the "Contamination check OK" box. The information for the contamination check will be found in the LIMS system.

7.9.2.9 Enter the initials of the person that received the containers in the "Initials" box.

7.9.2.10 After each case of containers has been properly entered into the Received Container Logbook, the AES ID # and the expiration date should be written clearly on each case of containers in permanent ink. The containers should then be placed in the for use bottle storage area.

7.9.3 A logbook of records shall be kept in the sample-receiving department. It should be checked periodically by the sample receiving department manager to ensure that it is properly maintained.

7.10 Subcontracting to Other Laboratories

7.10.1 Subcontract Laboratories

All subcontract laboratories are required to supply the Quality Assurance Manager, upon written request, with adequate proof of accreditation in applicable state, AIHA, TNI, or other programs, depending upon the client and origination of the samples. Documents shall be requested from all subcontract laboratories. The requested documents will include, but may not be limited to, a current Quality Assurance Manual, the scope of approved testing, proof of insurance, and AIHA, TNI and/or other applicable state accrediting authority certificates.

7.10.1.1 The following is a list of laboratories which may be utilized for the subcontracting of AES samples:

<u>TNI</u>	
Test America	800-765-0980
Pace Analytical	704-875-9092
ENCO	
<u>Non-TNI</u>	
EMSL Analytical, Inc.	301-937-5700
Clayton Group Services (GA)	770-449-7500
Sailor's Engineering (GA)	770-962-5922
S&ME	

7.10.2 Protocol for subcontracting work received at the laboratory to another facility.

7.10.2.1 When samples are received which have testing requirements that cannot be performed in-house, the samples must be sub-contracted to another laboratory. The facility must be properly accredited for the tests to be performed, and the QA office shall retain records demonstrating that this requirement was met.

7.10.2.2 The sample-receiving department prepares an aliquot and a chain-of-custody to send the sample(s) to the sub-contracted facility. This chain-of-custody will be submitted to the sub-contract facility. All information, including project name, project number, sample ID, collection date, collection time and analysis must be included on the COC. The project manager must review the chain-of-custody before the sample is sent out. Also, a purchase order number must be obtained from the accounting department and placed with the COC.

7.10.2.3 If the client did not provide sufficient sample to send out, the sample must be split. See the procedure for splitting samples to correctly obtain a representative portion of the sample contained within individual standard operating procedures (SOPs).

7.10.2.4 The client must be contacted in writing of the intent to subtract any portion of the testing to another party. The results from the subcontracted laboratory must be reported utilizing a copy of the original report received from the subcontract laboratory.

7.10.2.5 The project is entered into the LIMS system in the Sample Login Procedure with a note in the comment section "SUB OUT."

7.10.2.6 Fill out a FedEx label with the name, address, and phone number of the subcontracting facility, and all the necessary information for the shipper. Prepare a cooler with packing material to ensure that the containers will arrive at the facility unbroken and in a condition that meets the method requirements.

7.11 Purchasing Services and Supplies

7.11.1 Procurement Document Control
Vendors of analytical material supplied to AES Inc. are regarded as a resource to and an extension of the laboratory organization. The standards for quality identified in this document shall be applicable to vendors.

7.11.2 The purpose of the procurement control document is to assure the quality and traceability of procured items (equipment, materials, or services) in instances in which the specifications could affect the quality of the services provided by AES, Inc. This includes such quality related items as the calibration of instruments by outside laboratories, purchase of standards, subcontracted services, and materials requiring testing before use.

7.11.3 Control of purchased materials, equipment, and services is a system designed to insure that these products and services conform to the procurement requirements. This system includes provisions for vendor evaluation and selection, objective evidence of quality furnished by the vendor, inspection of the vendor's or sub-vendor's facilities, and examination of products or services upon delivery. Prior to the use of such products and services, documented evidence of conformation to the procurement requirements must be provided. This evidence is maintained in the analytical department office records.

7.11.4 It is the responsibility of the Accounting Department to insure the development and implementation of procedures to control purchased products and services. It is the responsibility of the purchasing agent to specify quality objectives for procured items and services. Purchased materials that fail to meet established criteria are documented by Non-conformance reports issued by the purchaser.

7.11.5 Procedures and Responsibilities

7.11.5.1 It is the responsibility of the purchasing agent to provide assurance, when required, that all applicable regulatory requirements, industry codes, and standards appear with the purchase documentation for the affected services and products.

7.11.5.2 The Purchasing Department retains Purchase Orders for control purposes.

7.11.5.3 Purchased items which do not meet the minimum standards set forth by the purchasing agent are processed according to procedures set forth in Section 13.0, "Corrective Action."

7.11.5.4 The appropriate manager or supervisor and QA Manager review purchase orders to insure that quality related services or products meet the criteria of the laboratory's accreditations.

7.11.5.5 Purchase orders for standard catalog items do not require QA review unless they include thermometers, thermistors, hydrometers, pipettors, or analytical balance weights.

8.0 ANALYTICAL PROCEDURES

8.1 Method Sources and supporting procedures include the following:

8.1.1 The analytical methods currently accepted and approved by the US EPA, NIOSH, and "Standard Methods".

8.1.2 Other reference procedures for non-routine analyses including methods stipulated by specific states, such as Underground Storage Tank methods, or by ASTM.

8.1.3 Appendix XI includes the list of controlled outside reference documents maintained by AES. Control and updating of the reference document is completed annually by the Technical Director. Electronic document updates or web links to current revisions are posted to the laboratory portal server library, and Appendix XI is updated with the annual update to the QA Manual.

8.2 Document Control

This section describes the procedures for control and maintenance of documentation through a document control system, which ensures that all standard operating procedures, manuals, outside reference documents, and documents clearly indicate the time period during which the procedure or document was in force. Regardless of which analytical procedures are used in the laboratory, the methodology shall consist of carefully documented Standard Operating Procedures (SOPs) and approved methods which may be periodically modified, updated or replaced entirely due to advances in technology or changes in regulatory protocols. Some clients may require pre-approval of method revisions before modifications are used to generate data. Documentation of analytical procedures for generating laboratory data shall be clear, concise, adequately referenced, and reflect the actual steps employed by the analyst.

8.2.1 Procedures

Methodologies employed in the laboratory are documented by the creation of an SOP. This document provides the analyst with the information necessary to perform the analysis. Every SOP is created in accordance with this QA document. It follows the intent of the method it is patterned after, but provides any additional information essential to the specific instrument instructions, specific quality concerns, etc.

8.2.1.1 If an SOP is not available for a specific analysis, the analyst will follow EPA, Standard Methods, NIOSH, or other regulatory methodology as required. No deviations of any kind are allowed.

8.2.1.2 Before a new method is accepted for routine use, adequate performance must be demonstrated. This includes an MDL study, IDOC, and all related QA/QC procedures as required by the method.

8.2.1.3 Appropriate management personnel evaluate the merits of all new methods and recommend approval or rejection based on the available data. This committee includes, at a minimum, the Laboratory Manager and Technical Director. If the method is approved, a Standard Operating Procedure is created and the procedure is implemented.

8.2.1.4 All analytical procedures must provide documentation so that the complete process used to produce data can be reconstructed.

8.2.1.5 All deviations from an approved analytical procedure are authorized and documented by the Technical Director.

8.2.2 All changes to an approved procedure require, at a minimum, an Interim Change Notice. A complete revision and re-issuance of the SOP may be required. SOPs are reviewed at least annually.

8.2.3 A list of all current SOPs including their review and revision status is maintained electronically on AES_server\L\Current SOP\SOP Masterlist. Current SOPs are maintained electronically on the AES Portal Server in the Technical Management folder. All controlled documents are in "Read Only" format and password protected. The Vice-President of Operations, QA Manager, Technical Director and their appointees are the only laboratory employees with edit access to these folders. In addition, a master list of controlled documents is maintained for documents other than SOPs. This includes various forms, software, references, etc. It is located at AES_server\L\Current SOP\ Documents_Master_List_Non-SOPs.

8.3. Instructions and Procedures

It is the policy of AES Inc. that all analyses and operations are performed using approved written procedures which are to be available to the personnel conducting the analysis /operation. The procedures assume one of two general formats. These formats are "Temporary Procedures" and "Standard Operating Procedures."

8.3.1 Temporary procedures are designed to accommodate the transition from a developing analytical service or method to an established procedure in the most efficient manner. They are less than formal procedures but are adequate to document the procedural treatment of samples. Effective dates and expiration dates are documented. Temporary Procedures, approved by a manager and the Technical Director, can be handwritten procedures and contain at a minimum the following information:

8.3.1.1 Health and safety requirements to perform procedure (if necessary).

8.3.1.2 Actual analytical method (step by step).

8.3.1.3 Materials list (if necessary).

8.3.1.4 Reagents (if necessary).

8.3.1.5 Calculations needed to perform procedure.

8.3.1.6 Reference sources from which procedure was developed.

8.3.2 Standard Operating Procedures (SOPs) are a formal treatment of an analytical or administrative procedure. Analytical SOPs shall be generated using nationally recognized procedures and incorporate AES, Inc., operations and instrumentation. The SOPs are revised as required by the appropriate Managers and are reviewed and authorized for continued use at least annually. Analytical SOPs contain the following information:

8.3.2.1 Title, issue date and revision number

8.3.2.2 Approval signatures

8.3.2.3 Approval signatures

8.3.2.4 Sample preparation, handling, storage and disposal

8.3.2.5 Definitions

8.3.2.6 Responsibilities

8.3.2.7 Hazards and safety requirements

8.3.2.8 Materials and equipment

8.3.2.9 Standardization and calibration requirements

8.3.2.10 QC sample frequency and performance criteria

8.3.2.11 Operating instructions

8.3.2.12 Example calculations and data sheets

8.3.2.13 References

8.3.3 Administrative Procedures contain the following sections
8.3.3.1 Contents Page

8.3.3.2 Purpose and scope paragraphs

8.3.3.3 Text

8.3.4 Emergency procedures are divided into three sections:
8.3.4.1 Symptoms

8.3.4.2 Immediate actions

8.3.4.3 Subsequent actions

8.4 Electronic Document Control
The laboratory SOPs are maintained electronically by the Technical Director through the electronic document control system. Hard copy signed originals of the procedures are Maintained by the Technical Director or appointee. Any staff member may request revision to the procedures.

8.5 Creating and Maintaining Standard Operating Procedures
"Standard Operating Procedures" describes the system for preparation, issue, implementation, and revision of formal Standard Operating Procedures for Analytical Environmental Services, Inc. Standard Operating Procedures are defined as written procedures for personnel to perform

analyses, technical operations, tests, processes, administrative operations and tasks, or inspection of samples submitted to Analytical Environmental Services, Inc.

8.5.1 Procedures are tracked, issued, revised, and filed.

8.6 Responsibilities

All technical and administrative staff is familiar with the requirements of this procedure and is responsible for its implementation. To ensure uniform and accurate procedures, the following personnel are assigned with the stated responsibilities:

8.6.1 SOP Author - The Author, when writing SOPs ensures the following:

8.6.1.1 The SOP meets applicable regulatory requirements.

8.6.1.2 The SOP includes the actual instruments and materials associated with AES, Inc.

8.6.1.3 The SOP follows the requirements of the published standard method(s) that the SOP is based on.

8.6.1.4 The SOP conforms to guidelines established in this document.

8.6.1.5 The SOP meets the applicable requirements of the laboratory's QA Manual.

8.6.1.6 That he responds to reviewer(s) comments in a timely manner.

8.6.2 Section Supervisor - The Section Leader is responsible for the following:

8.6.2.1 Review all new SOPs originating within their section.

8.6.2.2 Ensure the personnel in their department are aware of the SOP and understand their responsibility pertaining to the SOP.

8.6.3 Technical Director - The Technical Director is responsible for the following:

8.6.3.1 If a new SOP needs to be created, the Technical Director may assign the task of drafting SOPs to qualified individuals who possess the requisite experience and good communication/writing skills. The Technical Director may elect to write the SOP.

8.6.3.2 Ensures SOPs are in compliance with current regulations and established methods.

8.6.3.3 Reviews and approves all SOPs.

8.6.3.4 With the assistance of the QA Manager, maintains the SOP development, review, approval, and distribution system as stated in this procedure.

8.6.3.5 With the assistance of the QA Manager, maintains a protected archive of old SOP versions and current versions (controlled document system) for obsolete SOPs.

8.6.4 Laboratory Manager – the Laboratory is responsible for the following

8.6.4.1 Ensures that all sample analyses requested by the client have a current SOP. If a current SOP does not exist, the Laboratory Manager shall initiate a procedure for creation of an SOP.

8.6.5 QA Manager – the Quality Assurance Manager is responsible for the following:

8.6.5.1 With the assistance of the Technical Director, assists in SOP development, review, approval, and distribution system as stated in this procedure.

8.6.5.2 Ensures SOPs are in compliance with current regulations and established methods.

8.7 Definitions

8.7.1 Interim Change Notice (ICN) - A document accompanying any SOP or manual as a mandatory change, but is not included in the original text of the manual or SOP until the next revision.

8.7.2 Controlled Copy – A copy of an AES Document or SOP that is updated when revisions are issued. All controlled documents are electronic files.

8.7.3 Uncontrolled Copy – A printed copy that is labeled "uncontrolled" and is not updated when revisions are issued.

8.7.4 Technical SOPs – Any SOP that directly addresses the laboratory analysis procedure.

8.7.5 Non-Technical SOP – Any SOP that is used at AES but does not directly address the laboratory analysis procedures. Examples are QA SOPs, QC SOPs, Project Management SOPs, and Administrative SOPs.

8.8 New Procedure Initiation

8.8.1 Immediate Procedure Initiation

A Temporary SOP should be written when the laboratory receives projects which have requests for analytical procedures that do not have an SOP and the staff feels that the laboratory can perform the requested test procedure in-house.

8.8.2 Planned Procedure Initiation

The department manager/section supervisor, the Laboratory Manager, and the Technical Director determine the need for a new SOP.

8.8.3 As part of the New Procedure Request Form, the QA Manager and the Technical Director complete the following:

8.8.3.1 The Technical Director assigns the appropriate SOP number.

8.8.3.2 The Technical Director completes a Draft SOP or assigns an alternate author.

8.8.3.3 The draft SOP is forwarded to the affected laboratory personnel for their review (see Section 8.11). The draft includes all of the text, tables, and attachments formatted as outlined in this SOP.

8.8.3.4 After review by the affected personnel, the Technical Director finalizes the SOP. A hard copy of the SOP is produced for signature and placed into a folder in the QA Managers office. Controlled electronic copies are made available to laboratory staff in "Read Only" format on the AES Server and Portal Server.

8.9 Standard Operating Procedure Formatting

8.9.1 Title Page

8.9.1.1 Standard Operating Procedure Title Page Format. (Every procedure is preceded by the Procedure Title sheet. See Attachment 2).

8.9.1.2 Title – The procedure is given a concise, descriptive title. When appropriate, Operational Procedure titles should include the parameter(s) analyzed, sample type, method (if applicable), and analysis technique description (e.g., "Fluoride in Water by Ion Selective Electrode, based on EPA Method 353.3").

8.9.2 Comments – This section includes any reasons for revisions and additional comments as necessary.

8.9.3 Approval Signatures

8.9.4 Header

8.9.4.1 All SOPs have the following header on each page:

AES, Inc.		SOP No:	XX - #####
3785 Presidential Pkwy		Date Initiated:	MM / YY
		Date Revised:	MM / YY
Atlanta, GA. 30340		Revision No:	#
		Page No:	## of ##

8.9.4.2 The following header fonts are used:

	Font	Font Size
AES, Inc.	Times New Roman – Bold	12
Address	Times New Roman	8
SOP No, etc	Times New Roman	9

8.9.4.3 Each procedure is uniquely identified by a five digit number preceded by one of the following identifiers to indicate the type of procedure:

Identifier	SOP Type	# Assignments
QA	Quality Assurance	01000 – 01999
AD	Administrative	02000 – 02999
HS	Health & Safety	03000 – 03999
EM	Emergency	04000 – 04999
QC	Quality Control	05000 - 05999
PM	Project Management	06000 – 06999
GL	General Laboratory	08000 – 08999
SR	Sample Receiving	09000 – 09999
OA	Organic Analytical	11000 – 11999
IA	Inorganic/Metal Analytical	13000 – 13999
LP	Leaching Procedure	14000 – 14999
MB	Microbiology	15000 – 15999
ABS	Asbestos	01000 - 01999
WM	Waste Management	17000 - 17999

8.9.4.4 Revision – The first issue of a procedure is not assigned a revision number. It is assigned an "N/A" entry. As revisions are made to the procedure, the revision number is increased sequentially starting with Revision 1 (one).

8.9.4.5 Effective Date – The date when the procedure becomes effective. Use the following format: 12/97.

8.9.4.6 Revision Date – The date that the current revision became effective. Use the following format: 12/97.

8.9.4.7 Number of Pages – The correct form for this is, Page No.: x of y. Example the fifth page of a 24 page document would be formatted as: Page No.: 5 of 24.

8.10 Table of Contents

Section and sub-sections are listed in the Table of Contents using the font in the body of the SOP. See Attachment 5 for an example of an SOP. In addition, all Tables and Attachments are included in the Table of Contents.

8.10.1 Each Manual has a Table of Contents that includes the following information: SOP document number(s), name(s) of the SOP, date(s), revision number(s), and associated Method Number. When SOPs are revised, this list is edited to reflect the changes.

8.10.1.1 The Title of each SOP is Centered, All Capital letters, and in Boldface type on the Table of Contents page.

8.10.2 SOP Body – Technical Procedures.

8.10.2.1 All procedures are formatted using this section numbering system:

1.0 SECTION
1.1 Sub-Section
1.1.1 Sub-Sub-Section
1.1.1.1 Sub – Sub – Sub – Section
2.0 SECTION

8.10.2.2 To keep all the SOPs uniform, use Times New Roman, Font Size 12 for the body of the document

8.10.2.3 Each Section is underlined and all capital letters.

8.10.3 All Technical SOPs include the following sections in the same order:

TABLE 8-1 Technical SOPs

Section Number – Title	Purpose	Required Information
1.0 SCOPE AND APPLICATION	- Describes what the method does - Describes the matrices to which a method applies. -May also describe when the method is to be employed.	1. All matrices which may be analyzed using the method. 2. Analytes the method is capable of quantifying. 3. Quantitation range of analytes. 4. Reference to sample

2.0 SUMMARY OF METHOD	Provides a brief description of the procedure or method and the type of chemistry / instrumentation employed by the laboratory in performing the method.	
3.0 INTERFERENCES	List most common interferences which affect performance of the method. For preparative methods, include interferences which affect the sample analysis.	
4.0 SAMPLE COLLECTION, PRESERVATION , AND HOLDING TIMES	List preservation, storage, and holding time requirements for each matrix listed in Section 1.0.	1. Preservatives 2. Holding Times 3. Acceptable container types.
5.0 REAGENTS AND STANDARDS	List all reagents and standards.	1. Purity of reagents. 2. All concentrations of reagents and standards required. 3. Detailed preparation instructions for each reagent and standard to include initial concentration(s), aliquot volume(s) or weight(s), final volume, final concentration(s), and expiration dates. 4. Listing of the Vendor(s) used to purchase the reagent including the catalog number, vendor address, and telephone number.
6.0 APPARATUS AND MATERIALS	List all apparatus, materials, and equipment, inclusive of data collection and reduction systems.	List make and models or equivalents that might be used in the laboratory
7.0 PROCEDURE	1. This section defines the analytical procedure from start to finish. 2. Address QA/QC requirements when they are appropriate in the overall sequence of activities. 3. Addresses specific record keeping requirements (i.e. when and where to record specific information in run logs and other required laboratory documentation). 4. Includes the handling and disposal of waste when appropriate in the overall sequence of activities. 5. Calculations are included in the text where applicable following the example of SW-846 methods.	Includes at a minimum: 1. Instrument set-up and conditions. 2. Calculations of retention times if applicable. 3. Initial calibrations. 4. Continuing calibrations 5. Analysis sequence, including QC requirements. 6. Calculations – inclusive of conversions for solids. 7. Units required for reporting.

8.0 QUALITY ASSURANCE REQUIREMENTS	Defines additional QA requirements which must be met in addition to all criteria previously listed in the SOP.	Includes a minimum: 1. Blank requirements. 2. Laboratory Control Sample (LCS) requirements. 3. Matrix spike requirements 4. Matrix spikes duplicate or sample duplicate requirements. 5. Any method specific requirements (e.g. MSA for GFAA metals, surrogates for GC/MS procedures, tracers for alpha spectroscopy methods). 6. Corrective actions required when requirements are not met. 7. Frequency of QC samples
9.0 HEALTH AND SAFETY	Details specific health and safety requirements for the method and references any general health and safety requirements which may apply.	1. Protective clothing required. 2. Special hazards associated with chemicals or equipment used in the procedure. 3. Storage and / or disposal of all sample extracts and chemicals used.
10.0 DATA REPORTING	Defines the method for data reporting by the staff to clients.	Includes a minimum: 1. Reporting limits in LIMS. 2. Rounding of data.
11.0 FILE MAINTENANCE	Defines the procedures for data transfer and archiving of data for long term storage.	1. Frequency of data transfer from local computer to server. 2. Method used to transfer data to server. 3. Data storage requirements
12.0 INSTRUMENT MAINTENANCE	Defines the procedures for routine instrument maintenance and entry into logbooks.	
13.0 METHOD PERFORMANCE	Describes the acceptance criteria published in the method.	1. Spike, duplicate precision and accuracy.
14.0 POLLUTION MANAGEMENT	Describes the procedures required to dispose of hazardous wastes.	1. Waste disposal from received samples. 2. Waste disposal from laboratory generated wastes. 3. Required forms to be completed.
15.0 DEFINITIONS	Provides a definition for terms that are used in the SOP.	

16.0 REFERENCES	Provides the source(s) of the information from which the SOP was derived.	
17.0 VALIDATION DATA	Provides the location of information for method validation data.	

Note: The author may add any subsections that are necessary and do not fit in any of the above categories.

8.10.4 Copies of any forms or logbook pages used in conjunction with the SOP and are unique to the SOP are attached as Tables or Attachments and sequentially numbered and referenced in the body of the SOP.

8.11 SOP Body – Non – Technical (Administrative)

8.11.1 See Sections 8.10 and 8.11

8.11.2 The author may add any subsections that are necessary.

8.11.3 Copies of any forms or logbook pages used in conjunction with the SOP and are unique to the SOP are attached as Tables or Attachments and sequentially numbered and referenced in the body of the SOP.

8.11.4 SOP Body – Immediate SOP (See section 8.8.6 for the definition of "Immediate SOP").

8.11.5 Copy the Regulatory Method

8.11.6 Attach a procedure title sheet

8.11.7 Complete the following sections: 1.0 Health and Safety, 2.0 Reagents and Supplies, and 3.0 Step by Step Procedure. If these sections are included in the regulatory method, the following note can be included under each section: "See Regulatory Method attached section_____".

8.11.8 This is forwarded to the QA Manager who then initiates a new procedure, beginning with section 8.2.3.

8.12 Procedure Review And Revision

Procedures undergo periodic review and are updated whenever regulatory, programmatic requirements or internal process change.

8.13 Technical Review

8.13.1 A technical review of the draft SOP is performed by affected laboratory personnel and addresses the following items:

8.13.1.1 Does the SOP comply with the technical requirements of the regulatory agency (EPA, USACE, etc.) method?

8.13.1.2 Does the SOP state the step by step procedure of how AES completes the procedure?

8.13.1.3 Does the procedure formatting follow the procedures outlined in this section?

8.13.2 Comments are written directly on the Draft SOP or on another sheet of paper if needed.

8.13.3 The reviewer(s) discuss comments with the Technical Director and arrive at a finalized document.

8.13.4 The Technical Director makes the necessary changes electronically. The changes include any Interim Change Notices (ICNs) that have been generated for the SOP and are incorporated as stated in the ICN. The electronic copy is stored in the server in the appropriate year labeled folder.

8.13.5 The reviewed SOP is printed and all approval signatures are obtained on the original hard copy.

8.13.6 The approved SOP is electronically placed in the "Current Revisions" folder by the Technical Director. All employees have access to these files in a "read only" format.

8.13.7 SOP Acknowledgement forms (Attachment 1) are distributed to all area supervisors to distribute to all employees who will be using the procedure.

8.13.8 All employees using the new procedure sign SOP Acknowledgement forms and return them to the Supervisor who forwards them to the Technical Director or designee for final approval and scanning.

8.14 Procedure Changes

8.14.1 Analysts, supervisors, or management have the ability to request changes to procedures as part of the continuing procedure maintenance using the "Interim Change Notice" (ICN) form (See Attachment 4).

8.14.2 To complete an ICN, make the required changes to a copy of each affected procedure page. Revise and edit these copies using appropriate standard editor's marks and symbols.

8.14.3 The employee requesting the change ensures the department manager signs the ICN and forwards the ICN to the Technical Director.

8.14.4 The Technical Director signs the ICN, supplies a copy to each applicable department supervisor, ensures that a copy is placed in the controlled SOP folders (see section 8.2), and files it with the controlled QA SOP files.

8.15 Standard Operating Procedures Electronic Document Control Process

8.15.1 All controlled documents are electronic files which are password protected and managed by the Technical Director or designee.

8.15.2 All laboratory personnel have access to a controlled, electronic copy of the SOPs applicable to their job description.

8.15.3 Only uncontrolled documents are issued to clients.

8.15.4 The electronic document control files are arranged such that laboratory personnel have access to only current revisions of controlled documents. All archived revisions, draft procedures, etc. are accessible only to authorized QA or Technical Direction personnel via password access.

8.16 Uncontrolled copies of Standard Operating Procedures are printed, working copies of the documents, and in that regard, are not monitored or tracked.

8.17 Procedure Archive

The Technical Director is responsible for archiving any procedures that are no longer used at AES.

8.17.1 The Technical Director electronically moves the procedure to the designated archive directory.

8.17.2 The Technical Director removes the folder from the "active" files and places it in the archived files.

8.18 Temporary Change

Temporary changes to an SOP may be required for the following reasons: a sample matrix does not permit the SOP steps to be followed as written, or if a client desires a change to an SOP that is currently in use at AES.

8.18.1 The Temporary Change Notice is completed and approved prior to the use of a revised procedure. See Attachment 4.

Attachment 1

QUALITY ASSURANCE MANUAL STANDARD OPERATING PROCEDURE ACKNOWLEDGEMENT

Name (Printed): _____

SOP Title: **Quality Assurance Manual**

SOP Number: **QA-01000** **Rev. No.**_____

The laboratory analyst signature on this approved SOP signifies the following: The analyst has read the SOP in its entirety and has read the analytical methods referenced in the SOP.

The analyst understands that the SOP is to be followed explicitly. Any deviation from the SOP must be noted in writing. Furthermore, the deviation from the SOP must be approved in writing by the laboratory supervisor and the QA staff prior to the analyst's adoption of the deviation from the SOP.

The controlled electronic copy of this SOP is located on the portal server at: Documents: Quality Assurance: QA Manuals: QA Manual: **2011_QA_Manual_Rev_15**.pdf. If a hard copy is desired, you may request one from the Supervisor.

Do not make a copy or print out the QA Manual yourself. Printed copies are uncontrolled documents.

Print Name: _____ Date: _____

Analyst's Signature: _____ Date: _____

Department Manager Signature: _____ Date: _____

Technical Director's Signature: _____ Date: _____

Attachment 2

APPROVAL OF ATTACHED DOCUMENT FOR IMPLEMENTATION

NOTE: THIS IS A CONTROLLED ELECTRONIC DOCUMENT

PRINTED COPIES OF THIS DOCUMENT ARE UNCONTROLLED

ORIGINAL SIGNED DOCUMENT RESIDES IN AES QA OFFICE

DOCUMENT TITLE:

DOCUMENT CONTROL NUMBER:
DOCUMENT DISTRIBUTION NUMBER:

ELECTRONIC DOCUMENT LOCATION: AES Portal Server
htpp\\aes/home/Document Library/Documents/Technical Management/Standard Operating Procedures

The attached Document has been reviewed by the individuals listed below. By signature, each of these individuals acknowledges that the document is ready for distribution, in a controlled manner, to all responsible parties for use and/or reference.

By definition, a "Controlled Copy" of a document cannot be changed without review and approval by designated members of management. At no time may a "controlled copy" be written on or otherwise defaced with notes or other unauthorized additions.

If an uncontrolled copy of this document is desired, please see the Quality Assurance or Laboratory Manager. They will issue you an uncontrolled copy. DO NOT MAKE THE COPY YOURSELF.

By signature below the following employees of Analytical Environmental Services, Inc. have approved this document for distribution.

Technical Director: Date:

Laboratory Manager: Date:

Quality Assurance Manager: Date:

Department Supervisor: Date:

Attachment 3

Example SOP

STANDARD OPERATING PROCEDURES FOR CONDUCTIVITY
BY EPA METHODS 120.1

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to drinking, surface, and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition).

2.0 SUMMARY OF METHOD

2.1 The specific conductance of a sample is measured by use of a self-contained conductivity meter, Wheatstone bridge-type or equivalent.

2.2 Samples are preferable analyzed at 25°C. If not, temperature corrections are made and results reported at 25°C.

3.0 INTERFERENCES

3.1 Temperature variations and their corrections are the only interference that is of concern. Make sure sample temperatures are as close as possible to 25°C.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

4.1 Analyses can be performed either in the field or laboratory.

4.2 If analysis is not completed within 24 hours of sample collection, samples should be filtered through a 0.4 micron filter and stored at 4°C. Filter and apparatus must be washed with high quality DI water and pre-rinsed with sample before use.

5.0 REAGENTS

5.1 Standard potassium chloride solutions, 0.01 M: Dissolve 0.7456 gram and pre-dried (2 hour at 105°C) KCl in DI water and dilute to 1 liter at 25°C. And alternative is a commercially available standard for conductivity.

6.0 APPARATUS AND MATERIALS

6.1 Conductivity bridge, range 1 to 1000 µmhos per centimeter.

6.2 Conductivity cell, cell constant 1.0 or micro dipping typing cell with 1.0 constant.

6.3 Thermometer with no greater than 1 °C increments and calibrated against an NBS thermometer.

7.0 PROCEDURE

7.1 The analyst should use the standard potassium chloride solution and the table below to check the accuracy of the cell constant and conductivity bridge.

Conductivity of 0.01 M KCl

°C	**Micromhos / cm**
21	1305
22	1332
23	1359
24	1386
25	1413
26	1441
27	1468
28	1496

7.2 Follow the directions of the manufacturer for the operation of the instrument.

7.3 Allow samples to come to room temperature (23 to 27°C), if possible.

7.4 Determine the temperature of samples within 0.5°C. If the temperature of the samples is not 25 °C, make temperature correction in accordance with the instruction in Section 7.5 to convert reading to 25°C.

7.5 The following temperature corrections are based on the standard KCl solution.

7.5.1 If the temperature of the sample is below 25°C, add 2% of the reading per degree.

7.5.2 If the temperature of the sample is above 25°C, subtract 2% of the reading per degree.

7.6 Report results as Specific Conductance, µmhos / cm at 25°C.

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 A blank should be analyzed with every batch (20 samples or less analyzed within a twenty-four period).

8.2 Each sample should be analyzed in duplicate if sample volume allows. This will verify that there is no stratification of dissolved salts with in the sample container.

8.3 The KCl standard should be analyzed after the blank but before any samples. It should be with in 5% of the expected conductivity. If it is not check the temperature and make sure that it is corrected. If it is still out of the acceptable range, see the instructions for adjusting the instrument to read the correct amount. Usually a conductivity meter has a calibration screw in the bottom that can be adjusted to read correctly. Care must be taken however not to rely too heavily on this. Other problems, such as a bad standard, may be the cause of a low or high reading. If the instrument needs to be calibrated often, it should be serviced by an authorized repair service.

9.0 HEALTH & SAFETY REQUIREMENTS

9.1 Health and Safety: Safety glasses and latex type gloves must be worn at all times when dealing with any chemicals, samples, or reagents. A lab coat is also highly recommended. Close-toed shoes and clothing that covers the legs (no shorts or dresses) must be worn at all times an analyst is working in the laboratory.

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a health hazard and exposure should be as low as reasonably possible. All health and safety concerns for these and any other chemicals are listed in the Material Safety Data Sheets (MSDS) provided by the supplier or manufacturer of these chemicals. A copy of any MSDS is available for review at any time.

9.2 Proper disposal of all wastes is essential. Containers are provided for all waste according to the type. Please follow the waste disposal Standard Operating Procedure for how each type of chemical is disposed of.

10.0 DATA REPORTING

10.1 Report results to two significant digits. For example, if the result is less than 1, report the result as “0.08”.

10.2 When taking duplicate measurements, average the two readings

11.0 FILE MAINTENANCE

11.1 Data from this test is stored in logbooks. When the logbooks are complete, they are stored in banker boxes for 5 years.

11.2 Data is entered into LIMS by the analyst performing the work

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks. Instrument logbooks must be completed each time that any maintenance is performed upon the instrument.
Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: Conductivity Meter
Manufacturers name. Example: Orion Research
Serial Number. Example: 13226589A
Date Received. Example: 11/01/04
Date Placed into Service. Example: 11/05/04

12.2 Routine Maintenance: Typical routine maintenance consists of keeping the system clean.

12.3 Non-routine maintenance: Typical non-routine maintenance consists of replacement of circuit boards and or replacement of photomultiplier tubes on the spectrophotomet

13.0 METHOD PERFORMANCE

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The reporting limit RL is defined as the concentration of a substance that is above the level of uncertainty. The concentrations listed in the table in Section VIII were obtained using reagent water. Similar results can be achieved using representative wastewaters. The MDL actually achieved is a given analysis will vary depending on instrument sensitivity and matrix effects.

13.2 Forty one analysts in 17 laboratories analyzed six synthetic water samples containing increments of inorganic salts, with the following results:

Increment as Specific Conductance	Precision as Standard Deviation	Accuracy as Bias %	Accuracy as µmhos/cm
100	7.55	-2.02	-2.0
106	8.14	-0.76	-0.8
808	66.1	-3.63	-29.3
848	79.6	-4.54	-38.5
1640	106	-5.36	-87.9
1710	119	-5.08	-86.9

13.3 In a single laboratory using surface water samples with an average conductivity of 536 µmhos / cm at 25°C, the standard deviation was 6.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.1.1. Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.
14.1.2. Wastes with other pH levels may be directly discharged into the sinks.
14.1.3. SOP WM-17001 Waste Disposal and SOP SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.
Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade –A dry chemical that has been dried at 250°C for 4 hours cooled and stored in a desiccator.
15.2 LCS - Laboratory Control Check Standard. A known amount of sought for analyte is added to distilled water or clean soil and the concentration is measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.
15.3 DI water- Deionized water
15.4 RSD – Relative Standard Deviation
15.5 RF – Response factor. Determined as the concentration of a sample divided by the chromatographic area of the peak produced by the sample.
15.6 MS- Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.
15.7 MSD- Matrix Spike Duplicate.
15.8 CCV - Continuing calibration verification standard. Must be varied thought the daily batch, that is the concentration must be low, middle, and sometimes at the upper end of the calibration curve. CCV must be analyzed, at a minimum, at the beginning and end of the analytical batch. See specific test methods for additional CCV frequency requirements.
15.9 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot that the calibration standard.
15.10 LCSD - Laboratory Check Standard Duplicate
15.11 <CR> - Carriage return key

16.0 REFERENCES

16.1 USEPA Methods Manual, July 1982, Method 120.1, “Conductance”.
16.2 Code of Federal Regulations, 40CFR part 136, Appendix B.

Attachment 4

Temporary SOP or **Interim (Temporary or Permanent) Change Notice** (circle one as appropriate)

Date:
Employee Requesting Change:
SOP Number:
Reference Method Number:
SOP Title:
Permanent Change Requested:

Technical Director: Date:

Laboratory Manager: Date:

Quality Assurance Manager: Date:

Department Supervisor: Date:

9.0 CALIBRATION PROCEDURES AND FREQUENCY

9.1 Identification and Control of Materials, Parts and Components

9.1.1 General

Materials, components or items that are used directly in the production of samples or data that, if not controlled, could jeopardize data quality must be identified.

9.1.1.1 AIHA Traceability of Measurement Policy

Under Analytical Environmental Services' AIHA-LAP, LLC accreditation, the laboratory shall demonstrate, when possible, that calibrations of critical equipment and hence the measurement results generated by that equipment, relevant to their scope of accreditation, are traceable to the SI (International System of Units) through an unbroken chain of calibrations.

External Calibration services shall, whenever possible, be obtained from providers accredited to ISO/IEC 17025. Calibration certificates shall be endorsed by a recognized accreditation body symbol. Certificates shall indicate traceability to the SI or reference standard and include the measurement result and if available the associated uncertainty of measurement.

Where traceability to the SI is not technically possible or reasonable, the laboratory shall use certified reference materials provided by a competent supplier.

Reference materials shall have a certificate of analysis that documents traceability to a primary standard or certified reference material and associated uncertainty, when possible. When applicable, the certificate must document the specific NIST SRM® or NMI (National Metrology Institute) certified reference material used for traceability.

Calibrations performed in-house shall be documented in a manner that demonstrates traceability via unbroken chain of calibrations regarding the reference standard/material used, allowing for an overall uncertainty to be estimated for the in-house calibration.

Calibration shall be repeated at appropriate intervals, the length of which can be dependant on the uncertainty required, the frequency of use and verification, the manner of use, stability of equipment, and risk of failure considerations. Table 9-1 provides minimum frequencies required.

Periodic verifications shall be performed to demonstrate the continued validity of the calibration at specific intervals between calibrations. The frequency of verifications can be dependent on the uncertainty required, the frequency of use, the manner of use, stability of the equipment, and risk of failure considerations. Table 9-1 provides minimum frequencies required.

The laboratory has procedures describing their external and internal calibration and verification activities and frequencies, and the actions to follow if equipment is found to be out of acceptable specification.

Laboratory staff performing in-house calibration and verifications shall have received documented training.

Table 9-1

AIHA Minimum Calibration / Verification Frequency Requirements

Reference Standard / Equipment	Calibration Frequency	Verification Frequency
Balances	Initial and Annually	Each day of use
Mechanical Pipettors	Initial and when verification fails	Quarterly
Reference Thermometers	Initial and every 5 years	Not applicable
Digital Thermometers	Initial and when verification fails	Quarterly
Alcohol-Hg-Spirit Thermometers	Initial and when verification fails	Semi-annual
Reference Masses	Initial and every 5 years	Not applicable
Stage Micrometer	Initial, if damaged, and every 7 yrs	Not applicable

9.1.2 Control of Materials, Parts and Components

When appropriate, identification of each item is maintained by part number, serial number, or other appropriate methods, either directly on the item, or by labels or records traceable to the item. The system is designed to prevent the use of incorrect or defective items and to maintain identify and control inventory. When appropriate, the system controls items by batch number rather than by individual item. Instrumentation not currently in use or equipment undergoing repair is labeled as "Out of Service."

9.1.3 Handling, Storage and shipping

9.1.3.1 General

This criterion establishes requirements for the proper handling, storage, preservation and shipping of materials, supplies and equipment.

9.1.3.2 Procedures and Responsibilities

All items affecting quality are handled and stored in such a manner as to prevent deterioration and damage to the quality. Items that require shipping are packed to prevent damage. Managers and supervisors are responsible for items under their control.

9.1.4 Procurement Document Control

9.1.4.1 General

9.1.4.1.1 Vendors of analytical material supplied to AES are regarded as a resource to, and an extension of the laboratory organization. The standards for quality identified in this document shall be applicable to vendors.

9.1.4.1.2 The purpose of the procurement control criterion is to ensure the quality and traceability of procured quality related items (equipment, materials, or services), whose specification could affect the quality of the services of AES. This includes such quality related items as the calibration of instruments by outside laboratories (when appropriate), purchase of

standards, subcontracted services and materials requiring testing before use, as determined by the QA Manager.

9.1.4.2 Procedures and Responsibilities

9.1.4.2.1 It is the responsibility of the purchasing agent to provide assurance, when required, that all applicable regulatory requirements, industry codes and standards appear in the purchase documentation for affected services and products.

9.1.4.2.2 The Purchasing Department retains purchase orders for control purposes.

9.1.4.2.3 Purchased items which do not meet the minimum standards set forth by the purchasing agent are processed according to procedures set forth in Section 13, "Corrective Actions."

9.1.4.2.4 The appropriate Manager/Supervisor and QA Manager review purchase orders, which may affect quality-related services or products.

9.1.4.2.5 Purchase orders for standard catalog items except those described in herein, are exempt from QA review.

9.1.5 Non-conformance

The purpose of this criterion is to establish a system to control materials, parts, or components that do not conform to established requirements in order to prevent their inadvertent use. When significant deficiencies in analytical procedures, materials or components has or may lead to the release of incorrect analytical results to the customer, a Corrective Action Report (CAR) is issued.

9.1.5.1 Procedures and Responsibilities

The Laboratory Manager and the purchaser perform the inspection of the newly received material and equipment. Nonconforming items that fail incoming receipt inspection are identified and segregated until disposition is determined and documented by the Non-Conformance Report. Copies of these documents are maintained by the Purchasing Department or the QA Department, as applicable.

9.2 Instrumentation List

Appendix III, "Equipment List," summarizes the laboratory equipment used to analyze for the parameters specified in Section 5.0 and indicates (to the extent the information is available) each ID number, instrument number, type of equipment, manufacturer, model, serial number, condition when purchased, in-service date and present location. It also lists in-house standards of traceability such as certified analytical balance weights and calibration thermometers.

9.3 Measurement Traceability and Calibration

Procedures for achieving Traceability of Measurements

9.3.1 General

The purpose of this criterion is to assure that instruments and other measuring and testing devices used in activities affecting program quality are properly controlled, calibrated and adjusted at specified periods to maintain accuracy within design and/or procedure limits. Implementation procedures consist of the following as applicable:

9.3.1.1 Identification and control of the item

9.3.1.2 Creation of calibration schedules and procedures based on instrument type, planned use, and design limits and program requirements.

9.3.1.3 Development of any necessary calibration sources for use in confirming successful equipment operation.

9.3.1.4 Maintenance of equipment history records to indicate past and status, and to provide reproducibility and traceability of results.

9.3.2 Responsibility

Under the direction of the manager, the supervisors are responsible for the quality of measuring and test equipment under his/her control and for the maintenance of records of calibrations and checks.

9.3.3 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests shall be calibrated and/or verified before being put into service and on a continuing basis. The laboratory has an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers and control standards.

9.3.4 Traceability of Calibration

9.3.4.1 The overall program of calibration and/or verification and validation of equipment ensures that, wherever applicable, measurements made by the laboratory are traceable to national standards of measurement.

9.3.4.2 Calibration certificates indicate the traceability to national standards of measurement and provide the measurement results and associated uncertainty of measurement. Certificates are maintained in the Quality Assurance office files.

9.3.4.3 The laboratory maintains calibration certificates that provide traceability to each standard chemical used within the laboratory. As these standards are purchased, the certificates that accompany the standards are stored in logbooks. Information included in the logbooks includes labels provided by the manufacturer, expiration date, lot number, etc. This information is stored separately for standards purchased by each department and can be accessed by all personnel within the department.

9.3.4.4 Where the traceability of national standards of measurement does not apply, AES shall provide satisfactory evidence of correlation of results by participation in a program of interlaboratory comparisons, proficiency testing studies or independent analysis.

9.3.5 Reference Standards

9.3.5.1 Such reference standards as Class 1 weights or traceable thermometers are used for calibration only and no other purpose, unless it can be demonstrated that their performance as reference standards will not be invalidated. AES, Inc., maintains certified Class 1 weights, thermometers which have been calibrated by outside

agencies that can provide traceability to national standards of measurement. The stage micrometer will be calibrated by a NIST traceable reference.

9.3.5.2 The calibration and verification of reference standards occurs every five years for Class 1 weights and thermometers and every seven years for stage micrometers.

9.3.5.3 Where relevant, reference standards and measuring and testing equipment shall be subjected to in-service checks between calibrations and verifications. These reference materials shall, where possible, be traceable to national or international standard reference materials. Table 9-2 lists the major standards (traceable to NIST) which are used in the laboratory and their sources.

Table 9-2

Chemical Standard	Manufacturer/Vendor
PAH Mix	VWR-Restek, Supelco
Toxaphene	ERA, Accustandard, Absolute Stds
Chlordane	ERA, Accustandard, Absolute Stds
Hexavalent Chromium	ERA, Accustandard, Absolute Stds
LAS (MBAS)	ERA, Accustandard, Absolute Stds
Calcium Carbonate	ERA, Accustandard, Absolute Stds
TSS	ERA
O&G	ERA, Accustandard, Absolute Stds
Aroclor Mix (PCB)	ERA, Accustandard, Absolute Stds
8260B Matrix Spike	VWR-EM Science
EPA 625 Kit	Restek
Sodium Nitroferricyanide	VWR-Mallinckrodt
Sodium salicylate	VWR-J.T. Baker
Phosphate (P) Standard	Labchem, Inc.; Ricca
Mercuric Oxide	VWR-J.T. Baker
Multi-element Metals Std	SCP
Antimony Standard	SCP
Furan	Aldrich Chemical
Herbicides Mix	ERA, Accustandard, Absolute Stds
DRO/GRO	ERA, Accustandard, Absolute Stds
EDB, DBCP	ERA, Accustandard, Absolute Stds
turbidity	ERA, Accustandard, Absolute Stds
8270C Mix	ERA, Accustandard, Absolute Stds
Semi-Vols Mix	RTC
1,2-diphenylhydrazine	Restek

9.3.6 Calibration

Calibration requirements are divided into two parts: 1) requirements for analytical support equipment, and 2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

9.3.6.1 Instrument Calibration

9.3.6.1.1 Analytical instruments are calibrated in accordance with the proper analytical procedure to determine the analyte(s) of interest. After initial calibration of an instrument, a continuing calibration standard is analyzed at specific intervals. The calibration standards must meet the specified QC requirements associated with each test method (see Section 5).

9.3.7 Control of Measuring and Test Equipment

9.3.7.1 General

The purpose of this criterion is to assure that instruments and other measuring and testing devices used in activities affecting program quality are properly controlled, calibrated and adjusted at specified periods to maintain accuracy within design and/or procedure limits. Implementation procedures consist of the following as applicable:

9.3.7.1.1 Identification and control of the item.

9.3.7.1.2 Creation of calibration schedules and procedures based on instrument type, planned use, design limits and program requirements.

9.3.7.1.3 Development of any necessary calibration sources for use in confirming successful equipment operation.

9.3.7.1.4 Maintenance of equipment history records to indicate past and current status, and to provide reproducibility and traceability of results.

9.3.7.2 Equipment calibration specific to microbiological analysis.

The laboratory, under the direction of the section leader, determines and documents temperature stability, uniformity of temperature distribution, and time required to achieve equilibrium conditions in incubators and water baths. This procedure is performed during the following two conditions.

9.3.7.2.1 When new equipment is purchased

9.3.7.2.2 On an annual basis for existing equipment

9.3.7.3 Volumetric accuracy checks for disposable pipettes used in microbiological analysis. The laboratory, under the direction of the section leader, determines and documents volumetric accuracy of disposable pipettes. This is accomplished by checking 5 pipettes per case lot.

9.3.7.4 Mechanical timer accuracy checks.

The laboratory, under the direction of the section leader, determines and documents the accuracy of mechanical timers. This is done by the following method and frequency.

9.3.7.4.1 Accuracy check is performed on an annual basis. The accuracy check is documented in the logbook.

9.3.7.4.2 Accuracy is compared against an electronic timing device such as a stopwatch.

9.3.7.5 General Responsibility

Under the direction of the manager, the supervisors are responsible for the quality of measuring and test equipment under his/her control and for the maintenance of records of calibrations and checks.

9.3.8 Reference Measurement Standard List

Reference measurement standards must originate, wherever possible, from sources traceable to NIST. Table 9-3 describes the major standards used in the laboratory and their sources:

Table 9-3
Reference Measurement Standard List

Chemical Standard	Manufacturer/Vendor
PAH Mix	VWR-Restek, Supelco
Toxaphene	ERA, Accustandard, Absolute Stds
Chlordane	ERA, Accustandard, Absolute Stds
Hexavalent Chromium	ERA, Accustandard, Absolute Stds
LAS (MBAS)	ERA, Accustandard, Absolute Stds
Calcium Carbonate	ERA, Accustandard, Absolute Stds
TSS	ERA
O&G	ERA, Accustandard, Absolute Stds
Aroclor Mix (PCB)	ERA, Accustandard, Absolute Stds
8260B Matrix Spike	VWR-EM Science
EPA 625 Kit	Restek
Sodium Nitroferricyanide	VWR-Mallinckrodt
Sodium salicylate	VWR-J.T. Baker
Phosphate (P) Standard	Labchem, Inc.; Ricca
Mercuric Oxide	VWR-J.T. Baker
Multi-element Metals Std	SCP
Antimony Standard	SCP
Furan	Aldrich Chemical
Herbicides Mix	ERA, Accustandard, Absolute Stds
DRO/GRO	ERA, Accustandard, Absolute Stds
EDB, DBCP	ERA, Accustandard, Absolute Stds
turbidity	ERA, Accustandard, Absolute Stds
8270C Mix	ERA, Accustandard, Absolute Stds
Semi-Vols Mix	RTC
1,2-diphenylhydrazine	Restek

10.0 PREVENTIVE MAINTENANCE

10.1 Instrument Maintenance

All instrument maintenance is recorded in an instrument specific logbook. Entries are dated and initialed by the analyst making the entry.

10.1.1 Routine

All analytical instruments have a routine schedule of maintenance specified by the manufacturer. Routine maintenance is designed to keep the instrument in good operating

condition with as little "down-time" as possible. All Analysts should be proficient in maintaining the instruments for which they are responsible.

10.1.2 Non-Routine

Any maintenance which must be performed in order for sample analysis to proceed, but is not part of the systematic maintenance schedule, is considered non-routine. Non-routine maintenance must be reported to the Section Supervisor immediately so that is its impact on production can be determined. If the ability to analyze samples is adversely affected, the Section Supervisor notifies the Client Services Manager so that alternative action can be coordinated with the client.
(Note: See Appendix II for a complete instrument maintenance summary.)

10.2 Preventive Maintenance

10.2.1 Maintenance Schedule

AES is equipped with up-to-date computerized instrumentation. In order to gain maximum performance and minimize downtime, regular inspection, maintenance, cleaning, and servicing of all laboratory and field equipment is performed according to the manufacturers' recommendations.

10.2.2 A maintenance log is kept for each piece of laboratory and field instrumentation, detailing all maintenance performed on the instrument.

10.2.1.1 Routine repairs and maintenance are performed and documented by the analyst responsible for the particular instrument.

10.2.1.2 A log of non-routine maintenance is kept in the instrument repair logbook. As part of this information, the analyst or repair technician signs and dates the logbook.

10.2.1.3 Routine maintenance procedures for laboratory instrumentation are given in Appendix II. The service intervals listed in Appendix II are as follows: D = daily; W = weekly; M = monthly; Q = quarterly; SA = semi-annually; and AN = as needed. (A list of all laboratory equipment may be found in Appendix III.)

10.2.3 An extensive approved spare parts inventory is maintained for routine repairs at the facilities, consisting of GC detectors, AA lamps, fuses, printer heads, flow cells, tubing, certain circuit boards and other common instrumentation components.

10.3 Glassware Cleaning

Laboratory glassware cleaning procedures and guidelines are described in Table 10-1. Glassware must be Class A wherever possible.

TABLE 10-1
LABORATORY GLASSWARE CLEANING PROCEDURES

Analysis/Parameter	Cleaning Procedure (In Specified Order)
Extractable Organics (including	Solvents: 13, 1, 2, 3, 4, 5 or 7, (6 or 8 optional), 15, 17
Pesticides and Herbicides)	Or, Muffle Furnace: 13, 1, 2, 3, 4, 14, 15, 17
	Or, Oxidizer: 13, 1, 2, 3, 16, 3, 4, 15, 17
Purgeable Organics	1, 2, 3, 4, (7 optional), 11
	Or, 1, 2, 3, 4, (5 optional), (8 optional), 11
Trace Metals	1, 2, 3, 4, 10, 4
Nutrients, Other Wet Chemistry	1, 2, 3, 4, 9, 4
TKN	1, 2, 3, 4, 18, 4
Minerals, Demands, CN and Phenols	1, 2, 3, 4
Microbiology	1, 2, 3, 4
Residues	1, 2, 3, 4, 12

Key to Laboratory glassware cleaning procedures:

1 Remove all labels with sponge or acetone
2 Wash with hot tap water, scrub stopcocks and other small pieces with brush and inside glassware using a laboratory-grade detergent
Organics – Liquinox, Alconox or equivalent
Inorganic Anions – Liquinox or equivalent
Inorganic Cations – Liquinox, Acationox, Micro or equivalent
3 Rinse thoroughly with hot tap water
4 Rinse thoroughly with deionized water
5 Rinse thoroughly with pesticide-grade acetone
6 Rinse thoroughly with pesticide-grade methylene chloride
7 Rinse thoroughly with pesticide-grade methanol
8 Rinse thoroughly with pesticide-grade hexane
9 Rinse or soak with 1:1 HCl
10 Rinse or soak with 10% HNO_3
11 Bake at 105^{o}C for 3-4 hours (Note: Class A volumetric glassware must NOT be baked!)
12 Bake crucibles at 105 ^{o}C or 180 ^{o}C for 1 hour (prior to use, as per method)
13 After use, rinse with same solvent used
14 Drain, then heat in muffle furnace for 15-30 minutes
15 Store inverted or capped with suitable material or container stopper
16 Soak in oxidizing agent: chromic acid or equivalent
17 Rinse with solvent used in analysis as the last step prior to use
18 Rinse or soak with 1:1 H_2SO_4

11.0 QUALITY CONTROL CHECKS AND ROUTINES TO ASSESS PRECISION, ACCURACY AND METHOD DETECTION LIMITS

11.1 Control of Special Processes

11.1.1 In certain processes, the existence of a required level of quality cannot be assured by the examination of the end result alone. Such special processes that relate to the conduct of programs include performance of detailed chemical procedures, interpretation of raw data and the use of advanced data analysis techniques.

11.1.2 For such processes, quality assurance is obtained through the development of thorough analytical and operational procedures. QA is also obtained by personnel screening and documented training to insure the necessary level of personnel qualifications and capabilities and by the use of QC samples. This section describes how personnel are qualified in accordance with specified requirements.

11.2 Quality Control in the Laboratory

11.2.1 Various types of quality control samples are used at AES, Inc., in each of the following areas:

- Bulk Asbestos
- Air Asbestos
- Gas Chromatography/Mass Spectrometry
- Gas Chromatography
- Inorganic Analysis
- Wet Chemistry
- Microbiology
- Sample preparation

11.2.2 Some of the activities used to qualify the procedures (and data) are described:

11.2.2.1 Standards

The Section Supervisor (or designee) is responsible for the preparation and documentation of stock standards and working standards. Standard reference materials are obtained from suppliers and have Certificates of Analysis to certify the analyte concentrations. When available, traceable reference materials are to be used. As a minimum, information on reference materials includes manufacturer, lot or batch number, date of receipt, expiration date, and any other accompanying preparation or assay information. The most recent release of the NIST standards library shall be used for mass spectral interpretation.

11.2.2.2 Calibration and Performance Check of Instruments

Different types of reference material are used to calibrate the various analytical instruments in the laboratory areas.

For most of the analytical instruments used in the laboratory, calibration and performance checks are conducted at the beginning of an analytical run, periodically throughout the run and at the end of the run, (e.g., Atomic Absorption Spectrophotometers), while others are calibrated once then checked daily. The performance checks must be from an outside source, such as an alternate manufacturer, or may be from the same manufacturer as long as it originates from a different lot or batch. Calibration is also performed when the analytical method is

initially set-up, when an instrument has been through major maintenance, or the instrument fails its QC check.

11.2.2.3 Inter-Laboratory Analysis of QC Samples
Client and method requirements determine the frequency and type of spikes, blanks, splits, method standards, surrogate standard, internal standard and external source analyses. These normally account for 10 – 20% of the data points generated by the laboratory.

11.2.2.4 Inter-Laboratory Analysis
AES, Inc. participates in various accreditation and certification programs that require the analysis of either agency-supplied performance samples or proficiency test study samples purchased from a TNI or AIHA approved PT provider as required. Results of these performance results are reported and maintained in QA files. Results which are evaluated as "Not Acceptable" are documented and reviewed by the Quality Assurance department and resolved through discussion with analysts and their supervisors, examination of all raw data, re-assessment of sample preparation directions and techniques, and a review of data and calculations.

11.2.2.5 Computational Checks
Substantial portions of hand calculations are checked by a second individual, in most cases the section supervisor. The person performing the crosscheck must be qualified in the relevant technical discipline. For computations performed automatically using verified software, and which contain a hard copy of the entered computation, only the entries are checked.

11.2.2.6 Review and Analysis of Data
The review and analysis of data for analytical measurements are performed on a timely basis using Quality Control checklists. The data is checked for reasonableness and consistency by the section Supervisor and/or the manager.

11.2.2.7 Detection Limit Studies
The detection limit of an analyte is defined as the smallest amount of an analyte that can be detected (for instrumentation, above the background noise) within a stated confidence limit. There are several types of detection limits that may be applicable to a given method. The Instrument Detection Limit (IDL) is the amount of analyte needed to produce an adequate response above an instrument's baseline noise. The IDL may be use to estimate a Method Detection Limit (MDL). The Practical Quantitation Limit (PQL), also called the Reporting Limit (RL) is defined as the lowest level of quantitation achievable during routine laboratory operations. Some agencies define the PQL more rigidly as 3.33 times the MDL. However, the PQL is highly matrix dependent.

11.2.2.8 Recovery of Known Additions (Spikes)
Recoveries of known additions of analytes are used to determine the effect of the sample matrix on the given analytical procedure. The Laboratory Control Sample (LCS) and sample Matrix Spike/Spike Duplicate (MS/MSD) are used to monitor

and control the analytical process. The recovery of spiked analytes in the sample matrix gives a definitive measure of the sample preparation processes.

11.2.2.8.1 LCS data is used to monitor the laboratory's performance in respect to sample preparation and equipment operation. It is prepared in an analyte free matrix similar to the sample, i.e. water or soil. Recovery limits for the LCS are established by the laboratory through control charting of each analyte.

11.2.2.8.2 A matrix spike/matrix spike duplicate pair is analyzed to determine the effect of the sample matrix on extraction efficiency and analyte recovery. One MS/MSD pair should be prepared and analyzed in every batch of 20 or fewer samples when possible.

In some cases, the client may specify which sample is to be used for the MS/MSD. If not, the laboratory picks a representative sample at random. Advisory MS/MSD recovery limits are established for aqueous and soil matrices.

For TCLP analysis, a matrix spike is prepared and analyzed for each waste type (e.g. oil, solid) associated with a batch of 20 or fewer samples of similar matrix.

11.2.2.9 Surrogates

As a means of monitoring individual sample extraction efficiency, one or more surrogate compounds are added to each blank, LCS, client sample, and QC sample prior to preparation. Recovery limits for surrogate compounds are established by the laboratory through control charting of each analyte. Typically, one of the following actions will be required when a sample surrogate recovery is out of the established control limits.

- Re-extract and/or reanalyze the sample
- Flag the results as estimated

11.2.2.10 Clients may specify the required action to be taken for recovery failure. Client specific requirements are conveyed to the analytical sections through project management.

11.2.3 Tracking Internal QC Samples

The tracking of internal QC samples through the LIMS provides laboratory personnel with various types of information. This information is used for the following purposes:

11.2.3.1 Long term trends are monitored through the use of quality control charts. Any upward or downward change in the recovery of analytes signifies that some procedural change has taken place. If trending is observed, the Technical Director reviews all test procedures and makes any corrections as required.

11.2.3.2 The number of quality control samples as a function of total laboratory samples is monitored so as to ensure that the laboratory analyzes the adequate number of Quality Control samples for each extraction or analytical batch.

11.2.3.3 The following guidelines are followed when implementing and utilizing QC Charts:

11.2.3.3.1 Through LIMS the Technical Manager plots the percent recovery of the LCS analyte versus the date of preparation or analysis; whichever is most appropriate.

11.2.3.3.2 For organic analyses employing surrogates, the LCS surrogate % recoveries are monitored on QC Charts. The recovery of at least one target Aroclor (PCB) in the Pesticide/PCB LCS is monitored on a QC Chart (e.g. TPH).

11.2.3.3.3 For trace metals determined by inductively coupled plasma (ICP) at least three metals spiked in the LCS are monitored on QC Charts (e.g. Cd, Cr, Ni). For trace metals determined by graphite atomic absorption (GFAA) and cold vapor atomic absorption (CVAA), an LCS for each element is monitored on a QC chart.

11.2.3.3.4 For General Chemistry, an appropriate LCS for each method is used. Each LCS analyte recovery method is monitored on a control chart.

11.2.3.3.5 Each section, prior to the calculation of in-house limits establishes initial control limits. These preliminary limits are derived from published method criteria if available. If no such criteria are available, the preliminary limits will be mutually set and agreed to by the Section Supervisor, Laboratory Manager, Technical Director, and Quality Assurance Manager. A minimum of 20 points is recommended to establish the initial calculated control limits. In some cases, it may be appropriate to use fewer data points to establish the first set of calculated limits, however, at no time should fewer than seven data points are used.

11.2.3.3.6 Control chart limits are updated periodically when sufficient additional data points are available. Typically, limits are updated for each set of 20 to 50 new data points. More frequent updates may be warranted in some cases

11.2.3.3.7 Each control chart has upper and lower warning limits established at ± 2 standard deviations ($2\sigma_{n-1}$) from the mean % recovery (centerline).

11.2.3.3.8 Each control chart has upper and lower control limits established at ± 3 standard deviations ($3\sigma_{n-1}$) from the mean % recovery (centerline).

11.2.3.3.9 The analyst performing the method enters the data into LIMS. The data is evaluated frequently to identify trends that might occur in an “out of control” situation

11.2.4 The method blank is an analyte–free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

For the method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of interest can not exceed the method detection limit or required reporting limit. Section 5 lists certain conditions in which contaminated blanks may be used for quality control purposes.

11.2.5 An instrument blank may be run after any sample that gives a response that exceeds the calibration range for the instrument to show that there is no carry-over to the next analysis. The instrument blank shall consist of high purity solvent (e.g. hexane for pesticide analysis by GC/ECD, methylene chloride for semi-volatiles analysis by GC/MS).

11.2.6 An Initial Calibration Blank (ICB) is analyzed before sample analysis begins to verify there is no carryover contamination or instrument drift. ICB samples usually accompany inorganic instrumental analysis.

11.2.7 The analysis of sample duplicates that contain detectable quantities of analytes is an effective means for assessing the precision of an analysis. Refer to the individual analytical procedures or Table 5-6 for guidance concerning the frequency and criteria for sample duplicate analyses.

11.3 Inter-laboratory Quality Control

Each section of the laboratory may be given blind and double blind samples to analyze for requested parameters. Blind samples may be assigned in containers to be diluted, digested, and/or extracted and analyzed by the appropriate laboratory section. Double-blind samples may arrive on a pre-scheduled basis from a "client" as real samples to be analyzed by designated analytical sections for specific analytes.

11.3.1 Blind QC Samples

Blind QC samples may be used as a test of proficiency for analysts needing certification and/or qualification for performing an analysis. The Section Supervisor should obtain the QC sample from either the Quality Assurance Department of from a source independent from the source of standards for the analysis.

11.3.2 Double - Blind QC Samples

Quality Control samples may arrive from a "Client" to be analyzed for specific analytes. These samples will arrive as real samples and will not be known to anyone outside Quality Assurance and Project Management. The results of these double-blind samples will be sent to the "client" to be compared to the true value of the samples. The laboratory's performance on these samples will be compared to other laboratories in the program. These results will be mailed to the Quality Assurance Department. Results are used to identify areas needing improvement.

11.4 Out-of-Control Conditions in Laboratory Control Samples

11.4.1 Any of the following control chart conditions indicates the loss of process control:

11.4.1.1 Any one point that is outside of the control limits.

11.4.1.2 Any three consecutive points that are outside one of the warning limits.

11.4.1.3 Any eight consecutive points on the same side of the centerline.

11.4.1.4 Any six consecutive points are such that each point is larger (or smaller) than its immediate predecessor.

11.4.1.5 Any obvious cyclic or repetitive pattern seen in the points.

11.4.2 Reactions to "Out-of-Control" Conditions

In the event of an "out-of-control" condition, the analyst should respond to the condition in the following manner:

11.4.2.1 Stop analysis.

11.4.2.2 Investigate the root cause of the failure

11.4.2.3 Implement any required corrective action.

11.4.2.4 Document the situation in a non-conformance memo prior to initiating subsequent analyses.

11.5 Identification of Analytes

11.5.1 Organic Analyses

The identification of analytes is accomplished by comparison of unknown samples with known standards. All standards shall be traceable as specified by the applicable analytical procedure.

11.5.1.1 Gas Chromatography

All sample identifications are made by a comparison of the retention time of the standard peak to the retention time of the unknown peak. The identification of any analyte, which is identified during the primary analysis, is verified through the use of a confirmation column or by GC/MS unless specifically exempted in the applicable procedure.

11.5.1.2 Gas Chromatography/Mass Spectrometry (GC/MS)

For positive identification of an analyte by GC/MS, the spectrum of the analyte must conform to a spectrum of the authentic standard obtained after satisfactory tuning of the mass spectrometer. The appropriate analytical methods should be consulted for specific criteria for matching the mass spectra, relative response factors and relative retention times to those of authentic standards. Tentative identifications may be made based on conformance to published mass spectra in reference texts or spectral library databases.

11.5.2 Inorganic Analyses

The identification of analytes is accomplished by comparison of unknown samples with known standards. All standards shall be traceable as specified by the applicable analytical procedure.

11.5.2.1 Metals

The concentration of a metal analyte is based on the absorption or emission of light measured at a specific wavelength. The wavelength selected is in accordance with the applicable procedure. Standards used to generate the calibration curve are traceable to NIST or other nationally recognized (e.g. EPA).

11.5.2.2 Wet Chemistry

Standards used to prepare calibration curves or to standardize instruments are traceable to NIST or other national sources (e.g. EPA).

11.6 Quantitation and Reporting of Analytes

11.6.1 Reduction of Sample Data

Data reduction is defined as the processing of instrument generated numbers by an analyst to achieve a final result. Data reduction is used for sample analysis as well as for quality control criteria. Processing of numbers may be achieved using manual calculations and/or computer aided calculations.

11.6.1.1 All data reduction follows calculations found in approved procedures for the analysis.

11.6.1.2 An analyst who is qualified to perform the analysis performs all data reduction. If a Section Supervisor performs data reduction, another qualified analyst reviews the data.

11.6.1.3 All numbers used in the reduction of data are present on data reports and are easily retrievable.

11.6.1.4 All computer-generated calculations are performed using a validated program/spreadsheet.

11.7 Reporting Data

11.7.1 Significant Digits

All digits in a reported result are considered to be definite, except for the last digit, which may be in doubt. Such a number is said to contain only significant figures. If more than a single doubtful digit is carried, the extra digit or digits are not significant. The following rules apply to all reported analytical results from all laboratory sections:

11.7.1.1 All digits from a measurement are recorded. These numbers are used in the calculation of the results. After all calculations have been performed, the number is rounded to the required number of significant digits.

11.7.1.2 The number zero may or may not be a significant digit, depending on its placement of the reported result.

11.7.1.3 Final zeros, after a decimal, are always significant (Ex. 9.80 has three significant figures).

11.7.1.4 Zeros before a decimal point with non-zero digits preceding them are significant. Zeros with no non-zero digits before them are not significant (e.g. 10.3 has three significant digits, 0.53 has two significant digits).

11.7.1.5 If there are no non-zero digits preceding a decimal point, the zeros after the decimal point but preceding other non-zero digits are not significant. These zeros only indicate the position of the decimal point.

11.7.1.6 The final zero in a whole number may or may not be significant.

11.7.1.7 When mathematical functions are performed on multiple numbers, the number with the least number of significant digits dictates how many significant digits the end result should have.

11.7.2 Rounding Rules

11.7.2.1 Once the number of significant figures obtainable from a particular analysis is established, data resulting from the analysis are reduced according to the standard rules for rounding which state: If the number value to be rounded is 5 or greater, round up. If the number value is less than 5, round down.

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11.7.2.2 Rounding off numbers is a necessary operation in all analytical sections of the laboratory. It is automatically applied by the limits of measurement of every instrument and all glassware.

11.7.3 Reporting Units

The appropriate unit of measurement shall accompany all sample results reports.

11.7.4 Reporting on a Wet vs. Dry Weight Basis

When required, non-liquid sample results are reported on a dry weight basis and this information is documented in the report. When results are reported on a wet weight basis, the results are reported "as is".

11.7.5 Reporting % Recovery and RPD

Unless otherwise directed by the customer, the Technical Director, or the QA Manager, the % Recovery and RPD are reported to one decimal place.

11.8 Storage of Quality Related Data

The laboratory retains all data and information that pertains to a project for a period of 5 years (10 years for lead data per AIHA). The data may be stored electronically, as hard copy, or both.

11.8.1 Calibration Data

All calibration data, which pertains to a specific project, is stored in an easily retrievable manner. Easily retrievable manner is defined as retrievable in the same day for current projects, or within 24 hours for archived projects.

11.8.2 Quality Control Data

All quality control related data (i.e. blanks, blank spikes/duplicates, matrix spikes/duplicates, etc.) is stored in the associated project file. If more than one project is associated with the QC data, copies are made and stored with each associated project.

11.8.3 Logbooks (Notebooks)

Laboratory logbooks are kept in the laboratory while in use. Once completed, the logbooks are archived in an easily retrievable location.

11.8.4 QC Charts

While in use, QC charts are stored in LIMS. When the QC Chart is no longer being used, it is archived by the section in a central location in the Server.

11.9 Internal Performance Audits

Internal performance audits are a means for the Quality Assurance Department to determine the applicability, effectiveness, and utilization of procedures by all sections. Designated personnel perform the performance audits. At the beginning of each new year, and on an on-going basis, a

schedule of audits and surveillance is developed and updated by the Quality Assurance Section. Surveillance is performed on an unannounced basis with the sections so that objectivity may be maintained. Findings from audits and surveillance are documented and corrective actions are implemented. Additional surveillance is scheduled to ensure that all deficiencies are corrected.

11.10 Failure of Quality Control Indicators

When there is a quality control failure that impacts data quality, the event must be documented using the procedures described in Section 13 of this document.

12.0 DATA REDUCTION, REVIEW AND REPORTING

12.1 Introduction

In order to provide the highest quality data possible, an extensive system for data reduction, review, and reporting has been implemented.

12.2 Sample Analysis and Data Reduction

Through the use of the worksheets, the samples are prepared following the procedures given in each of the SOPs that follow EPA's approved methods. The preparation information is recorded in bound notebooks throughout the laboratory.

12.2.1 Data Reduction

Most sample concentration results are read directly from instrumentation without further reduction or calculations. Dilution factors are applied upon the dilution of samples having concentrations above the calibration range. In many cases, these are input into the instrument computer and correct results are calculated automatically. In other cases, a manual calculation may be made.

Data from methods requiring manual reduction prior to reporting include titrimetric methods, BOD, COD, conductivity, manual UV/VIS/IR and residue.

All laboratory pH meters are temperature compensated.

The laboratory raw data containing the instrument-generated reports, manually calculated results, and all supporting preparation, calibration, and analytical data are scanned as pdf file and posted in laboratory archives (portal server).

12.2.2 Chromatographic and Data File Identification

Chromatograms and data files are given a unique alphanumeric identification by the chemists initiating the analyses in each section. These file identification numbers reflect either the date the sequence was initiated (GC sections), the order in which the samples were analyzed (GC/MS sections), and/or the sample identification and log numbers given by the client and listed on the LIMS.

12.3 Data Transfer and Review

12.3.1 Data Transfer to LIMS

The analytical results are entered on the department worksheets after review or by direct electronic transfer from the instrument data system. The analysts enter the worksheet data into the LIMS. After the data is entered into the LIMS, approval sheets are printed and checked against the information entered into the LIMS for transcription errors and anomalies.

12.3.2 Data Review

Laboratory analytical results are reviewed by at least two analysts or a section supervisor prior to entering the reportable data into the LIMS. The review of the data includes checking the extraction, digestion, distillation, and other preparation logs, ensuring that all precision and accuracy requirements are addressed, and ensuring that all steps of the analyses have been completed. If any problems were indicated during the analysis of the sample batch, it is the responsibility of the analyst and the section supervisor to bring this to the attention of the project manager, section manager and QA manager through a written corrective action report.

12.3.3 Data flags

Data flags are used on reports as needed to inform the project manager and the client of any additional information that might aid in the interpretation of the data. The data flagging system incorporates data qualifiers which are similar to flags specified in the Contract Laboratory Program protocols, as well as additional flags used to help explain batch specific events.

12.3.4 Final Report

When data acquisition and reporting have been completed, the project manager reviews and prepares the final report. Because the project managers have extensive experience in evaluating analytical data, they have developed both objective and subjective techniques for data review. Each value reported is reviewed in the context of the respective environmental matrix and all available QC/QA data.

12.3.4.1 Abnormal values are carefully scrutinized, and samples are reanalyzed if the abnormalities cannot be explained.

12.3.4.2 If the results from spiked samples suggest interferences (low or high bias), attempts are made to remove the interferences, or the data is flagged and/or a project narrative is included with the report. Laboratory qualifiers are defined as follows:

* - Value exceeds maximum contaminant level
B - Analyte detected in the associated method blank
BRL - Below Reporting Limit
E - Estimated (Value reported above quantitation range)
H - Holding times for prepation or analysis exceeded
J - Estimated value detected below Reporting Limit
N - Analyte NELAC (TNI) certified
Narr - See Case Narraitve
NC - Not Confirmed
R - RPD outside accepted recovery limits
Rpt Lim - Reporting Limit
S - Spike recovery outside accepted recovery limits
> - Greater than Result value
< - Less than Result value

12.3.4.3 Clients are instructed to provide sufficient sample for the analysis of Matrix Spike and Matrix Spike Duplicate analysis, however there are times when the laboratory does not receive sufficient aqueous sample volume to perform these analyses. If an aqueous sample batch is analyzed without the inclusion of a spike/spike duplicate

sample(s), this fact is added to the report narrative per TNI requirements. Example verbiage is as follows:

The TNI requirement for the analysis of a matrix spike/matrix spike duplicate could not be performed on Batch (#) due to insufficient sample volume submitted.

12.4 Special Project or Data Package Review

If the client requests special handling and/or data packages, the Laboratory Director, Technical Director, or Quality Assurance Manager may also review the project report and the raw data. This review includes checking holding time requirements and calibrations, reviewing all quality control data and/or control charts, and initiating any corrective actions or re-analyses that might be appropriate.

12.5 Quality Control Reports

AES, Inc. offers four levels of quality control reporting. Each level contains all the information provided in the preceding level, in addition to its own specific requirements. The quality control packages provide data in the following levels:

12.5.1 Level I – method references, preparation and analysis dates, surrogate(s) recoveries and reporting limits.

12.5.2 Level II – Level I information plus results for the blank, LCS and MS/MSD and sample duplicates.

12.5.3 Level III – Level I and II information plus all raw data associated with sample preparation, instrument calibration (if applicable) and sample analysis.

12.5.4 Level IV – Level I, II and III information in a CLP "look-alike" format, and all sample raw data.

12.6 Reporting Criteria

The final report is printed and signed by the Laboratory Manager, the Director of Project Management or a Project Manager after all review has been completed. The Laboratory Manager, the Director of Project Management and Project Managers serve as designees for technical director for report signing. The data flags that may appear in a project report are defined and any additional comments are included in the Case Narrative.

12.6.1 If requested by the client or a project specific QA Plan, custom reports or data packages can be provided. When data packaging is requested, a paginated data package is provided in addition to the project report. The format of the project report and/or data package can be adjusted to meet the needs of the client. All LIMS reports can be downloaded onto diskettes or to most clients' computers.

12.6.2 When the project report must meet TNI requirements, the report will include a certification statement indicating the results meet TNI standards, an estimated uncertainty statement, and a format that includes the total number of pages in the report.

12.6.3 AES, Inc., will not intentionally divulge to any person (other than a client or person designated by a client in writing) any information regarding the services provided by AES or any information disclosed to AES by the client. Any information *known* to be potentially endangering to national security or any entity's proprietary rights will NOT be released.

12.6.4 Test results are reported according to client requirements. If a client requests to have reports or information sent by fax, the client is notified in advance of the transmission, whenever possible, and all documents include a cover sheet with the following statement:

NOTICE OF CONFIDENTIALITY

The information contained in this facsimile message may be legally privileged and is confidential information intended only for the use of the individual or entity named above. If the reader of this message is not the intended recipient, you are hereby notified that any use, dissemination, distribution or copy of this facsimile message is strictly prohibited. If you have received this facsimile message in error, please contact us by telephone at (770) 457-8177 and return the facsimile message to us at the address above via the US postal service.

12.7 Record Keeping

Procedures are in place to ensure that all records required under TNI Chapter 5 and AIHA program requirements are retained. The laboratory maintains a record keeping system that can produce unequivocal, accurate records that document all laboratory activities.

12.7.1 When an analytical batch is prepped and analyzed, the analyst enters the data into the LIMS system and gives the raw data, quality control data and a copy of the prep log (if applicable) to the department manager to review.

12.7.2 Any problems encountered during sample preparation and analysis are corrected and brought to the attention of the department manager.

12.7.3 Once the department manager has reviewed the data, it is validated in the LIMS system for reporting to the client.

12.8 Records of Analysis

12.8.1 Sample Preparation, Extraction, Distillation, and Digestion

All steps of the preparation, extraction, distillation and/or digestion of samples are thoroughly documented. Documentation is determined by the QA Manager, Laboratory Manager, and the Technical Director and includes (if applicable):

12.8.1.1 Standard Identification

12.8.1.2 Dilution Factors

12.8.1.3 Sample Identification

12.8.1.4 Reagent Identification

12.8.1.5 Date the extraction, digestion, and or analysis was performed

12.8.1.6 Initials of the analysts performing the digestion, extraction, and or analysis

12.8.1.7 Volume/weight of sample used

12.8.1.8 Final volumes/weights

12.8.1.9 Initial and final review signatures, where required

12.8.1.10 Instruments used

12.8.2 Preparation of Standards and Reagents

12.8.2.1 The preparation of all standards and reagents are documented. The lot numbers of all standards associated with a particular project are traceable either through the instrument logbook, a QC check list, a worksheet, or another approved document.

12.8.2.2 Original vendor Certificates of Analysis are distributed by the Shipping and Receiving Office to the intended departments.

12.9 **Standard and Reagent Traceability**

Standards and reagents are tracked in the LIMS chemical inventory system for traceability and auditing purposes. The method of standard and reagent tracking is outlined in the subsequent sections.

12.9.1 When a standard or reagent is needed that is not already on the approved vendor / materials order list, supervisors forward purchase requests to the Technical Director and / or Laboratory Manager for approval. The standard or reagent is ordered from a reputable supply house (AES typically uses VWR). The laboratory attempts to use certified reference materials from providers accredited to ISO/IEC 17025.

12.9.2 The information supplied to the Technical Director and / or Laboratory Manager must have the supplier standard or reagent name, order number, size or amount of each unit, grade or purity, price, if possible, and quantity. Upon receipt, supplies (and services) are reviewed to ensure they comply with requirements. When a vendor has been approved for services, a note is placed in the comments field of the Vendors database within LIMS.

12.9.3 When the standard or reagent arrives, it is logged into the LIMS, usually by the department supervisor or by the sample custodian. All reagents and standards received are electronically tracked and documented by computer via the Laboratory Information Management System.

12.9.4 Each standard or reagent is given a unique chemical inventory number upon receipt. The next available number in the LIMS is automatically assigned, starting with #5001. The computer entry is completed by entering the correct information in the required fields.

12.9.4.1 The expiration date for neat standards and reagents is determined using the manufacturer's expiration date, if available. Otherwise, a 1 year expiration date is assigned to volatile organic compounds and standards and 5 year date for acids, dry chemicals, solvents, reagents, and other chemicals. Each standard and reagent is clearly and permanently labeled with its expiration date in indelible ink. The assigned expiration date for intermediate standards will not exceed the manufacturer's expiration date of the stock standard.

12.9.4.2 Secondary standard containers are labeled with the corresponding LIMS tracking number of the source material, the date the contents were prepared, the six month expiration date, the name of the analyte(s), the concentration of each component of the solution, the matrix and the initials of the person who prepared it.

12.9.4.3 The chemical inventory number must appear on both the standard and reagent container, and the upper, right-hand corner of the certificate of analysis. It must also

be included, if applicable, in standard/preparation, analyses or sample preparation log books.

12.9.4.4 Secondary standard labels include the LIMS chemical inventory number, the standard name, intended use (spiking, surrogate, reference or calibration solution), and concentration with units, matrix, expiration date and initials of the person who prepared it. As long as this is available, all other information can be found in the LIMS.

12.9.5 Spiking, surrogate, reference and calibration solutions and calculations are recorded in the appropriate "Standard/Preparation Log Book." Logbooks cover the following areas: Organics, Organics Preparation, Semi-Volatile Organics, Microbiology, Metals, Mercury and Wet Chemistry.

12.9.6 Some containers such as standards containers for organics are small and there may not be enough room to list all of the required information on the container. Should this occur, it is permissible to attach a label to the bottle.

12.9.7 When a standard or reagent is added to a sample for any reason, the LIMS chemical inventory number of that standard or reagent and the amount added must be recorded in the appropriate logbook. For example, if a stock standard MET #33-89-5431 of 1000 mg/L is diluted to 100 µg/L, the following line is entered: 1 ml MET #33-89-5431 to 100 ml DI water, 1 ml of 100x to 100 ml DI water, final conc. = 100 µg/L. (NOTE: "MET #33-89-5431" = Metals Department Standard/ Preparation Log Book 33, page 89, LIMS Chemical Inventory Number 5431).

12.9.8 If the standard is used as a stock standard and aliquots of it are diluted to produce working standards, the stock standard's LIMS chemical inventory number is used. The standard concentration or a designator such as "1" or "A" is used to differentiate between each serial dilution.

12.10 Standard Verification

12.10.1 Certificates of Analysis

12.10.3.1 Each department is responsible for maintaining all certificates of analysis received with its standards and reagents. The LIMS-assigned chemical inventory number is written in the upper, right-hand corner of each COA. The certificates are maintained in the area offices in numerical order and bound into volumes as required, but not less than annually. Once bound, the certificates are moved to archival storage and held for a minimum of five years.

12.10.3.2 Most accrediting authorities require that a certificate of analysis is kept on file for all standards used in the laboratory. If at all possible, a certificate for reagents should also be obtained. This documentation serves two purposes; 1) it gives further traceability for the standard or reagent, and 2) it provides a manufacturer's guarantee that the standard is comprised of the compounds at the levels listed.

12.11 **AIHA Estimation of Uncertainty**

This Estimation of Uncertainty Policy follows the AIHA-LAP, LLC Accreditation Program requirements with respect to the estimation of uncertainty measurement for tests associated with their scope of accreditation. The requirement which underlies this policy is found in ISO/IEC 17025, Clauses 5.4.6 and 5.10.3.1 c).

12.11.1 Definition of the measurand - Quantity intended to be measured or analyte concentration. The measurands for methods under AIHA accreditation are available in the SOPs.

12.11.2 Identification of the Contributors to Uncertainty

From guidance spreadsheets provided on the AIHA website at:
http://www.aihaaccreditedlabs.org/PolicyModules/Pages/default.aspx

Contributors are listed for certified chemistry, chromatography, and lead testing methods in the following tables.

12.11.3 The laboratory utilizes Type A approach for the Estimation of Uncertainty. On or more of the following options are utilized:

12.11.3.1 Uncertainty specified within a standard method. In those cases where well recognized test method (such as NIOSH, OSHA, etc. method), specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, laboratories need not do anything more than to follow the reporting instructions as long as they can demonstrate they follow the reference method without modification and can meet specified reliability.

12.11.3.2 Laboratory Control Samples (LCS) and Matrix Spikes. In cases where matrix specific LCS (CRM or media spikes) and/or matrix spike data are available, include uncertainty estimated from the standard deviation of long term data collected from routine sample runs for existing test methods or from the standard deviation of the LCS or matrix spike data for method validation/verification studies for new test methods.

12.11.3.3 Duplicate Data. In cases where sub-sampling occurs and there are data over the reporting limit, include uncertainty estimated from long term duplicate data collected from routine sample runs for existing test methods or method validation/verification studies for new test methods.

12.11.3.4 Proficiency Testing (PT) Sample Data. In cases where the previous options are not available and where PT samples are analyzed with sufficient data above the reporting limit, pooled PT sample data can be used to estimate uncertainty.

12.11.4 Identification of the contributors of variability for qualitative test methods.

12.11.5 The reporting procedure.

Typically, measurement uncertainty is reported per the client's request or when the *known compliance* to a specification limit is affected. The result and the expanded measurement uncertainty are reported in the same units. Both the result and expanded measurement uncertainty will be rounded to the same number of significant figures.

Applicable to methods N0500 / N0600

Common Contributors to Measurement Uncertainty – Chemical Analyses

(representative list - may not be all inclusive for all types of analyses)
(QC sample types in this list are typical of those utilized in AIHA-LAP, LLC laboratories)
See additional tabbed sheets for examples

Contributors to Uncertainty	Representative and Applicable QC Data	Comments to Clarify Contributor Effects
Transportation/Storage/Handling:		
shipping time, container and temperature	NA / (FS)	**NOTE: This is not part of analytical uncertainty, but must be considered by labs providing sampling and when providing guidance regarding sample packaging and shipping.** Usually no impact if recommended shipping conditions and holding times in referenced methods are maintained. Improper packing materials, bulks shipped w/samples, etc. may adversely affect data. Field blanks, field spiked samples or duplicate field samples shipped with samples or included in method validation studies may reflect these contributors. Field variability (FS/DUP) is only considered when lab is responsible for sampling.
lab storage time, conditions and temperature	NA / (LCS, FS)	**Usually no impact if recommended storage conditions and holding times in reference methods are maintained.** Impact is monitored per sample batch only if LCS samples are prepared on receipt and stored with field samples. Field spiked samples or stability study samples included in some method validation studies may also reflect these contributors. Field variability (FS/DUP) is only considered when lab is responsible for sampling.
contamination in lab storage areas	NA / LCS, FB	**Usually no impact if recommended storage conditions and holding times in reference methods are maintained.** Improper storage such as sorbent tubes stored with bulk solvent samples or near solvent sources may adversely affect data. Impact is monitored only if LCS is prepared on receipt & stored with field samples. Field blank can be used to assess contamination from collection, transport, and storage.
Laboratory Subsampling		**NOTE:** This primarily applies to bulk/solid samples which requires use of laboratory duplicate sample data to determine impact on uncertainty. It is not applicable for LCS/LCSD media spike duplicates.
sample nonhomogeneity	DUP	Sample composition, etc.
blending techniques	DUP	Stirring, sieving, grinding, etc.
sample size	DUP	Large enough to allow adequate subsampling.
Sample Preparation:		
volumetric glassware	LCS, DUP	NA for Class A; applies for graduated tubes or cylinders, etc.
dispensing device	LCS, DUP	Pipettes, and other types of dispensers that are not Class A.

balance	LCS, DUP	Balance error is often insignificant compared to other MU sources.
temperature	LCS, DUP	Hot plate or ashing temperatures.
sample extraction	LCS, DUP	Applies to LCS or DUP if it goes throughthe entire sample preparation process.
extractant background	LCS, DUP, MB	Analyte or interferant is present in acids, solvents, etc.
Lab Environmental Conditions:		
temperature variance	LCS, DUP	Room temp during bulk asbestos, gravimetry, etc. processes.
humidity variance	LCS, DUP	Gravimetry involving hydroscopic media, etc.
Analysts:		
different analysts	LCS, DUP	Must use inter-analyst instead of intra-analyst repeat data, where applicable.
analyst training level and experience	LCS, DUP	Must use inter-analyst instead of intra-analyst repeat data, where applicable.
data interpretation by analyst	LCS, DUP	Chromatographic peak ID, interference corrections, etc. Must use inter-analyst instead of intra-analyst repeat data, where applicable.
Measuring Instruments:		
instrument stability	LCS, DUP	Baseline drift, repeatability of averaged readings, lab environmental stability, etc.
carry over effects	LCS, DUP	Impact of high samples on following sample readings; can be monitored by proper use of CCBs.
day to day calibration differences	LCS	Variation in instrument response and calibration process
interferences	LCS	Due to matrix, inter-element effects, co-eluting GC peaks, etc.
Calibration Standards/Reference Materials:		
preparation variances	LCS	Due to analysts, balances, dispensing devices used, etc
calibration stock material uncertainty	CERTIFICATE	Obtain from certificate or estimate, can be ignored if less than 1/3 of the largest contributor.
LCS reference material uncertainty	CERTIFICATE	Only has impact when LCS data are used to correct customer sample results. Obtain from certificate or estimate. Can be ignored if less than 1/3 of the largest contributor. Note that use of an LCS with a large uncertainty can result in over estimation of overall analytical uncertainty.
Test Procedure Variations:		
variation within and between reagent lots	LCS	Similar to extractant background effects under Sample Preparation above.
extraction or digestion times, temperatures, and conditions	LCS	May affect complete dissolution of analyte or loss of material in some cases.
sample dependent modifications	LCS	Changes in conditions due to sample size, customer requests, etc.
desorption efficiencies within and between lots for sorbents	LCS	May vary by lot or manufacturer; also applies to diffusion rates for passive monitors.
Data Manipulation:		
sampling media/blank correction	LCS, MB	When significant and when data are blank corrected.

instrument blank correction	LCS	When allowed.
accuracy of calculations	LCS	Manual, spreadsheet, LIMS, etc.
area or air volume sampled	NA	Typically provided by the customer. This is not part of analytical uncertainty, but must be considered by labs providing sampling and providing combined sampling and analytical uncertainty.

Where:
DUP = Duplicate, resulting from sub-sampling of a bulk (NOTE: NOT LCS/LCSD duplicate spiked sampling media)
FB = Field Blank
FS = Field Spike - Not typically conducted unless part of sampling method validation. Should be considered only when laboratory is responsible for field sampling.

LCS = Laboratory Control Standard, matrix matched and typically taken through the entire analytical process with each sample batch
MB = Method or Matrix Blank
NA = Not Applicable

Applicable to N1003, N1022, N1300, N1400, N1450, N1457, N1500, N1501, N1550, N2000, N2500, N5506, and 3M 3520

Example of Contributors to Measurement Uncertainty
Chemical Analyses of Sorbent Tubes using Chromatography
See Example Calculations (to the right of the table)

Contributors to Uncertainty	Representative and Applicable QC Data	Comments to Clarify Contributor Effects
Transportation/Storage/Handling		
shipping time, container & temperature	NA	Limited impact on most sorbent tubes
lab storage time, conditions & temperature	NA	Usually no impact if recommended storage conditions and hold times are maintained. LCS samples are representative if prepared on receipt & stored with field samples - usually no impact if recommended storage conditions and holding times are maintained. Field spiked samples or stability study samples included in some method validation studies may also reflect these contributors.
contamination in lab storage areas	NA	Usually no impact if appropriate storage conditions are maintained. Field blank can be used to assess contamination from collection, transport, and storage
Laboratory Subsampling		
sample nonhomogeneity	NA	Not applicable to sorbent tube analysis
blending techniques	NA	Not applicable to sorbent tube analysis
sample size	NA	Not applicable to sorbent tube analysis
Sample Preparation:		
volumetric glassware	LCS	Same type of glassware used for samples and LCS
dispensing device	LCS	Same type of dispensing device
balance	NA	Not applicable to sorbent tube analysis
temperature	NA	Not applicable to sorbent tube analysis
sample extraction	LCS	Applies to LCS if goes through sample preparation
extractant background	LCS, MB	Analyte or interferant in solvents or other prep reagents used, etc.
Lab Environmental Conditions:		
temperature variance	LCS	LCS results reflect any temperature effects on chromatography instrument
humidity variance	NA	Not applicable to sorbent tube analysis
Analysts:		

different analysts	LCS	LCS results reflect variability due to different analysts, as applicable, on different days
analyst training level & experience	LCS	LCS results reflect variability due to different analysts, as applicable, on different days
data interpretation by analyst	LCS	LCS results reflect variability due to different analysts, as applicable, on different days
Measuring Instruments:		
instrument stability	LCS	LCS results reflect instrument variability on different days
carry over effects	LCS	LCS results reflect instrument variability on different days
day to day calibration differences	LCS	LCS results reflect instrument variability on different days
interferences	LCS	LCS results reflect instrument variability on different days
Calibration Standards/Reference Materials:		
preparation variances	LCS	Due to analysts, dispensing devices used, etc
calibration stock material uncertainty	CERTIFICATE	Obtain from certificate or estimate
LCS reference material uncertainty	CERTIFICATE	Use if customer sample data corrected for desorption efficiency. Obtain from certificate or estimate.
Test Procedure Variations		
variation within and between reagent lots	LCS	LCS subjected to same treatment as customer samples
extraction or digestion times and temps	LCS	LCS subjected to same treatment as customer samples
sample dependent modifications	LCS	LCS subjected to same treatment as customer samples
desorption efficiencies within and between lots for sorbent tubes	LCS	LCS subjected to same treatment as customer samples
Data Manipulation:		
sampling media blank correction	LCS	LCS subjected to same treatment as customer samples
instrument blank correction	LCS	LCS subjected to same treatment as customer samples
Accuracy of calculations	LCS	LCS subjected to same treatment as customer samples

LCS = Laboratory Control Standard, typically taken through the entire analytical process with each sample batch
MB = Method or matrix blank
NA = Not Applicable

Applicable to SW-7420, N7000B, N7082, ~~N9100,~~ N91002, and N7300

Example of Contributors to Measurement Uncertainty
Chemical Analyses of Lead (Pb) using ICP-AES and FAA
See Example Calculations (to the right of the table)

Contributors to Uncertainty	Representative and Applicable QC Data	Comments to Clarify Contributor Effects
Transportation/Storage/Handling		
shipping time, container & temperature	NA	No impact on bulk paint samples from transportation, storage or normal handling
lab storage time, conditions & temperature	NA	
contamination in lab storage areas	NA	
Laboratory Subsampling		
sample nonhomogeneity	DUP	Sample composition, etc.
blending techniques	DUP	Stirring, sieving, grinding, etc
sample size	DUP	Large enough to allow adequate subsampling
Sample Preparation:		
volumetric glassware	LCS, DUP	NA for Class A; applies for graduated tubes or cylinders, etc.
dispensing device	LCS, DUP	pipettes, and other types of dispensers not Class A
balance	LCS, DUP	balance error is often insignificant compared to other MU sources
temperature	LCS, DUP	Hot plate or ashing temperatures
sample extraction	LCS, DUP	Applies to LCS or DUP if goes through sample preparation
extractant background	LCS, DUP, MB	Analyte or interferant in acids, or other reagents
Lab Environmental Conditions:		
temperature variance	NA	No impact on bulk paint samples
humidity variance	NA	No impact on bulk paint samples
Analysts:		Analyst contributors affect all aspects of analysis from subsampling through data manipulation
different analysts	LCS, DUP	
analyst training level & experience	LCS, DUP	
data interpretation by analyst	LCS, DUP	
Measuring Instruments:		
instrument stability	LCS	Baseline drift, repeatability of averaged readings, etc
carry over effects	LCS, DUP	Impact of high samples on following sample readings; can be monitored by proper use of CCBs
day to day calibration differences	LCS	
interferences	DUP, MS	Due to matrix, inter-element effects, etc. Cannot be routinely determined for typical industrial hygiene sampling media
Calibration Standards/Reference Materials:		
preparation variances	LCS, DUP	Due to analysts, balances, dispensing devices used, etc
calibration stock material uncertainty	CERTIFICATE	Obtain from certificate or estimate
LCS reference material uncertainty	NA	Sample results not corrected for LCS recovery

Test Procedure Variations		
variation within and between reagent lots	LCS	Similar to extractant background effects under Sample Preparation above
extraction or digestion times and temps	LCS	May affect complete dissolution of analyte or loss of material in some cases
sample dependent modifications	LCS	Changes in conditions due to sample size, customer requests, etc
desorption efficiencies within and between lots for sorbent tubes	NA	
Data Manipulation:		
sampling media blank correction	NA	No sampling media with bulk samples
instrument blank correction	LCS	when allowed
Accuracy of calculations	LCS	Manual, spreadsheet, LIMS, etc

DUP = Duplicate, resulting from sub-sampling of a bulk (NOTE: NOT LCS/LCSD duplicate spiked sampling media)
FB = Field Blank
FS = Field Spike
LCS = Laboratory Control Standard, matrix matched and typically taken through the entire analytical process, with each
sample batch
MB = Method or matrix blank
NA = Not Applicable

Applicable to Air (SOP MB-15019, MB-15022, MB-15028), Bulk (SOP MB-15020), and Surface Direct (SOP MB-15020) Exam

Example Contributors to Measurement Uncertainty – Direct Air Environmental Microbiology Analyses

(representative list - may not include of all contributors)
(QC sample types in this list are typical of those utilized in AIHA-LAP, LLC laboratories)
See Example Calculations (to the right of the table) and tabbed sheets for additional examples

Contributors to Uncertainty	Representative and Applicable QC Data	Comments to Clarify Contributor Effects
Temperature, Storage, Handling:		
shipping time, container & temperature	NA	No impact on direct air exam samples
lab storage time, conditions & temperature	NA	No impact on direct air exam samples
contamination in lab storage areas	NA	No impact on direct air exam samples
Laboratory Subsampling:		
sample nonhomogeneity	NA	Not applicable to direct air exam samples
blending techniques	NA	Not applicable to direct air exam samples
sample size	NA	Not applicable to direct air exam samples
Sample Preparation:		
slides & coverslip contamination	MB	With proper care there should be no contamination of daily blanks; therefore, no impact
mounting medium	MB	With proper care there should be no contamination of daily blanks; therefore, no impact
Lab Environmental Conditions:		
seasonal background spore variances	MB	Samples are not exposed to air for any length of time; therefore there should be no impact
Analysts:		
different analysts	RS	Reference slides analyzed by multiple analysts
analyst training level & experience	RS	Reference slides analyzed by multiple analysts
data interpretation by analyst	RS	Reference slides analyzed by multiple analysts
Measuring Instruments:		
microscope magnification level used	RS	Reference slides analyzed with multiple microscopes
eye piece graticule & field of view calibration	RS	Reference slides analyzed with multiple microscopes
Test Procedure Variations:		
portion and fields of sample analyzed	RS	Varies by analyst
microbial density	RS	High concentrations or clumps of spores may impact results
interferences	RS	Debris level and resolution of spores in field of view
ranges (high, medium, low)	RS	Uncertainty may be concentration dependent. Lab should evaluate this as part of method validation.
Data Manipulation:		
reading, interpreting and reporting results	RS	
Accuracy of calculations	RS	Manual, spreadsheet, LIMS, etc

area or air volume sampled	NA	Typically provided by the customer. This is not part of analytical uncertainty, but must be considered by labs providing sampling and providing combined sampling and analytical uncertainty.

MB = Daily method blank
RS = Daily reference slides

Please note that the original column I (CV of the pair) of the “culturable analyses” tabbed worksheet had a formula incorrectly entered. The worksheet has been corrected and any affected values have been highlighted in yellow.

Applicable to Culturable Air (SOP MB-15024), Bulk (SOP MB-15023), and Surface (SOP MB-15023) Exam

Example Contributors to Measurement Uncertainty – Culturable Environmental Microbiology Analyses

(representative list - may not include of all contributors)
(QC sample types in this list are typical of those utilized in AIHA-LAP, LLC laboratories)
See Example Calculations (to the right of the table)

Contributors to Uncertainty	Representative and Applicable QC Data	Comments to Clarify Contributor Effects
Temperature, Storage, Handling:		
shipping time, container & temperature	NA	May affect growth but not under control of lab so no impact on analytical uncertainty
lab storage time, conditions & temperature	NA	Affects growth rates of organisms in samples. Study samples included in some method validation studies may reflect these contributors.
contamination in lab storage areas	NA	As long as samples are contained there should be no impact
Laboratory Subsampling:		
sample nonhomogeneity	DUP	Area of bulk samples selected for analysis
blending techniques	DUP	Extraction/vortexing non-homogeniety of samples
sample size	DUP	
Sample Preparation:		
contamination during preparation	BLK	Sterility of materials used/autoclave operation
sample homogenization/subsampling	DUP	Contributions from this and the rest of Sample Prep contributors measured only if duplicate samples are prepared; not captured by inter-analyst readings of same sample prep
sample dilution (pipettes, etc)	DUP	Uncertainty related to method used to make serial dilutions
balance	DUP	Balance error is often insignificant compared to other MU sources
plating technique	DUP	How distributed on media
composition of sample	DUP	Organism competition on media
Lab Environmental Conditions:		
seasonal background organism variances	BLK	Should have minimal impact when proper aseptic techniques are used
Analysts:		
different analysts	DUP	Must use inter-analyst instead of intra-analyst repeat analyses.
analyst training level & experience	DUP	Must use inter-analyst instead of intra-analyst repeat analyses.
data interpretation by analyst	DUP	Must use inter-analyst instead of intra-analyst repeat analyses.
Measuring Instruments:		
microscope magnification level used	DUP	Determines level of detail of organisms that can be easily seen
eye piece graticule calibration	DUP	Proper calibration required to facilitate spore identification based in part on size

data logger	DUP	When used to assist in organism identification, e.g. bacteria
Test Procedure Variations:		
media used	DUP	Different media show preference for different organisms.
incubation conditions	DUP	Affects rate of growth but cannot be monitored unless using known concentration control cultures or do time/analysis study
identification techniques used	DUP	Especially for bacteria (chemical tests vs. data logger, etc)
microbial density	DUP	Organism competition (inhibition, overgrowth, etc)
interferences	DUP	Presence of organisms other than those of interest
Data Manipulation:		
reading, interpreting and reporting results	DUP	
Accuracy of calculations	DUP	Manual, spreadsheet, LIMS, etc

BLK = Lab Blank prepared when samples are prepared
DUP = Inter-analyst duplicate preparation and analysis

12.12 Recommended Storage Conditions

The locations for the storage of all standards, reagents, and working solutions are based upon compatibility of the material with other materials, flammability, and intended use of the material. The following general guidelines apply to the storage of standards and reagents.

12.12.1 The locations for the storage of all standards, reagents, and working solutions are based upon compatibility of the material with other materials, flammability, and intended use of the material. The following general guidelines apply to the storage of standards and reagents.

12.12.2 The recommended storage conditions are included in the chemical inventory of LIMS when adding information pertaining to new standards and reagents.

12.12.3 Each department maintains storage locations for standards, reagents, working solutions, and samples. Department supervisors ensure that all chemicals are properly kept. Department supervisors periodically audit storage areas for possible hazards and violations.

12.12.4 Samples are never stored in the same location as standards or reagents.

12.12.5 The following major categories of chemicals, compressed gases, and samples determine standard and reagent storage conditions in the laboratory:

12.12.5.1 Flammables

12.12.5.2 Oxidizer

12.12.5.3 Acids

12.12.5.4 Bases

12.12.5.5 Compressed flammable gas cylinders

12.12.5.6 Compressed non-flammable gas cylinders

12.12.5.7 VOC Samples

12.12.5.8 Inorganic and SVOC Samples

12.12.6 The certificate of analysis or Material Safety Data Sheet provides relevant information regarding recommended storage conditions for all standards and reagents.

12.13 Handling Standards and Reagents

12.13.1 Safety glasses and latex type gloves must be worn at all times when handling chemicals, samples, standards or reagents. A lab coat is also highly recommended. Closed-toe shoes and clothing that cover the legs (no shorts or dresses) must be worn whenever an analyst is working in the laboratory.

12.13.2 The toxicity or carcinogenicity of each reagent used in the laboratory has not been fully established. Each chemical should be regarded as a health hazard and exposure to it should be kept as low as reasonably possible. All health and safety concerns for these and any other

chemicals are listed in the Material Safety Data Sheets (MSDS) provided by the supplier or manufacturer of these chemicals. A copy of any MSDS is available for review at any time in notebooks maintained in the Sample Receiving Department

12.13.3 Proper disposal of all wastes is essential. Containers are provided for all waste according to the type. Follow the waste disposal guidelines found in Section 17.0 for disposing of chemicals.

12.14 Record Keeping Definitions

12.14.1 Prep Log: A prep log is defined as a log of the preparation process that is applied to samples before they are analyzed. This log includes initial volume/weight, final volume, date prepped, batch number, spike amount, all spike information and any comments pertaining to the sample preparation.

12.14.2 Back Log Report: A backlog report is defined as a list of all the samples that need to be analyzed for a specific department. This list is generated from the LIMS system. The list is used by each department manager to create a batch for analysis.

12.14.3 Extraction or Digestion Log: An extraction or digestion log is defined as a log of samples that are either extracted or digested for subsequent analysis. This log includes initial volume/weight, final volume, date prepped, batch number, spike amount, all spike information and any comments pertaining to the sample preparation.

12.15 Procedures for Record Keeping

12.15.1 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data. Each department has a different system for storing data:

12.15.1.1 Metals Department: All raw data (including Quality Control information), summary data, copies of prep logs and back log reports for each batch are placed in a folder with the batch ID number on the folder. Checklists for prep and analysis are placed in the front of the folder. Any non-conformances for the batch are placed in the folder. These folders are stored in numerical order, according to the batch ID, in the metals department. In addition, the electronic data associated with each instrument is downloaded into a separate file and saved periodically onto a CD-ROM to be stored off site.

12.15.1.2 Wet Chemistry: All raw data, prep information and QC data is recorded in logbooks for most analyses. If samples are analyzed on the Lachat, ion chromatograph or TOC analyzer, the raw data (including Quality Control information) and the prep log (where applicable) for each batch are placed in a folder with the batch ID number on the folder. Any non-conformances for the batch are placed in the folder. These folders are stored in numerical order, according to the batch ID, in the wet chemistry manager's office. In addition, the electronic data associated with each instrument is downloaded into a separate file and saved periodically onto a CD-ROM to be stored off site.

12.15.1.3 Organics department: All QC data and copies of the extraction logs for each batch are placed in a folder with the batch ID number on the folder. In addition, any non-conformances for the batch are placed in the folder. These folders are stored in numerical order, according to the batch ID, in the organic manager's office. All organic raw data is given to the project manager and kept with the client's folder.

In addition, the electronic data associated with each instrument is downloaded into a separate file and saved periodically onto a CD-ROM to be stored off site.

12.15.1.4 Project Management: Each project manager has a folder with the COC, sample receipt checklist (SRCL) and organic data (if analyzed for organics) in their office until the project is completed. Once the project is completed, the report and invoice are printed, along with a cover letter and case narrative (if necessary). If everything is correct, the project is reported to the client. A copy of the COC, SRCL, invoice and report are printed and placed in the folder before the report is mailed. If the raw data is to be sent to the client, a copy of the data is printed and placed in the folder before it is sent out. Once the project has been mailed, it is filed in numerical order in the file storage room. Reports are kept for five years.

12.15.1.5 LIMS System: The LIMS system holds all the information relevant to each project that is received at the laboratory, including all client information, and prep and analysis information for each test preformed. LIMS data is backed up daily onto CDs. Copies are stored both on and off site.

12.15.1.6 Entries in manually recorded records are not obliterated by methods such as erasures, overwriting, whiteout or markings. All corrections to record-keeping errors are made by one line marked through the error. The individual making the correction initials and dates the correction.

12.15.1.7 Corrections to electronic records are made by a manual notation that indicates the change to the record. This notation is kept with the affected record.

12.16 Record Storage

12.16.1 All records for each project that is received at the laboratory must be held for a minimum of five years (also, now 5 years for lead analysis records per AIHA). Once the file has been reported and moved to the file storage area, it can only be retrieved again through an access log. This log is located in the sample-receiving department.

12.16.2 Hard copies of records are stored and filed numerically, alphabetically or chronologically by date or batch as appropriate for the type of record. Periodically, all records are transferred to storage boxes that are labeled with the month(s) and year(s) in which the records were generated. Each box is given a unique number and entered into an archive log that includes a description of the contents of each box and the box location. The archived boxes are stored on-site for approximately one year and then transferred to an off-site storage facility. Boxes are stored in such a way to allow easy retrieval of records upon request. Final reports are also maintained electronically on computer hard drives and daily back-up tapes.

12.16.3 Electronic records are stored by department on the laboratory's portal server after scanning or converting the documentation to a PDF file format using Adobe Acrobat®. Customer Service stores the client reports by workorder number. Laboratory data is downloaded and stored by department (Asbestos, Inorganic Chemistry, Metals, Microbiology, Sample Prep, Semi-Volatile Organics, Volatile Organics, and Wet Chemistry). Data contained in the Laboratory Information Management System (LIMS) and on other servers is backed up daily onto CDs.

12.16.4 Archive areas are protected against fire, theft, loss, environmental deterioration and vermin. Electronic records are also protected from electronic or magnetic sources. Archive areas are regularly inspected as part of the Internal Audit program. Representatives of an accrediting authority may have access to archived information.

12.16.5 In the event that AES, Inc. transfers ownership, the new proprietors retain sole custody and responsibility for all records. If AES were to close, records shall be maintained at a commercial archive facility or maintained by another laboratory within the network. Records may also be transferred back to clients, if requested.

12.17 Quality Assurance Records

Where necessary, records are generated and maintained for all quality associated activities conducted during all phases of the analytical work. QA records provide sufficient evidence that all specified QA requirements have been accomplished and satisfied and provide sufficient documentation to substantiate all reported findings and conclusions. These records are retained by AES, Inc. after the initial issuance of the report for a minimum of five years in accordance with AIHA and TNI requirements. This ensures the availability of the QA historical information. The following types of records shall be identifiable and retrievable:

12.17.1 General QA Records – Records pertaining to procurement activities; results of reviews & audits; qualifications of personnel; Standard Operating Procedures and Document Control Records.

12.17.2 Inspection and Test Data Records – Records pertaining to in-process inspection and tests, Equipment Logs and Maintenance Logbooks.

12.17.3 Generated raw data, reports, etc.

13.0 CORRECTIVE ACTION AND NON-CONFORMANCES

Deficiencies or non-conformances in analytical procedures, materials, components or methodology may lead to the release of incorrect analytical results to the customer. Once a deficiency or non-conformance has been identified, corrective actions must be implemented to insure proper data qualification and narration on the final client report and, when possible, prevent the deficiency being repeated. To document and track the non-conformance, a Corrective Action Report (CAR) is issued through the LIMS system. An example of a Corrective Action Report is contained in Appendix VII.

13.1 Standard Procedure for Defining, Implementing, and Closing a Corrective Action Report (CAR).

13.1.1 Non-conformance: A non-conformance is defined as any situation that is either outside acceptable limits (data) or does not comply with the procedure/method in some way (preservation, matrix, etc.). The following are examples of situations considered non-conformances for which the completion of a CAR report is required.

13.1.1.1 Contamination in the Blank: The presence of target analytes in the blank that are above the reporting limit or in some cases, the MDL.

13.1.1.2 Failing Laboratory Control Spike (LCS): When the percent recoveries of target analytes in an LCS fail to meet the acceptable limits for an analysis.

13.1.1.3 Failing Matrix Spike (MS): When the percent recovery of a target analyte in a MS fails to meet the acceptable limits of analysis.

13.1.1.4 Failing Duplicate: When the relative percent difference (RPD) of results between two aliquots of the sample exceed the maximum allowable RPD.

13.1.1.5 Improper sample preservation: When a sample does not have the correct preservation (usually this involves temperature or pH).

13.1.1.6 Exceeding EPA recommended holding time: When a sample is prepared (extracted or digested) and or analyzed after holding time has expired.

13.1.1.7 Sample integrity has been compromised: When a sample container is broken, is improperly sealed, is inappropriate for the analysis, or has headspace (volatiles).

13.1.1.8 Surrogates/Internal standards fail (organic analysis): When a surrogate(s) or internal standard fails to meet the acceptable quality control limits associated with the test method.

13.1.1.9 Dilution test (metals analysis): When the sample dilution test fails to meet the acceptable quality control limits associated with the test method.

13.1.1.10 Failure to meet batch requirements (insufficient sample volume for MS/MSD, etc.)

13.1.1.11 Poor chromatography or missing analytes.

13.1.1.12 Expired standards and reagents.

13.1.1.13 Failed Proficiency Test (PT) analyte.

13.1.2 Procedure for the issuing, completing, and closing of an analytical or technical related CAR.

13.1.2.1 When a non-conformance occurs, the employee performing the work, the initial data reviewer, or the Department Manager must issue a CAR in the LIMS system as indicated below.

13.1.2.1.1 From the "**Categories**" menu select "**Quality Control**". Then from the "**Options**" menu select "**Corrective Action Reports**".

13.1.2.1.2 Click "**Add**" and the LIMS will create a new CAR and automatically number it. Fill in the fields for "**Department**", "**Instrument ID**", "**Batch ID**", "**Initiated By**" and "**Initiated On**" as appropriate.

13.1.2.1.3 Fill in the "**Summary**" field with a brief description of the non-conformance.

13.1.2.1.4 Fill in the "**Complete Description of Non-conformance**" field with a detailed description of the non-conformance including batch numbers, affected samples by number, recoveries and control limits if applicable, etc.

13.1.2.1.5 The complete data file or log book is then forwarded to the Dept. Manager for review. This file must include raw data, prep information, review checklists, etc. and a reference to the CAR by number.

13.1.2.1.6 The Dept. Manager brings the Corrective Action Report to the Laboratory Manager, who determines whether the non-conformance is a "**deficiency**"

or "**anomaly**". An anomaly is an occurrence that affects only the group of data in the associated batch or sequence. Human errors or mistakes are usually anomalies. A deficiency is an occurrence that is system related and may affect more than the batch and may require more extensive corrective actions which could include retraining, replacing equipment, revising SOPs, etc. If the CAR is anomaly, the Department Manager is instructed to document required corrective action in the "**Corrective Action Required**" field. If the CAR is an anomaly, the QA Manager or Technical Director then reviews the data and documents the required corrective action in the "**Corrective Action Required**" field.

13.1.2.1.7 These corrective actions may include narrating the non-conformance to the affected jobs, sending affected samples to be re-prepped and/or reanalyzed, performing instrument maintenance, etc. Common non-conformances and their suggested corrective actions are described in Table 5-6. Non-conformances may also be referred directly to the QA Dept. for more extensive action if necessary. The person filling in the "**Corrective Action Required**" field then fills in the "**Completed By**" and "**Date**" fields.

13.1.2.1.8 If the non-conformance is determined to be an anomaly, the Dept. Manager completes the "**CAR Closed By**" and "**Date**" fields at the end of the CAR form.

13.1.2.1.9 If the non-conformance is determined to be a deficiency, full QA review and documented corrective action to prevent re-occurrence is required. A root cause will be identified for deficiencies. Root causes will be categorized as one of the following: personnel, (LIMS) database, Quality Control, procedure, or laboratory controls.

13.1.2.1.9.1 Personnel: Root causes attributed to personnel may require training or retraining to insure individuals understand their responsibilities in the process.

13.1.2.1.9.2 Database: A Root cause from a database issue primarily refers to the Laboratory Information Management System (LIMS). This type of nonconformance will require the database to be updated. This may include method information (test codes), client information, project information, login entries, calculations, audit trail, and reports among others. Database root cause will also include individual instrument databases and software (GCs, ICPs, AA, Lachat autoanalyzers, etc.)

13.1.2.1.9.3 Quality Control: QC root causes result from incorrect QC acceptance ranges in logbooks, LIMS or are the result of trend changes. These will be reviewed and updated as necessary.

13.1.2.1.9.4 Procedure: This root cause covers procedures, policies, checklists, standard operating procedures (SOPs) that will be reviewed for modifications.

13.1.2.1.9.5 Laboratory Controls: Root causes from instrumentation, software and equipment will be investigated. These may require maintenance, repair, or updates.

13.1.2.1.9.6 A deficiency may require stopping analysis for the procedures affected, notifying clients when previous data may have been affected or other significant corrective actions.

13.1.2.1.10 Once the required corrective actions associated for a deficiency have been completed, fully documented and systems deemed back in control the QA Dept or Technical Director will close the CAR and affected procedure may again be used. The CAR is then printed out, signed by the Technical Director or QA Manager, placed with the data and scanned and posted to the portal server.

13.1.2.1.11 The Technical Director, QA Manager, or any employee may determine that a potential nonconformance requires a preventive action report. Preventive actions are potential sources of nonconformance and needed improvements. "Preventive Action Report" can be initiated by an employee from the results of employee suggestions, data review, audits, etc. and then reviewed by the Technical Director or the QA Manager. Preventive actions are incorporated in the corrective action template (due to software limitations). When the corrective action template is to be used for a preventive action report, the phrase 'PREVENTIVE ACTION' is typed in the **"QA Action"** field. This distinguishes a preventive action template from a corrective action template. Where appropriate, action plans shall be developed; implemented and monitored that will reduce the likelihood of nonconformance. Action plans shall include the application of controls to ensure that actions taken are effective, and may involve the analysis of data, additional auditing, control charts and trends, additional proficiency or QC testing, and issuance of correspondence to clients.

13.1.2.2 The CAR must be prepared at the time the analytical batch has been calculated. Do not wait until all data from the batch is completed. This will lead to unnecessary delay in reprocessing the batch (if necessary) and informing laboratory management, project management, and the client.

13.1.2.3 When completing a CAR, include all accompanying data, information, etc in a "Data Package" along with the NCR and submit this to the Technical Director or Quality Assurance Manager for review. Data packages include the following information.

- Digestion or extraction bench sheets
- ICP and other instrument data such as LACHAT printouts
- All chromatograms within the analytical batch including CCVs
- GC/MS tune criteria
- Analytical "run logs"
- MB, LCS, MS, CCV, post dilution spikes, etc which clearly indicate the

results and or percent recoveries (where applicable).

- Any other test specific quality control criteria such as surrogate recoveries and method of additions results.

13.2 General Procedures and Responsibilities for Corrective Action Reports Involving Deficiencies.

13.2.1 When the QA Dept. or Technical Director issues a corrective action report (CAR) for a non-conformance classed as a deficiency, the Laboratory Manager, Assistant Laboratory Manager, or Technical Director will be informed immediately.

13.2.2 The QA Manager will track the completion of the corrective actions required to correct the deficiency. The assigned personnel are responsible for completing the corrective action within the specified time frame.

13.2.3 The chain of custody and the Sample Receipt Forms are used to document non-conformance during log-in.

13.3 Method Suspension or Restriction

13.3.1 In some cases, it may be necessary to suspend or restrict the use of a method that constitutes significant risk and or liability to AES. Suspension or restriction procedures can be initiated by the Quality Assurance Manager, Technical Director, Laboratory Manager, or VP of Operations.

13.3.1.1 Prior to suspension or restriction, confidentiality is respected, the problem and the required corrective action is stated in writing on the associated CAR and presented to the Laboratory Manager.

13.3.1.2 The Laboratory Manager, Technical Director, Quality Assurance Manager, and the affected supervisor are notified.

13.3.1.3 The Laboratory Manager arranges for the appropriate operations people to speak with the Quality Assurance Manager or Technical Director the day of notification. This meeting is held to confirm that there is a problem, and that suspension or restriction of the method is required.

13.3.2 The suspension or restriction meeting will conclude with a discussion of the steps necessary to bring the method or test fully back on line if the method is suspended or restricted. The Quality Assurance Manager will also specify any documentation necessary to verify that corrective action has occurred.

13.3.3 After suspension or restriction, the laboratory will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. It is the responsibility of the Laboratory Manager to hold all reports. Clients will not generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

13.3.4 Upon completion of the required corrective actions per the CAR, laboratory management will determine if the affected systems are back in control and reports can be released. If systems are still deemed out of control, further corrective actions are required. A team, with all principals involved can devise a start up plan to cover all steps from client notification through compliance of method and release of reports.

13.3.5 If the QA Dept. or Technical Director recommends client notification regarding affects on past or current data quality, all associated information is forwarded to the Laboratory Manager and VP of Operations. These will review the data and determine appropriate actions.

13.3.6 Client notifications are the responsibility of the Laboratory Manager and VP of Operations.

13.4 Procedure for the issuing, completing, and closing of a Project Management or Customer Service related CAR.

13.4.1 CAR should be opened for the following reasons:

a) Any client complaints regarding prices, customer and laboratory service provided, courier service, bottle orders, shipping, invoices, analyses, additional requests after reports have been issued and files archived, etc.

b) Any situation that might have occurred within the laboratory such as results not reported on time, missing information (i.e. reporting limits, analysis dates and times, missing samples, missing analytes, etc.).

13.4.2 CAR must be generated through LIMS as follows:

a) From Main Menu go to "Quality Assurance"
b) Select Corrective Action Reports
c) Click "Add" and number will be automatically assigned through the LIMS
d) Fill in the "Summary" field by writing short description of the CAR
e) Fill in the "Initiate By" and "Initiated On" fields
f) Write a complete and thorough description of the Nonconformance in the "Complete Description of the Non-Conformance" field. For all CARs details must include: client's name, job#, time and name of the person spoken to; if CAR is related to the bottle order or quote, please make sure to include bottle order or quote number. If a credit needs to be issued please make sure to include explanation why, prices used, new prices and documentation supporting new prices, such as quotes, previous invoice, etc.

13.4.3 Once the CAR number is assigned, this number must be entered in the comment section of LIMS under Workorder / Workorders associated with the CAR.

13.4.4 Every CAR must contain supporting documentation. This documentation must be present for the CAR to be closed. CARs that are missing information or details will be returned to the PM. Complete CARs must be forwarded to the Director of Project Management or Laboratory Manager if Director of Project Management is absent.

13.4.4.1 Examples of supporting documentation are as follows:

13.4.4.1.1 In case of NCR about incorrect prices or invoice please make sure to provide following info: old invoice; COCR, copy of COC, price quote. If invoice is being changed in the LIMS system please make sure to issue revision note on the cover letter. We are required by TNI and AIHA to document any changes that were made after final copy of the report is mailed to the client. This cover letter is for in-house purposes only unless requested by client. All revised documents must be given to receptionist for rescanning.

13.4.4.1.2 In case of NCR about bottle order or shipping please provide a copy of the bottle order, tracking number and any other documentation that will support the NCR, such as client's fax, etc.

13.4.5 After all the facts and documents are gathered and submitted to Director of Project Management, a review of the CAR will result in the appropriate corrective action to be described in the "Description of Corrective Action" section of the CAR. This action must be implemented immediately and initialed by every person involved with its completion. Finally, the CAR and associated documentation will be returned to the Director of Project Management or Laboratory Manager for scanning into the archived files.

13.5 Exceptionally Permitted Departures from Documented Policies and Procedures

13.5.1 Due to the frequently unique nature of environmental samples, it may be necessary to depart from documented policies and procedures when dealing with the sample(s). When the analyst encounters this type of situation, he presents the problem to his supervisor for advice. The supervisor may elect to discuss it with the Technical Director or have a technical representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst notes it in the raw data folder. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

13.6 Addressing Complaints

13.6.1 Addressing complaints is a normal function of conducting business and a valuable tool to improve services to and relationships with clients. The goal of AES is to provide expeditious resolution of complaints. At AES, the supervisor and the management team handle complaints related to sample results. Client Services resolves specific complaints concerning container orders, shipping, expected report dates, and results. This information is documented in LIMS. The procedure used for addressing complaints follows the Corrective Action Report.

13.6.2 In the event that a complaint is related to the laboratory's compliance with its own policies and procedures, the rules of an accrediting agency, or the validity of data, the Quality Assurance Manager and or Technical Director initiate an internal audit of the areas involved. These personnel document the complaint, audit findings and recommendations.

13.7 Immediate and Long Term Corrective Action

Immediate corrective actions are necessary to correct or repair non-conforming equipment and systems. This type of corrective action is usually identified by the section supervisor through the use of calibration checks and QC sample analysis.

13.7.1 Long term corrective actions are necessary to eliminate causes of non-conformance. The need for such actions may be identified by audits. Examples of this type of action include:

13.7.1.1 Staff training in technical skills or in implementing the quality assurance program.

13.7.1.2 Rescheduling of laboratory routines to ensure analyses are performed within holding times.

13.7.1.3 Identifying vendors to supply reagents of sufficient purity.

13.7.1.4 Revision of quality assurance system or replacement of personnel.

13.7.2 Various auditing authorities may also initiate a corrective action, when deemed necessary.

13.7.3 For either immediate or long term corrective actions, the steps comprising a closed loop corrective action system are as follows:

13.7.3.1 Define the problem.

13.7.3.2 Assign responsibility for investigating the problem.

13.7.3.3 Investigate and determine the cause of the problem.

13.7.3.4 Determine a corrective action plan to eliminate the problem.

13.7.3.5 Assign and accept responsibility for implementing the corrective action.

13.7.3.6 Establish effectiveness of the corrective action and implement the correction.

13.7.3.7 Verify that the corrective action has eliminated the problem.

13.8 Responsibility for Document Control

The QA department is responsible for document control for the laboratory. Critical documents include the QA Manual, the SOPs, the Corrective Action forms and reports, internally used forms and information, the training files, the MDL studies, the retention time studies, safety training files, performance evaluation reports, certification correspondence and manuals, audit reports and responses, and traceability certificates.

14.0 PERFORMANCE AND SYSTEM AUDITS

14.1 Purpose

The purpose of conducting audits is to monitor and verify compliance and overall effectiveness of the QA Program. Communication of audit findings to management is required for timely consideration and implementation of corrective actions.

14.2 External Audits

14.2.1 External audits are performed when certifying agencies or clients submit samples for analysis and or conduct on-site inspections. It is AES' policy to cooperate fully with certifying agencies. It is also AES' policy to comply fully with system audits conducted by regulatory agencies and clients.

14.2.2 The laboratory is involved in external performance audits conducted semi-annually through the analysis of Performance Testing (PT) samples provided by a third party. EPA performance testing studies have been referred to as Water Pollution Study (WP) and Water Supply Study (WS). Additional PTs including soil studies are analyzed per the requirements of TNI and AIHA.

14.2.3 During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment".

When information is claimed as business confidential, the laboratory must place on, or attach to, the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend, or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents must always be clearly identified. Confidential business considerations may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. Sample identifiers may not be obscured from the information.

14.3 System Audits

14.3.1 It is the responsibility of the Quality Assurance Manager to plan and organize audits as required by a predetermined schedule and as requested by management. Such audits are carried out by trained and qualified personnel who are, whenever resources permit, independent of the activity to be audited. Personnel may not audit their own activities except when it can be demonstrated that an effective audit will be carried out.

System audits are split into smaller audits that are performed within the calendar year at the specified frequency. Audits are performed monthly, quarterly and by the Quality Assurance Manager, the Quality Assurance Officer or an appointed representative. Internal audits can be categorized into three types: annual audit, quarterly audit and monthly audit. During the annual internal audit, compliance with the ISO standard and AIHA requirements will be verified using the current ISO Guide and AIHA Site Assessment checklist. An example audit checklist can be found in Figure 14-1. Additional audits may be necessary throughout the year to address specific project requirements or issues that arise from other audits. Findings of all audits are presented in management reports.

14.3.2 Routine report audits are the responsibility of the laboratory Quality Assurance Manager. The Quality Assurance Manager performs an independent systems review of reports generated by the laboratory. Comments from this review are recorded on Figure 14-1.

14.3.2.1 The reviewer is not expected to pursue the correctness of every reference in the file contents, but instead concentrates on the internal consistency of the data package.

14.3.2.2 Areas that are reviewed include the chain-of-custody, correspondence with the analytical request, batch QC status, completeness of any corrective action statements, calculations, format, holding time, sensibility and completeness of the project, and file contents. A list of reports reviewed is maintained in an audit file.

14.3.3 Internal audits are planned and conducted in accordance with a schedule developed by the QA Manager. Unscheduled audits or surveillance are also conducted when the QA Manager or the Vice-President of Operations deems it necessary.

14.3.4 The responsible management personnel are required to make all personnel, records, reports and documents available to the audit team.

14.3.5 Responsible management of the areas audited is required to provide prompt corrective action in accordance with the provisions of this manual.

14.3.6 Follow-up audits or surveillance is performed, as required, to verify the implementation of corrective action.

14.3.7 When the required corrective action is not implemented within the specified time period, the QA Manager notifies the Vice-President of Operations. A Corrective Action Notice form is used for this purpose. The Vice-President of Operations performs any required corrective actions.

14.3.8 Audit planning and findings are recorded and filed as part of the QA records.

14.3.9 At the discretion of the Vice-President of Operations, impacted clients are notified in writing if the audit result findings indicate any reported data has been compromised.

14.4 Blind Sample Audits

14.4.1 Blind sample audits are performed through the submittal of QC samples to the analyst along with the sample true values, which are only made known to the analyst after the test is complete. Blind sample audits are carried out by the Quality Assurance Manager, Technical Director, clients and certifying agencies as necessary to assure the laboratory is capable of achieving success with a blind QC sample. For continuing TNI and AIHA accreditation, the laboratory must, on a continuous basis, successfully complete two of the last three consecutive proficiency rounds for a given PT field of testing.

14.4.2 In addition to the PT samples submitted to the laboratory through third party vendors, the laboratory may also participate in a company-wide internal PT program to evaluate methods that are not commonly included in the semi-annual PT studies. These studies usually occur between January and February and more frequently if deemed necessary.

14.4.3 It is recognized that PT samples are often not representative of "real world" samples either in their form (e.g., vials), content (e.g., multiple target analyte hits), or documentation (e.g., no chain of custody) and, as such, present the laboratory with special challenges.

14.4.4 It is the policy of AES that PT samples are treated as typical samples in the normal production process wherever possible. Further, if PT samples present special or unique problems in the normal production process, then they should be treated differently, as would any special or unique request submitted by any client. Holding time begins when the vial is opened. Full volume PT samples follow normal holding time procedures and storage requirements.

14.4.5 Login obtains the normal COC information from the documentation provided with the PT samples with review by QA or other designated staff.

14.4.6 Vials are prepared as required in the instruction set provided with the samples. After preparation to full volume, the samples may be spiked, digested, and or concentrated as necessary in a manner similar to normal samples received at the laboratory.

14.4.7 In special cases, the following procedures may be required for the analysis and reporting of PT samples.

14.7.7.1 PT samples will not undergo multiple preparations, multiple runs, multiple methods (unless they are being used to evaluate multiple methods), or multiple dilutions, unless these are the procedures that are normally applied to typical client samples.

14.7.7.2 PT sample(s) will not be subjected to special reviews by operational staff or QA unless this would be normal laboratory practice. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory would apply special review procedures as would be performed for any client requesting unusual reporting or login processes.

14.4.8 Special QC samples can be included in any analytical run.

14.5 Quality Systems and LIMS Management Review

14.5.1 The Laboratory Manager, Quality Assurance Manager, and Technical Director conduct an annual review of the laboratory's quality systems and LIMS to ensure their continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements.

14.5.2 The quality systems and LIMS management review uses information generated during the preceding year to assess the total laboratory and ensures that routine quality actions taken and reviewed on a quarterly basis are not components of larger systematic concerns. The quarterly review (see section 15) is designed to keep the quality systems current and effective.

14.5.3 Significant issues from the following documentation are summarized by the Quality Assurance Manager prior to the review meeting:

14.5.3.1 Matters arising from the previous annual review.

14.5.3.2 Prior Quarterly Quality Assurance Reports.

14.5.3.3 Review of report reissue requests.

14.5.3.4 Minutes from prior management and staff meetings

14.5.3.5 Minutes from prior senior management meetings that discuss adequacy of staff, equipment and facility resources.

14.5.3.6 Prior customer service or business development meeting information.

14.5.3.7 Internal and external audits, including, but not limited to LIMS audits performed during the past year.

14.5.4 The annual review can occur anytime during the year. Based upon an annual review, a report is generated by the Quality Assurance Manager. This report includes the following information.

14.5.4.1 The date of the review and the names and titles of participants.

14.5.4.2 References to the existing documents and topics that were covered in the review process.

14.5.4.3 Quality system or LIMS changes or improvements that will be made as a result of the review.

14.5.4.4 An implementation schedule including assigned responsibilities for the changes.

14.5.5 Following any review, the Quality Assurance Manual or SOPs may be revised to reflect any significant changes made to the quality systems.

14.6 Corrective Action

14.6.1 All deficiencies found during audits are reported to the Laboratory Manager, Quality Assurance Manager, and the Technical Director (see Section 15, "Quality Assurance Reports to Management"). The Laboratory Manager, Technical Director, and Quality Assurance Manager agree upon a time frame for correction. The laboratory's response and corrective action procedures are evaluated by the Quality Assurance Manager and when acceptable, are attached to each audit and filed. If issues arise that may require method suspension or restriction, the procedures outlined in Section 13, "Corrective Action," are followed.

14.6.2 External audits often require written reports that include proof of correction. The Quality Assurance Manager coordinates the written response to the external auditing facility.

14.6.3 Written responses to PT results are required. The response must address the reason for any unacceptable or "Check for Error" result. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

14.6.4 Whenever a laboratory fails a study, it shall determine the root cause for the failure and take any necessary corrective action. If a laboratory fails two out of the three most recent studies for a given PT field of testing, its performance is considered unacceptable under the TNI and AIHA standards for that field. The laboratory shall then need to meet the requirements of initial accreditation. For initial studies, the PT samples shall be analyzed at least 15 days apart. The laboratory must successfully complete two PT studies out of the most recent three rounds attempted for each requested PT field of testing. If analytes are on the Experimental Fields of Testing, participation is mandatory but passing the PT studies is not.

Figure 14-1
Internal Audit Checklist (Annual)

Month: **Year:**

Balances - Annual Maintenance	**Performed (√)**	**Date**	**Certificate Posted**	**Comments**
#2 (AES #1091)	____	_____	____	
#3 (AES #1090)	____	_____	____	
#4 (AES #1182)	____	_____	____	
#5 (AES #1089)	____	_____	____	
#6 (AES #1263)	____	_____	____	
#7 (AES #1214)	____	_____	____	
#8 (AES #1213)	____	_____	____	
#9 (AES #1215)	____	_____	____	
#10 (AES #1506)	____	_____	____	
#11 (AES #1635)	____	_____	____	
#12 (AES #1700)	____	_____	____	
#13 (AES #1717)	____	_____	____	
#14 (AES #1841)	____	_____	____	

Annual Incubator Temperature Study	**Performed (√)**	**Date**	**Posted**	**Comments**
	____	_____	____	

Annual Laboratory Water Acceptance Criteria:

Heavy Metals	**Performed (√)**	**Date**	**Passed**	**Posted**	**Comments**
Cd, Cr, Cu, Ni, Pb, Zn	____	_____	Y or N	____	

Heavy Metals Aggregate	**Performed (√)**	**Date**	**Passed**	**Posted**	**Comments**
	____	_____	Y or N	____	

Bacterial Growth Ratio	**Performed (√)**	**Date**	**Passed**	**Posted**	**Comments**
	____	_____	Y or N	____	

Annual Inhibitory Residue Test	**Performed (√)**	**Date**	**Passed**	**Posted**	**Comments**
	____	_____	Y or N	____	

Stage Micrometer Calibration	**Date Last Calibrated**	**Due for Calibration**	**Performed (√)**	**Date**
	4/17/2007	4/17/2014	____	_____

Imhoff Cone (E160.5) Calibration	**Date Last Calibrated**	**Due for Calibration**	**Performed (√)**	**Date**
	_____	_____	____	_____

Computer Audits	**Performed (√)**	**Date**	**Posted**	**Comments**
Software	____	_____	____	
Hardware	____	_____	____	

Quality Control Acceptance Limits	**Performed (√)**	**Date**	**Posted**	**Comments**
Updated	____	_____	____	

QA Manual	**Performed (√)**	**Date**	**Posted**	**Comments**
Reviewed/Revised	____	_____	____	

Verify Compliance	**Performed (√)**	**Date**	**Comments**
ISO 17025 Standard	____	_____	Refer to Current ISO Guide
AIHA Requirements	____	_____	Refer to AIHA Site Assessment Checklist

SOPs Revised See Tech. Mgmt Summary

Annual Training	**Performed (√)**	**Date**	**Posted**	**Comments**
QA Manual	____	_____	____	
Legal & Ethical	____	_____	____	
Temp. Recording	____	_____	____	
Correction Factor	____	_____	____	

Management Report Submitted	**Performed (√)**	**Date**	**Posted**	**Comments**
	____	_____	____	

Subcontractor Info	**Available (√)**	**Date**	**Posted**
Current Certificate	____	_____	____
Current Scope	____	_____	____

NVLAP	**Performed (√)**	**Date**	**Posted**
Annual Bulk (PLM) Audit Checklist: Handbook 150-3	____	_____	____
Annual Airborne (TEM) Audit Checklist: Handbook 150-13	____	_____	____
Annual General Audit Checklist: Handbook 150	____	_____	____
Annual PLM Control Charts	____	_____	____
Annual PLM Point Count Comparison	____	_____	____
Annual Refractive Index Control Charts	____	_____	____
Annual PLM Precision and Accuracy	____	_____	____

Departmental Data Audit	**Performed (√)**	**Work Order #**	**Posted**
Volatiles	____	_____	____
Semi-Volatiles	____	_____	____
Metals	____	_____	____
IC	____	_____	____
Wet Chemistry	____	_____	____
Micro	____	_____	____
Asbestos	____	_____	____

Figure 14-1 (cont.)
Internal Audit Checklist (Quarterly)

Quarter: **Year:**

Balances	**Calibrated Daily (√)**	**Failures Addressed (√)**	**Posted**	**Comments**
#2 (AES #1091)	____	____	____	
#3 (AES #1090) •	____	____	____	**•1.0 g (AES 1962) & 0.002 g (AES 1854) weights used for 1664 O&G / TPH should be checked the 1st day of each month as well as twice daily in the logbooks**
#4 (AES #1182)	____	____	____	
#5 (AES #1089)	____	____	____	
#6 (AES #1942)	____	____	____	
#7 (AES #1214)	____	____	____	
#8 (AES #1213)	____	____	____	
#9 (AES #1215)	____	____	____	
#10 (AES #1506)	____	____	____	
#11(AES #1635)	____	____	____	
#12 (AES #1700)	____	____	____	
#13 (AES #1717)	____	____	____	
#14 (AES #1841)	____	____	____	

Include copy of current Balance Weights Stage Micrometer Log

Weights	**ID**	**Last Calibrated**	**Calib. Due**	**Schedule Calib.**	**Comments**
Primary Calib. Set	1244	10/2/2007	Oct. 2012	Y or N	
Backup 100 g Wt.	1960	10/25/2011	Oct. 2016	Y or N	
Backup 10 g Wt.	1961	10/25/2011	Oct. 2016	Y or N	
Backup 1g Wt.	1962	10/25/2011	Oct. 2016	Y or N	
Backup 1 mg Wt.	1963	10/25/2011	Oct. 2016	Y or N	
Backup 100 mg Wt.	1964	11/9/2011	Nov. 2016	Y or N	
Backup 2 mg Wt.	1855	9/15/2008	Sept. 2013	Y or N	
Primary 2 mg Wt.	1854	9/15/2008	Sept. 2013	Y or N	
Backup 20 mg Wt.	1939	9/30/2010	Sept. 2015	Y or N	Used with Set 1244

Thermometers	**ID**	**Last Calibrated**	**Calib. Due**	**Schedule Calib.**	**Comments**
NIST Traceable	1877	5/15/2009	5/15/2014	Y or N	
Backup NIST Trace.	1884	8/13/2009	8/13/2014	Y or N	.
Backup NIST Trace.	1597	5/21/2009*	5/21/2014	Y or N	*Internally Checked

Include copy of Current Thermometer Log

Equipment Thermometers	**ID**	**Last Calibrated**	**Calib. Due**	**Posted**	**Comments**
Speed Vap III	1604	_____	_____	____	

Pipettors - Copy of Current Pipettor Log

Employee Training Forms	**Performed (√)**	**Posted**	**Comments**
QA Manual SOP Form	____	____	
QA Manual Training Form	____	____	
Legal & Ethical Training Form	____	____	
Employee Signature	____	____	

Posted Method Checklists	**Performed (√)**	**Posted**	**Comments**
Checklists Updates are posted to Portal	____	____	(i.e. Prep Checklists)

Bottle Checks	**Check Performed (√)**	**Lot #'s**	**Posted**	**Comments**
Sterility Check	____	_____	____	
Micro Coliform		_____	____	
		_____	____	
Metals	____	_____	____	
		_____	____	
		_____	____	
TOC - NC	____	_____	____	
		_____	____	
		_____	____	

Glassware pH Check	**Check Performed (√)**	**Posted**	**Comments**
	____	____	

Micro. Materials Checks	**Performed (√)**	**Posted**
Brilliant Green Media Lot	____	____
Dilution Containers Tolerance Check	____	____
EC Media Lot	____	____
EC Media w/MUG Lot	____	____
HACH P/A Broth Lot	____	____
IDEXX Colilert Media Lot	____	____
Lauryl Tryptose Lot	____	____
M-Endo Lot	____	____
M-FC w/Rosalic Acid Lot	____	____
Plated Media Lot and Reagents		
Positive Control / Negative	____	____
Media Check for Materials > 90 Days		
Positive Control / Negative	____	____
SIM Plate Broth Lot	____	____
Tryptic Soy Double Strength Broth Lot	____	____
Tryptic Soy Single Strength Broth Lot	____	____

AIHA IH environmental micro	**Performed (√)**	**Posted**	**Comments**
Positive & Negative Controls			
Direct Exam Blank Tape Slide performed daily	____	____	

AIHA 5% Inter / Intra Analyst Checks:	**5% Inter**	**5% Intra**	**Posted**	
Air - Culturable (MB-15024)	____	____	____	
Bulk - Culturable (MB-15023)	____	____	____	**Certified but don't do**
Surface - Culturable (MB-15023)	____	____	____	**Certified but don't do**
Air-Direct Exam (MB-15019, MB-15022, MB-15028)	____	____	____	
Bulk & Surface - Direct Exam (MB-15020)	____	____	____	

	Performed (√)
PQL Verification Check with each Batch (NIOSH Methods)	____

AIHA Desorption Studies	**New Lot (Y or N)**	**Lot #**	**Study Performed (√)**	**Posted**
N1003	____	_____	____	____
N1501	____	_____	____	____
N5506	____	_____	____	____

AIHA PT Desorption Studies	**Study #**	**Posted**
N1003	____	____
N1501	____	____
N5506	____	____
N1400, N1450, N1457	____	____
N2000	____	____

Microbiology Checks	**Check Performed (√)**	**Post Logsheet (√)**	**Comments**
Coliform Sample Bottle / Dilution Vessel Sterility	____	____	

Membrane Filter Sterility Checks	**Check Performed (√)**	**Lot #'s**	**Posted**	**Comments**
	____	_____	____	
		_____	____	
		_____	____	

Micro Use Test	**Performed (√)**	**Date**	**Passed**	**Posted**	**Comments**
Student t Test	____	_____	Y or N	____	

Mechanical Timers	**Check Performed (√)**	**Posted**	**Comments**
Autoclave	____	____	

TCLP Tumbler	**Check Performed (√)**	**Posted**	**Comments**
Rates Recorded	____	____	

MS - MSD Frequency	**Check Performed (√)**	**Estimated Frequency**	**Comments**
Semi-VOA (Prep)	____	1 per ____	
Metals (Prep)	____	1 per ____	
Volatiles	____	1 per ____	
Wet Chemistry	____	1 per ____	
Method Blank Frequency	**Check Performed (√)**	**Estimated Frequency**	**Comments**
Semi-VOA (Prep)	____	1 per ____	
Metals (Prep)	____	1 per ____	
Volatiles	____	1 per ____	
Wet Chemistry	____	1 per ____	

LCS Frequency	**Check Performed (√)**	**Estimated Frequency**	**Comments**
Semi-VOA (Prep)	____	1 per ____	
Metals (Prep)	____	1 per ____	
Volatiles	____	1 per ____	
Wet Chemistry	____	1 per ____	1 Per batch for TS & TVS

Asbestos Checks	**Last Performed**	**Date Performed**	**Frequency Required**	**Comments**
PLM Refractive Index Liquid Calibration	_____	_____	Semi-Annually	

IDL (Instrument Detection Limit)	**Check Performed (√)**	**Posted**	**Comments**
EPA 6020 (ICP/MS-TJA)	____	____	
EPA 6010 (ICP_PE)	____	____	
EPA 6010 (ICP_Varian)	____	____	
IEC (Interelement Correction)	**Check Performed (√)**	**Posted**	**Comments**
ICP_PE	____	____	

Environmental Checks	**Check Performed (√)**	**Posted**	**Comments**
Lead Wipe	____	____	Posted w/ Health & Safety

Linear Calibration Range	**Check Performed (√)**	**Posted**	**Comments**
LCR for 180.1 (Required Semi-annually)	____	____	Last performed: _____

Other Observations
General — Review of CARs and / or evidence of inappropriate actions related to data integrity. Follow up on QA CARs and Action Plan from Preventive Actions.
_____
_____

Sample Receipt — Check login to confirm pH is recorded in LIMS for samples received the previous day.
_____

Review 1 set of QA'ed data

Dept.	Run ID	Dept.	Run ID	Dept	Run ID
Prep	_____	Semi-Volatiles	_____	Metals	_____
Wet Chemistry	_____	IC	_____	Volatiles	_____
Microbiology	_____				

Spot check of Logbooks. Logbooks are reviewed for completeness by QA Manager after scanning and prior to posting. Also, person scanning signs off on logbook pages verifying logbooks are complete.

Dept.	Logbook ID	Dept.	Logbook ID	Dept	Logbook ID
Prep	_____	Semi-Volatiles	_____	Metals	_____
Wet Chemistry	_____	IC	_____	Volatiles	_____
Microbiology	_____				

Audit Performed by: Date:

Figure 14-1 (cont.)
Internal Audit Checklist (Monthly)

Month: **Year:**

Monthly Checks

Temp. Checks	**ID**	**Recorded**	**Post Logsheet (√)**	**Schedule Service**	**Comments**
Hotblocks					
Prep	1511	Y or N	____	Y or N	
Prep	1512	Y or N	____	Y or N	
Micro	1849	Y or N	____	Y or N	
Prep	1691	Y or N	____	Y or N	
Micro	1692	Y or N	____	Y or N	
Incubators					
Micro	IN#1	Y or N	____	Y or N	
Micro	IN#2	Y or N	____	Y or N	
Micro	IN#3	Y or N	____	Y or N	
Micro	IN#4	Y or N	____	Y or N	
Micro	IN#5	Y or N	____	Y or N	
Micro	IN#6, shelf 1	Y or N	____	Y or N	
Micro	IN#6, shelf 2	Y or N	____	Y or N	
Ovens					
Wet Chem	Oven#1	Y or N	____	Y or N	
Prep	Oven#2	Y or N	____	Y or N	
Refrig/Freezers					
Micro	Sanyo	Y or N	____	Y or N	
Sample Receipt	R-12a	Y or N	____	Y or N	
Sample Receipt	R-12b	Y or N	____	Y or N	
Prep	F-12/R-28	Y or N	____	Y or N	
Prep	R-15	Y or N	____	Y or N	
Prep	R-16	Y or N	____	Y or N	
Semi-Volatiles	F-5*/R-25	Y or N	____	Y or N	
Semi-Volatiles	F-7	Y or N	____	Y or N	
Semi-Volatiles	F-9	Y or N	____	Y or N	
Semi-Volatiles	R-6	Y or N	____	Y or N	
Semi-Volatiles	R-8	Y or N	____	Y or N	
Semi-Volatiles	F-8/R-24	Y or N	____	Y or N	
Wet Chem	BOD-3	Y or N	____	Y or N	
Wet Chem	BOD-4	Y or N	____	Y or N	
Wet Chem	F-3*/R-5	Y or N	____	Y or N	
Wet Chem	R-17	Y or N	____	Y or N	
Wet Chem	R-23	Y or N	____	Y or N	
Volatiles	F-6	Y or N	____	Y or N	
Volatiles	F-11	Y or N	____	Y or N	
Volatiles	R-1	Y or N	____	Y or N	
Volatiles	R-10	Y or N	____	Y or N	
Volatiles	R-19	Y or N	____	Y or N	

* Unit Not in Use

Waterbaths				
Wet Chemistry	WB#1	Y or N	____	Y or N
Wet Chemistry	WB#2	Y or N	____	Y or N
Prep	WB#3	Y or N	____	Y or N
Prep	WB#4	Y or N	____	Y or N
Prep	WB#5	Y or N	____	Y or N
Asbesto				
Asbestos	Ambient	Y or N	____	Y or N

Sonicator Check

Prep	#1	Y or N	____	Y or N
Prep	#2	Y or N	____	Y or N

Microbiology Checks	**Check Performed (√)**	**Post Logsheet (√)**	**Comments**
SM 9223			
Duplicate	____	____	
Positive Control	____	____	
Negative Control	____	____	
SM 9222D			
Duplicate	____	____	
Positive Control	____	____	
Negative Control	____	____	
SM 9222B			
Duplicate	____	____	
Positive Control	____	____	
Negative Control	____	____	
Quanti Try 2000 Sealer Leak Check	____	____	
UV Lamp Cleaning Log	____	____	

Monthly Air Monitoring	**Check Performed (√)**	**Posted**	**Comments**
Micro Air Fungal	____	____	Posted w/ Health & Safety

AIHA Monthly Blind Culture	**Check Performed (√)**	**Posted**
Blind culture from collection Per Analyst	____	____

Asbestos Checks	**Required Frequency**	**Check Performed (√)**	**Posted**
PLM			
Instrument & Material for each microscope (Microscope Alignment Calibration)	Daily	____	
Contamination Control Testing (of instruments, blades, Petri dishes, etc.)	Daily	____	
Blind recounts (5% of daily analyses)		____	
Blank Contamination Control (Fiberglass / Cellulose check)	Weekly	____	

Air Monitoring Check Job/sample #_____	Monthly	____	**with H & S**
Monthly Precision Summaries (for each analyst)	Monthly	____	____
Summary of Monthly Accuracy (for each analyst)	Monthly	____	____

Asbestos Checks	**Required Frequency**	**Check Performed (√)**	**Posted**
TEM			
Monthly Quality Assurance Summary	Monthly	____	____

Daily Water Quality Checks Performed (Located in Logbooks or on Logsheets)

Conductivity	**All <1.0 umhos**	**Schedule Service**	**Posted Logsheets (√)**	**Comments**
Water System #1	Y or N	Y or N	____	
Water System #2	Y or N	Y or N	____	
Water System #3	Y or N	Y or N	____	

Glassware pH	**Checked Daily**	**5.5 - 7.5**	**Posted**	**Comments**
In Micro Logbooks	Y or N	Y or N	____	

Residual Chlorine	**Checked Daily**	**<1.0 mg/L**	**Posted**	**Comments**
In Micro Logbooks	Y or N	Y or N	____	

Monthly Water Quality Checks Performed (Should be submitted on Form)

Parameter	**Performed (√)**	**<1000 CFU/mL**	**Posted**	**Comments**
Heterotrophic Plate Count (HPC)	____	Y or N	____	
	Performed (√)	**<0.1 mg/L**	**Posted**	**Comments**
Ammonia (NH_3)	____	Y or N	____	
	Performed (√)	**<0.1 mg/L**	**Posted**	**Comments**
Organic Nitrogen	____	Y or N	____	
	Performed (√)	**<1.0 mg/L**	**Posted**	**Comments**
Total Organic Carbon (TOC)	____	Y or N	____	
	Performed (√)	**<0.2 mg/L**	**Posted**	**Comments**
Chlorine	____	Y or N	____	

Monthly Metals LCS / LCSD Checks - Since the concentrations in the test codes come from the standard in use, this check verifies that the lot number and concentrations have not changed.

Test Code	**ID / Cert. / Catalogue #**	**Lot #**	**Pb Conc.**	**Units**	**Date**
7420_S (7000B)	_____	_____	_____	____	_____
PAINT_LEAD	_____	_____	_____	____	_____
WIPE_MET_AA (Pb Only)	_____	_____	_____	____	_____

Audit Performed By: Date:

Annual Computer Audit Checklist

Software Audit

LIMS

	Yes	No
1. Are quality control data referenced to sample results? (standards, blanks, calibrations, replicates, duplicates, spikes, instrument conditions, surrogates, internal standards, etc.)		
2. Are references to quality control data protected or can they be easily changed?		
3. Are references sufficient to associate quality data with individual sample results?		
4. Are data outside acceptance criteria flagged?		
5. Are the detection limits for target analytes clearly referenced in the LIMS data?		
6. Are the units correct?		
7. Can the results be traced back to the original data associated with a specific batch?		
8. Are all out of range results either prevented or flagged?		
9. Has security been maintained (old passwords, logons eliminated from the system)?		
10. Are data transfers periodically audited and documented?		

For data linked to an analytical instrument, is the following information available:
(Either in LIMS or with the instrument documentation)

	Yes	No
11. Date and time generated?		
12. Identification of instrument?		
13. QC flags indicating the level of acceptability of the data?		
14. Is there a computer generated record of the changed and unchanged data?		

	Yes	No
15. Are all data quality flags defined? (QA Manual)		
16. Are qualifying flags correct?		
17. Are printouts of report modifications routinely checked for accuracy? By whom: (Project Managers) _____		
18. Are final copies of reports properly archived with limited access, security, and protection against natural disaster (fire, flood, etc.)?		

Documentation

	Yes	No
19. Are there written backup procedure?		
20. Is there a disaster recovery procedure?		
21. Does the software management (LIMS) include validation? Vendor (Khemia)		
22. Have the mathematical calculations validated? How is this documented? Vendor (Khemia)		
23. Are software revisions tested to determine how the entire program is affected?		
24. Is there a logbook to document software revision implementation?		

	Yes	No
25. Is a password required to access the system?		

	Yes	No
26. Is there documented operator training? (Checklist)		

Backups

	Yes	No
27. Are system backups performed?		

What frequency?	Daily	Weekly
LIMS		
AES Servers		
Portal Server		

Who performs backups? (When not Automatically) _____

	Yes	No
28. Are media storing backups properly labeled?		

	Yes	No
29. Is data from backups stored short term? How is it stored? (Network Attached Storage)		

	Yes	No
30. Is data from backups stored long term? How is it stored? (Written to DVDs)		

	Yes	No
31. Is long term backup data stored off site?		

	Yes	No
32. Have report formats that are no longer in use been deleted or inactivated so that they are not mistakenly used?		

	Yes	No
33. Have past employees' names been removed for LIMS pick lists, internal email, and external email? (Checklist)		

Hardware Audit

	Yes	No
1. Are there procedures for performing and documenting preventive maintenance?		
2. Is there regularly scheduled preventive maintenance?		
3. Is preventive maintenance documented?		
4. Is non-routine maintenance performed by in-house staff?		
5. How is it documented? (Logbook)		
6. If the system fails because of electrical glitches or power outage, what happens to the system? (UPS Backup System)		
7. Is a backup power source available?		
8. Are problems documented after a power outage?		

Audit Performed By: Date:

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

15.1 Internal Reports

The Quality Assurance Manager submits quarterly reports regarding the status of QA/QC activities to the Vice-President of Operations. Section 15.3 lists the minimum content of this report. The Quality Assurance Manager also submits an annual report to the Vice-President of Operations.

15.2 External Reports

Certain projects under regulatory review require establishment of explicit quality assurance objectives and quarterly summaries of QA conformance and corrective action. The laboratory technical and quality assurance staffs provide the necessary information required to establish quality assurance objectives for particular projects. Once the QA deliverables options are selected for the project, sufficient quality control data will be provided in the individual analytical report to allow a periodic assessment of the overall progress of the project. Upon request, any information or reports needed are provided by laboratory management with review by the QA Manager.

15.3 Quarterly and Annual Reports

The quarterly or annual report to management includes the following information.

15.3.1 SOP. The report indicates any changes to existing SOPs or any new SOPs.

15.3.2 Corrective action reports. The report contains information about any corrective action reports that may have been written during the time period since the last QA report.

15.3.3 MDL. Any changes in MDL should be included in the QA report.

15.3.4 Audits. The QA report includes the results of any audits performed during the time period since the last report.

15.3.5 PE samples. The report includes the results of PE samples analyzed since the last report. The PE report indicates the status of performance as it relates to current laboratory accreditations.

15.3.6 Certifications. Any changes or additions to the laboratory's certifications are addressed in the reports.

15.3.7 The annual report is reviewed and signed by the Vice President of Operations, Laboratory Manager, and the Technical Director. A copy of this report is kept for 5 years.

16.0 REAGENT STORAGE AND DOCUMENTATION

16.1 Safety and Shelf Life

Reagents are stored with consideration for safety and maximum shelf life. Storage conditions and documentation maintenance status for various classes of reagents are given in Table 16-1 and Table 16-2, and are discussed below.

16.1.1 All acids, except those poured into small marked containers for immediate use and those that are standardized for specific purposes, are stored in the original containers in acid storage cabinets.

16.1.2 All bases, except those poured into small containers for immediate use and those that are standardized for specific purposes, are stored in the original containers within designated areas or storage cabinets.

16.1.3 All flammable solvents, except those poured for immediate use, are stored in original containers in approved, vented, flammable storage cabinets, which are located indoors.

16.1.4 Dry reagents are stored in designated cabinets in cool, dry areas. Reactive chemicals, cyanides and sulfides are labeled and isolated from other chemicals.

16.1.5 All acids used for metal sample digestions and all solvents used for semi-volatile sample extraction may be tested prior to initial use. Lot numbers used for digestions or extractions are recorded in bound notebooks in the appropriate departments.

16.1.6 Reagent blanks are analyzed with each sample batch for all methods, validating the purity of all reagents. All reagent containers are dated when received, and dated and initialed when opened (except high use items consumed in less than one week). Documentation is maintained to provide traceability of the reagents used with the analysis of any batch to specific reagent lot numbers.

TABLE 16-1
STORAGE OF REAGENTS AND CHEMICALS

i. CHEMICAL REQUIREMENTS	STORAGE
ii. Concentrated acids and bases	1
ii) Standards for metals analysis	2
Standards for extractable organics	3
Standards for volatile organics	4
Bulk dry chemicals	5
Working solutions containing organic compounds	6
Working solutions containing only inorganics	7
Flammable solvents	8
Non-flammable solvents	9

Table 16-2
(a) STORAGE REQUIREMENT KEY

1. Stored in the original containers in acid/base cabinets. All organics must be stored separately.
2. Stored at room temperature in the standards cabinet of the metals department.
3. Stored below 0^{o} C in the department.
4. Neat standards are stored at room temperature in the standard cabinet in the department. Stock solutions and working solutions are stored in the freezer.
5. Bulk reagents are stored at room temperature in reagent storage cabinets located throughout the laboratory.
6. Stored refrigerated at 1-4^{o} C in the department.
7. Stored at room temperature in the department; refrigeration is optional.
8. Stored in solvent cabinets in the organic extraction laboratory.
9. Stored separately from the flammable solvents in cabinets in the organic extraction laboratory.

17.0 WASTE DISPOSAL

17.1 AES operates as a conditionally exempt, small quantity generator.

17.2 All waste disposal is carried out in accordance with AES' Waste Disposal SOPs, WM-17001, "Organic Waste Disposal," and WM-17002, "Inorganic Waste Disposal". These documents include procedures for identification, storage, personnel training, tracking forms, report forms and safety, as well as details of the disposal. Hazardous waste disposal procedures are discussed below.

17.3 Hazardous Waste Requirements:

17.3.1 Hazardous waste is stored in non-leaking containers that are in good condition with close-fitting lids. The lids are kept closed when wastes are not being added or removed.

17.3.2 Hazardous waste storage containers are accurately labeled with waterproof labels. The labels specify the words "Hazardous Waste", the composition and physical state of the waste, the hazardous properties of the waste (e.g., flammable, reactive, etc.), and the name and address of the generator.

17.3.3 Each hazardous waste container is clearly labeled with the date that the period of accumulation began. The date is also documented on the Hazardous Waste Tracking Log Form (see Section 17.5.8).

17.3.4 All containers are handled in a way that minimizes the possibility of spills and escape of wastes into the environment.

17.3.5 Wastes are stored in an area that is regularly inspected for deteriorating or leaking containers.

17.3.6 All wastes are segregated during temporary accumulation, storage, and for disposal. Prior to disposal, waste materials are carefully combined into categories or waste streams based upon their compatibility

17.3.7 The following three types of waste are stored in 55-gallon drums.

17.3.7.1 Halogenated solvents such as methylene chloride (closed cap metal drum)

17.3.7.2 Non-halogenated flammable solvents (closed cap metal drum).

17.3.7.3 Heavy metals or other aqueous wastes except cyanide (poly drum)

17.3.8 All other wastes are stored in the original container or 4-liter glass bottles and disposed of via a "lab pack" (i.e., packed by a disposal company in 55-gallon open top drums).

17.4 Sample Disposal (See also AES SOP HS-03005)

17.4.1 After completion of the analysis, unused sample portions, extracts, or digests are transferred to a central secured storage area until they are disposed. Unless a client requests that the project manager save unused samples, digests, or extracts, disposal from the central storage occurs 30 days after submission for test results.

17.4.2 Requests for extended sample, digest or extract storage must be provided by the client to the AES project manager in writing (contract form) prior to sample receipt. Extended storage may

result in the charging of additional fees by the AES project manager prior to sample receipt. AES is not responsible for evaporation or other deterioration of samples, extracts, or digests during extended storage periods.

17.4.3 Clients that desire the return of samples may pick them up at the laboratory, request shipment by Federal Express (at the client's expense for packaged shipping), or utilize any other legal means that they choose. Clients requesting the return of samples should provide detailed shipping instructions.

17.4.4 If a client, by contract, specifies sample disposal by a hazardous waste contractor, the client's name and EPA ID number will be used on the manifest and the client will be invoiced for all disposal-related costs.

17.4.5 Other excess sample portions are composited by the laboratory according to matrix (solids, oils or aqueous). The composited soils, sediments and other solid samples are sub-sampled and analyzed for hazardous waste characterization (ignitability, reactivity, (releasable cyanide and sulfide), corrosivity (pH), toxicity (TCLP by SW-846 Method 1311) and PCBs). If the pooled sub-sample is characterized as hazardous by any of the hazardous waste characteristics or contains greater than 50 ppm PCBs, the excess sample is disposed of through the use of a hazardous waste contractor. If the pooled sub-sample is not deemed hazardous based upon the results of these tests, the composited excess material is disposed of in an industrial/municipal landfill.

17.4.6 Aqueous samples are neutralized and disposed of via the municipal sewer system, following all discharge requirements outlined in 40 CFR Part 261.3 (a)(2)(iv)(E).

17.5 Organic Waste Disposal (See also AES SOP HS-03005)

17.5.1 Similar waste disposal procedures for samples from the volatile, semi-volatile and GC/HPLC pesticide laboratories are employed at AES.

17.5.2 All personnel should be familiar with this SOP prior to the disposal of any wastes within the laboratory.

17.5.3 AES is considered as a Conditionally Exempt, Small Quantity Generator under 40 CFR Part 261.5 (a generator who generates no more than 100 kilograms of hazardous waste or 1 kilogram of acute hazardous waste in a calendar month and accumulates no greater than 1000 kilograms of hazardous waste). Hazardous waste storage is limited to quantity and/or accumulation and must comply with RCRA regulations as specified in 40 CFR. These wastes are packaged and separated according to compatible groups (e.g., solvents, acids, etc.)

17.5.4 The pH of the discharged waste MUST be between 5 and 10. If the pH of the discharged waste is out of this range, it is diluted with water or treated with the appropriate acid or base.

17.5.5 Apparatus and Equipment

17.5.5.1 Respirator and gloves

17.55.2 5-gallon plastic buckets with lids

17.5.6 Reagents and Chemicals.

17.5.6.1 Marble chips for neutralizing acid waste

17.5.7 Procedure

Prior to the disposal of any waste, the Health and Safety Officer provides a sample disposal list to the laboratory employee performing the task. Included in this list is the method of disposal and location of disposal for each sample. The Health and Safety Officer obtains this information from the AES LIMS system and categorizes the samples as hazardous or non-hazardous.

17.5.7.1 The procedure for the collection and disposal of expired organic chemicals and solutions is outlined in the subsequent sections.

17.5.7.1.1 Neat standards are sealed and labeled.

17.5.7.1.2 All stock standards, working standards and unused sample extracts are emptied into a properly labeled (contents are listed using the official waste storage labels) 4-L empty solvent bottle.

17.5.7.1.3 Waste standards or samples containing Silvex (2,4,5-TP), 2,4,5-T, or PCBs are stored separately from other waste standards. These compounds are potential dioxin wastes. All acid herbicide standards or sample waste are stored separately from other standard wastes.

17.5.7.1.4 HPLC/GC vials containing solvents, standards and extracts are stored in a labeled, 4-liter, empty solvent bottle.

17.5.7.1.5 Wastes are never allowed to accumulate in the laboratory for longer than 3 days. Wastes that are stored for longer time periods are stored in the waste storage room located at the back of the laboratory. All dated waste is disposed of in drums.

17.5.7.1.6 Each drum is labeled according to contents, i.e., chlorinated, non-chlorinated solvents, acid and mercury waste. Acid wastes are stored in the acid waste room that is separate from the solvent waste room.

17.5.7.1.7 All wastes are treated inside the fume hood using appropriate safety equipment such as a respirator, gloves, laboratory coat, and safety glasses.

17.5.7.1.8 The Safety Officer is notified in the event of any leaks or spills of hazardous wastes.

17.5.7.1.9 The waste drums available are:

Flammable Waste
Soil Waste
Acid Waste
Methylene Chloride Waste
Neutralized Waste

17.5.7.1.10 Autosampler vials full of sample waste are placed into an empty 4-liter solvent bottle, properly labeled, dated, and stored in waste room, where they are lab-packed.

17.5.7.1.11 High-level organic wastes are treated as hazardous substances and are placed in clearly labeled containers. Full containers are stored in the inorganic waste storage room.

17.5.7.1.12 Containers that have been used for the storage of high level wastes are not reused.

17.5.7.1.13 Soil samples are transferred to 5-gallon buckets. When full, a composite sample is analyzed for TCLP and characterized for disposal through the use of a Hazardous Waste Contractor.

17.5.7.1.14 The contents of used VOC vials are neutralized prior to disposal in the sanitary sewer system.

17.5.7.2 The neutralization of alkaline or acidic wastes is performed with the following procedure.

17.5.7.2.1 A 5-gallon bucket with a strainer bottom is placed directly into a sink.

17.5.7.2.2 The bucket is filled with 6 to 8 inches of marble chips.

17.5.7.2.3 A generous flow of water is allowed to pass through the bucket containing the marble chips.

17.5.7.2.4 The samples are added to the bucket at the same time that the water is flowing allowing the samples to drain through the chips and become neutralized.

17.5.8 The Waste Disposal Logbook is located in close proximity to each drum. The following information is added to the logbook:

AES WORK ORDER Number
Client Sample I.D. Number
Employee(s) Name(s)
Nature of Disposal

17.5.9 The Health and Safety Officer maintains a separate waste disposal record file. These files contain the master list of samples that have been disposed, TCLP analytical results, raw data, and disposal manifest receipts.

17.6 Inorganic Waste Disposal (See also AES SOP HS-03005)

The procedure for the collection and disposal of expired inorganic chemicals and solutions is outlined in the subsequent sections.

17.6.1 AES is considered as a Conditionally Exempt, Small Quantity Generator under 40 CFR Part 261.5 (a generator who generates no more than 100 kilograms of hazardous waste or 1 kilogram of acute hazardous waste in a calendar month and accumulates no greater than 1000 kilograms of

hazardous waste). Hazardous waste storage is limited to quantity and/or accumulation and must comply with RCRA regulations as specified in 40 CFR. These wastes should be packaged and separated according to compatible groups (e.g., solvents, acids, etc.). Waste water containing toxic waste from the laboratory that does not exceed 1% of total waste water flow can be disposed of into the sanitary sewer system as specified in 40 CFR part 261.3E.

17.6.2 The pH of the discharged waste MUST be between 5 and 10. If the pH of the discharged waste is out of this range, it is diluted with water or treated with the appropriate acid or base.

17.6.3 Apparatus and Equipment

17.6.3.1 Large polyethylene drum (5 L - 25 L)

17.6.3.2 Latex gloves

17.6.3.3 Stirring rod (glass or wood)

17.6.3.4 Five gallon plastic bucket with strainer bottom

17.6.4 Reagents and Chemicals

17.6.4.1 Solid sodium bicarbonate ($NaHCO_3$)

17.6.4.2 Marble chips

17.6.5 Procedure

Prior to the disposal of any waste, the Health and Safety Officer provides a sample disposal list to the laboratory employee performing the task. Included in this list is the method of disposal and location of disposal for each sample. The Health and Safety Officer obtains this information from the AES LIMS system and categorizes the samples as hazardous or non-hazardous.

17.6.5.1 Strong acid solutions, such as digestates, are collected in a 25-liter polyethylene drum labeled as “the Water Drum”. When the drum is approximately half full, the drum is wheeled outside the laboratory and the building.

17.6.5.2 Solid sodium bicarbonate is slowly added to the waste solution while it is stirred with a wooden stick. The solution will effervesce, as the bicarbonate neutralizes the acid in the solution. Bicarbonate is added to the solution until the effervescence stops.

17.6.5.3 When the pH of the liquid is between 5 and 10, the liquid is returned the laboratory department and the neutralized waste is disposed of in a sink. The sink is flushed for 5 to 10 minutes.

17.6.5.4 The following procedure is used to dispose of weak acid solutions such as samples with preservatives:

17.6.5.4.1 A 5-gallon bucket with a strainer bottom is placed directly into a sink.

17.6.5.4.2 The bucket is filled with 6 to 8 inches of marble chips.

17.6.5.4.3 The tap water is turned on and allowed to flow through the bucket containing the chips.

17.6.5.4.4 The samples are poured into the bucket while maintaining the water flow.

17.6.5.4.5 The samples are allowed to drain through the water and chips and are neutralized.

17.6.5.5 Samples that have observed concentrations of measured analyte that is above the calibration level of the various instruments are treated as hazardous waste. This includes the sample waste generated from the flame AA or ICP instrument. This waste is collected in a storage bottle and is disposed of as an acidic waste when the bottle is filled.

17.6.5.6 High-level inorganic wastes in organic solvents are treated in the following manner:

17.6.5.6.1 The high-level waste is placed into a clearly labeled container. When the container is full, the container is placed into the waste storage room.

17.6.5.6.2 Containers used for the storage of high-level wastes are not reused.

17.6.6 The Waste Disposal Logbook is located in close proximity to each drum. The following information is added to the logbook:

AES WORK ORDER Number
Client Sample I.D. Number
Employee(s) Name(s)
Nature of Disposal

17.6.7 The Health and Safety Officer maintains a separate waste disposal record file. These files contain the master list of samples that have been disposed, TCLP analytical results, raw data, and disposal manifest receipts.

APPENDIX I

WASTE DISPOSAL PROCEDURES

Waste Type	Associated Analytical and Sample Prep Methods	Storage Procedures	Disposal Procedures
Halogenated Solvents Methylene Chloride	Pesticides, Herbicides, BNA, GPC, etc.	Store in glass bottles, then in drums.**	Reclaimed by HW contractor.
Freon	Oil & Grease, Petroleum Hydrocarbons	Store in glass bottles.	Reclaimed by laboratory.
Mixed Solvents (Flammable & nonhalogenated)	VOC Standards, Herbicides, Pesticides	Store in glass bottles, then in drums.	Disposal by HW contractor.
All neat standards	All analyses	Store in original bottles of glass/plastic bottles, then lab pack.	Disposal by HW contractor (Packed by also)
Heavy Metals Solutions	Metals, COD, Chloride	Store in glass bottles, then in drums.	Disposal by HW contractor.
Acid Solutions	Metals, General Inorganics, Extractions	Store in glass bottles or add to neutralizing chambers.	Neutralize; sanitary sewer.
Alkaline Solutions	General Inorganics, Extractions	Store in glass bottles.	Neutralize; sanitary sewer.
All samples containing Organics or Inorganics exceeding hazardous waste standards*	All analytical groups	Store in original bottles or jars in sample custody storage area.	Return to client or disposal by HW contractor.

* Hazardous Waste Characteristics (D001-D017) (40 CFR Part 261), HCN>250 mg/kg, TCLP Toxicity Characteristics (Federal Register, 55FR 11798), March 29, 1990, or contains greater than 50 ppm PCBs.

** Bottles are kept in each laboratory and are periodically moved to the hazardous waste storage area.

APPENDIX II

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

	Service Interval							
EQUIPMENT ITEM	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE LEVEL**
ICP-AES and ICP-MS								
Pump Tubing				X				Change
Nebulizer			X					Clean
Filters			X				X	Inspect - clean or replace.
Spray Chamber			X					Clean
Quartz Torch					X			Clean and realign.
D-Shaped Mirrors			X				X	Inspect - clean or replace
MERCURY ANALYZER AND AUTOSAMPLER								
Pump Tubing	X						X	Inspect – replace
Standard Cups	X						X	Inspect – replace
Drying Tube	X							Repack
Mixing Coil		X						Inspect - clean or replace
Sample Probe			X					Inspect - clean or replace
Mercury Lamp							X	Clean or replace
CONDUCTIVITY METER								
Battery							X	Check or replace
Probe Contacts							X	Clean or replace
pH METER								
Probe(s)	X							Check fluid levels and fill
Connectors	X							Check for corrosion and clean if necessary
AUTOANALYZER (TRAACS/LACHAT)								
Pump Platen							X	Replace
Pump Tubes				X				Replace
Flow Cell				X				Inspect and clean.
Autosampler	X							Check alignment
Cobalt Column							X	Inspect for channeling and repack
BLOCK DIGESTER								
Heating Elements							X	Replace as needed
Thermostat					X			Check against calibrated thermometer for accuracy
UV/VIS SPECTROPHOTOMETER								
Light Source							X	Replace
Belt	X							Check for wear, replace if frayed
Cuvettes	X						X	Check for scratches and buildup - replace
ION SELECTIVE ELECTRODE								
fluid filled probe	X						X	Check fluid level - empty and replace if crystals form
solid probe	X							Check for salt build-up on tip, clean if necessary
BOMB CALORIMETER								
Thermometer						X		Calibrate Thermometer
Seals	X							Check for breaks in seals and replace if needed
GAS CHROMATOGRAPH – SEMIVOLATILES								
Autosampler System							X	Syringe and tubing cleaned – Needles/ tubing replaced
Septa		X						Replace
Column/Injector							X	Chance sleeve and cut front of guard column.
Gas Cylinder	X							Inspect - change when pressure reads <500 psi.

APPENDIX II

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

	Service Interval							
EQUIPMENT ITEM	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE LEVEL**
GAS CHROMATOGRAPH - MASS SPEC SEMIVOLATILES								
Column/Injector		X						Chance sleeve and cut front of column.
Septum		X						Replace
Splitless Disc					X			Replace
Autosampler	X					X		Syringe and tubing cleaned Needles and tubing replaced
Rough Pump						X		Oil change by HP service
Mass Spectrometer							X	Clean
Gas Cylinder	X							Inspect - Change when pressure reads <500 psi.
Hard Drive		X						Archive
ATOMIC ABSORPTION								
Pump	X							check for leaks and corrosion
Lamps							X	If intensity drops, replace
Nebulizer		X						Clean, sonicate
Tubing	X							If leaking or weak, replace
Burner Head		X						Clean, sonicate
Bottled Gases	X							Replace if pressure reaches 500 psi.
Spray Chamber			X					Clean, sonicate
GAS CHROMATOGRAPH – VOLATILES								
Column							X	Replace
Septum			X					Replace
Gas Cylinder	X							Inspect - change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap						X		Replace
GAS CHROMATOGRAPH - MASS SPEC VOLATILES								
Column							X	Replace
Rough Pump						X		Oil change by HP service
Gas Cylinder	X							Inspect - change when pressure reads <500 psi.
Septum			X					Replace
Transfer Line							X	Check for leaks
GAS CHROMATOGRAPH – ECD								
Autosampler	X					X		Syringe cleaned Needles and tubing replaced
Column							X	Replace
Septa							X	Replace
Glass Insert							X	Replace
Gold Disk							X	Replace
Gas Cylinder	X							Inspect - change when pressure reads <500 psi.
EC Detector(s)						X		Send off for replacement of radioactive nickel foil.
GAS CHROMATOGRAPH – FID								
Autosampler	X						X	Syringe and tubing cleaned Needles and tubing replaced
Column							X	Replace
Septa							X	Replace
Gas Cylinder								Inspect daily, change when pressure reads <500 psi.

APPENDIX II

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

	Service Interval							
EQUIPMENT ITEM	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE LEVEL**
Glow Plug								Determine if glow is enough to ignite Hydrogen
Housing and chimney								Check for rust and corrosion that will cause a short, and clean if necessary.
Glass Insert							X	Replace
Column							X	Replace
PURGE AND TRAP								
Sorbent Trap					X			Change
Heater Pockets	X							Check, replace if defective
Transfer Lines							X	Inspect and replace if needed
Purge Flow					X			Inspect, adjust as needed
TCLP EQUIPMENT								
Volatile Rotator	X							Check rotation (± 30 rpms)
Semi-volatiles/Metals Rotator	X							Check rotation (± 30 rpms)
BALANCES								
Balances	X							Calibrate, service annually
Auto-Pipettors				X				Calibrate
BALANCE WEIGHTS – for daily balance checks								
Set "B" – 10 weights								Verified every 5 years by a body that can prove traceability to NIST
THERMOMETER (CERTIFIED) – for in-house thermometer calibrations								
HB #28199 (CMI #32478) –1 to 200°C							X	Certified every 5 years by a body that can prove traceability to NIST
DISSOLVED OXYGEN METER								
Batteries	X							Check for strength, if < 13.20 replace
Membrane				X				Replace. Sooner if signal will not stabilize
Spill housing and stirrer	X							Clean

The service intervals listed in Appendix II are as follows: D = daily; W = weekly; M = monthly; Q = quarterly; SA = semi-annually; and AN = as needed.

APPENDIX III
LAB EQUIPMENT LIST

ID No.	Instrument	Type	Manufacturer	Model	Serial Number	Age
1000	MS-4	Auto Sampler	Varian	Archon	13405	1999
1001	MS-4	Sample Concentrator	OI Analytical	4560	J448460426	1999
1002	MS-4	GC	HP	6890	430021BJ4	1999
1003	MS-4	MS	HP	5973	US82311468	1999
1004	MS-5	Auto Sampler	Varian	Archon	12110	2001
1005	MS-5	Sample Concentrator	OI Analytical	4560	H413460123	2001
1695	MS-5	Sample Concentrator	OI Analytical	4660	D63646651P	2006
1006	MS-5	GC	Agilent	6850	US00001050	2001
1007	MS-5	MS	Agilent	5973	US94240080	2001
1008	MS-7	Auto Sampler	Varian	Archon	12999	1999
1009	MS-7	Sample Concentrator	OI Analytical	4560	D310211	1999
1838	MS-7	Sample Concentrator	OI Analytical	4660	D807466325P	2008
1010	MS-7	GC	Agilent	6850	US00001051	2001
1011	MS-7	MS	Agilent	5973	US94240092	2001
1012	MS-8	Auto Sampler	Varian	Archon	13322	2001
1013	MS-8	Purge & Trap/Sample Concentrator	Teckmar	3000	98259003	2000
1623	MS-8	Purge & Trap	OI Corporation	Eclipse 4660	D524466126P	2005
1014	MS-8	GC	Agilent	6850	US00001100	2001
1015	MS-8	MS	Agilent	5973	US94240107	2001
1016	HOOD NO#16	Hood	Labconco	47"x31"	N/A	
1017	GC-1	GC	HP	5890SII	3336A52981	1993
1018	GC-1	Auto Sampler	HP	18596B	3106A24283	1993
1019	GC-1	Tower	HP	18593B	3442A40453	1993
1020	GC-2	GC	HP	5890SII	3336A5502	1994
1021	GC-2	Auto Sampler	HP	18596M	3209A27907	1994
1022	GC-2	Tower	HP	18593B	3202A29321	1994
1023	GC-3	GC	HP	5890SII	3140A38355	1995
1024	GC-3	Auto Sampler	HP	18596M	3433A36260	1995
1025	GC-3	Tower	HP	18593B	3341A36564	1995
1026	GC-4	GC	HP	5890SII	302218A29420	1997
1027	GC-4	Autosampler	HP	18596B	3320A32113	1997
1028	GC-4	Tower	HP	18593B	3013A22544	1997
1029	GC-5	GC	HP	5890SII	3140A39201	1998
1030	GC-5	Auto Sampler	HP	18596B	3050A23709	1998
1031	GC-5	Tower	HP	G1513A	US81205611	1998
1643	GC-6	GC	HP	5890SII	3235A46102	1995
1644	GC-6	Auto Sampler Controller	HP	7673 / 18594B	3318A32045	1995
1645	GC-6	Tower	HP	7673 / 18593B	3442A40453	1995
1536	GC-7	Monitor 19"	Agilent	19"	FANU45199020U	2004
1537	GC-7	Computer	Agilent	MXZ3460BJW	MXZ3460BJW	2004
1538	GC-7	GC (ECD)	Agilent	6890N	CN10427041	2004
1539	GC-7	Tower	Agilent	7683	CN42437159	2004
1032	MS-6	GC	HP	6890	US00021363	1999
1033	MS-6	MS	HP	5973	US80310957	1999

1034	MS-6	Auto Sampler	Agilent	G2614A	US00807551	1999
1035	MS-6	Tower	Agilent	G2613A	US00811878	1999
1036	MS-3	GC	HP	5890SII	336A55978	1995
1037	MS-3	MS	HP	5972A	3501A02369	1995
1038	MS-3	Auto Sampler	HP	18596B	3342A33508	1995
1039	MS-3	Tower	HP	18593B	3013A22290	1995
1040	HPLC-1	Degasser	HP	G1322A	JP73017078	1999
1041	HPLC-1	Quatpump	HP	G1311A	DE91606476	1999
1042	HPLC-1	ALS	HP	G1313A	DE91608580	1999
1043	HPLC-1	Colcom	HP	G1316A	DE91609970	1999
1044	HPLC-1	UV Detector	HP	G1314A	JP92108737	1999
1045	HPLC-1	Fluorescence Detector	Jasco	FP-920	D398 1892	1999
1046	HPLC-1	Interface	HP	35900E	CNDDQ1250	1999
1047	TOC-1	Total Organic Analyzer	Shimadzu	TOC5050A	36201577A	1999
1048	TOC-1	Auto Sampler	Shimadzu	ASI5000A	36N02328A	1999
1049	IC-Dionex	Auto Sampler	Dionex	AS40	99030213	1999
1050	IC-Dionex	PED	Dionex	RDM-1	912902	1999
1051	IC-Dionex	VWD	Dionex	VDM-2	912910	1999
1052	IC-Dionex	Pump	Dionex	AQP-1	913051	1999
1053	IC-Dionex	Liquid Chromatograph	Dionex	LCM-3	913205	1999
1054	HOOD #1	Hood	Airflow Supreme	48"x31"	N/A	
1055	HOOD #2	Hood	Labconco	49"x26"	N/A	
1056	HOOD #3	Hood	Labconco		N/A	
1057	HOOD #4	Hood		48"x32"	N/A	
1058	HOOD #5	Hood		98"x23"	N/A	
1059	HOOD #6	Hood		72"x23"	N/A	
1060	HOOD #7	Hood	Airflow Supreme	48"x31"		
1061	HOOD #8	Hood	Kewaunee Scientific	48"x33"		
1062	HOOD #9	Hood	Fisher Scientific	98"x23"	N/A	
1063	HOOD #10	Hood		75"x23"	N/A	
1064	HOOD #11	Hood		86"x23"	N/A	
1065	HOOD #12	Hood		72"x23"	N/A	
1066	HOOD #13	Hood		72"x23"	N/A	
1067	HOOD #14	Hood		98"x23"	N/A	
1068/ 1202	HOOD #15	Hood		98"x23"	N/A	
1069	HOOD #16	Hood	Labconco	96"x31"	Labconco	
1070	HOOD #17	Hood		96"x31"	N/A	
1071	R1	Refrigerator	TRUE	R1-1071	N/A	1998
1072	R8	Refrigerator	Frigidare	1072	N/A	1999
1073	R10	Refrigerator	TRUE	R10-1073	N/A	2000
1074	R6	Refrigerator	VWR	1074VWR	N/A	2000
1075	R7	Refrigerator	Magic Chef	UNK	N/A	
1076	F1	Freezer	Sanyo	UNK	N/A	
1077	F2/R2	Freezer/Refrigerator	Sears	UNK	N/A	

1078	R9	Refrigerator	UNK	UNK		
1079	R4	Refrigerator	absocold	Cold-PRO18		1998
1080	BOD1	Refrigerator	Roper	UNK	N/A	
1081	F3/R5	Freezer/Refrigerator	Hotpoint	UNK	N/A	1997
1082	MW-1	Microwave	Sharp	Carosel II	N/A	
1083	DR-2	Oven	VWR	1350D	N/A	1998
1084	IB-1	Incubator	Scientific Prod	Tempcon	N/A	1999
1085		Oven	SanPlantrac	Drykeeper	N/A	1999
1086	DR-1	Oven	VWR	1305	N/A	1996
1087	DR-3	Oven	VWR	1305	N/A	2000
1088	INCUBATOR 2	Oven	Fisher Scientific	655G	N/A	2002
1089	Balance 5	Top Loader	Denver Inst	AL500	B039416	2002
1090	Balance 3	Analytical	Denver Inst	XA100	17311	1998
1091	Balance 2	Top Loader	Denver Inst	AL3K	B039404	2001
1092	Balance 1	Analytical	American Scientific	SP180	2903470	2000
1093		Digestor	Tecator	2020	16780	
1094		Hot Plate	Thermolyne	HP47135	61195025158	1998
1095	Muffle Furnace	Furnace	Thermolyne	FB1315M	347910258 581	1996
1096	Muffle Furnace	Furnace	Thermolyne	FB1315M	347920812 750	2000
1097		Digestor	CPI	MOD	05-C0530-0029	
1098		Sonicator	Branson	B2200R-1	N/A	1997
1099	MIDI Distillation	Distillation	Lachat	1700	2000-419	2002
1100	Flash Point	Flash Point Analyzer	Precision Scientific	74537	BD-010	
1101		Heating Plate	Fisher Scientific	11-500-4H	692941120 349	2002
1102		Heating Plate	Fisher Scientific	11-500-4H	692941120 367	2004
1103		Hot Plate	Corning	PC300	N/A	2000
1104		Hot Plate	Corning	PC300	N/A	2000
1105		Hot Plate	Corning	PC300	N/A	2000
1106		Hot Plate	Corning	PC300	N/A	2000
1107		Hot Plate	Corning	PC300	N/A	2000
1108		Hot Plate	Corning	PC300	N/A	2000
1109		Heater	Glasco	TM108	124700A	
1110		Heater	Glasco	TM108	124119A	
1111		Heater Controller	Glasco	PL312	325887	
1112		Heater Controller	Glasco	PL312	326583	
1113	Lachat-1	Lachat	Lachat	RAS	8A1004-165	2002
1114	Lachat-1	Auto Analyzer	Lachat	QuikChem8000	A83000-1018	2002
1115		Reagent Pump	Lachat	1115	A82000-403	2002
1116	pH Meter	pH Meter	Orion	230A	7320	1998
1117		Stirrer	Thermolyne	S46415	621910362 845	
1118		Stirrer	Thermolyne	S46415	621910254 346	
1119		Ion Analyzer	Orion Research	EA940	TR36A	2000
1120		Ion Analyzer	Orion Research	EA940	PO84A-M	2000
1121	Meter	Electrode	Orion Research	970899	N/A	1998
1122		Spectrophotometer	Hach	DR2010	000-100016553	2000
1123		Stirrer	Equatherm	267-955	4409300148 022	2002
1124	Meter	Conductivity Meter	LabCraft	N/A	N/A	1998

1125		Water Bath	VWR	1200	400699	1999
1126		Rotator	Associated	3740-8-BLE	N/A	2000
1127		Rotator	Associated	3740-8-BRE	N/A	1996
1128		Rotator	Associated	3740-8-BRE	1248	
1129		Concentrator	Zymark	TurboVap II	TV9909N8714	
1130		Water Bath	VWR	1245PC	UNK	2002
1131		Water Bath	VWR	1204	300200	2000
1132		Flash Point Analyzer	Precision Scientific	74537	10AZ-4 393745-0012	
1133		Centrifuge	IEC	Clinical	42833750	1996
1134		Water Bath	Precision Scientific	1B5	10AZ-8 394893-0014	
1135	CF-1	Centrifuge	Damon/IEC	Clinical	4280180	1999
1136	CF-2	Centrifuge	IEC	Clinical	3703	2001
1137		O2 Bomb Calorimeter	Parr	13031	1422	1994
1138		Stirrer	Thermolyne	S46415	621910362 945	1998
1139		COD Reactor	Hach	4500	960500014 152	2000
1140		Heater/Stirrer	Thermolyne	Cimarec 2	1069970704 265	2001
1141		Cleaner/Sonicator	Branson	B5200R-4	N/A	2001
1142		Hot Plate	Corning	PC100	N/A	1997
1889		Ultrasonic Convertor	Fisher Scientific	F550	F2420	2000
1890		Ultrasonic Convertor	Fisher Scientific	F550		
1144	Threads Tap & Die Set		Westward			2008
1145		Heater/Stirrer	Thermolyne	Climarec 2	640920255 329	
1146	Meter	Turbidity Meter	LaMotte	2008	1846-2793	1999
1147		Flow Meter	Fisher Scientific	650	291277	1996
1148	FAA	AA	Varian	Spectra 220	EL97103119	1998
1149		Auto Sampler	Varian	SPS-5	94031199	1998
1152	Meter	Electrode, Flouride	Orion	94-09	VX1	2000
1153	Meter	Conductivity Meter	Orion	150	19462	2001
1154		Timer	VWR	N/A	N/A	
1155		Pipette, graduated	Oxford	BenchMate	130046	
1156		Pipette, graduated	Oxford	BenchMate	30043	
1157		Pipette, graduated	Eppendorf	2500	115476	
1158		Pipette, graduated	Pipetman	P5000	B16278B	
1159		Pipette, graduated	Oxford	Macroset	5067	
1160		Pipette, graduated	VWR	Calibra 832	641004	
1161		Pipette, graduated	Wheaton	Calibra 822	544321	
1162		Pipette, graduated	Wheaton	Socorex	9061466	
1882		Pipette, graduated	Oxford	BenchMate	130173	
1164		Pipette, graduated	VWR	UNK	0 131242	
1165		Pipette, graduated	Eppendorf	Reference	179614	
1166		Dispenser	Optifix	Basic	N/A	
1167		Dispenser	Optifix	Basic	N/A	
1168		Dispenser	Optifix	Basic	N/A	
1169		Dispenser	Optifix	Basic	N/A	
1170		Pipette, graduated	Wheaton	Calibra 822	71760	

1171	ZHE #1	ZHE		N/A	N/A	
1172	ZHE #2	ZHE		N/A	N/A	
1173	ZHE #3	ZHE		N/A	N/A	
1174	ZHE #4	ZHE		N/A	N/A	
1175	ZHE #5	ZHE		N/A	N/A	
1176	ZHE #6	ZHE		N/A	N/A	
1177		Velometer	Alnor	Jr.	N/A	
1178	#3	Pipette, graduated	Oxford	Macroset	6309	
1179		Dispenser	Optifix	Basic	N/A	
1180		Heater Controller	Glasco	PL312	325858	
1181	#5	Repipetter			N/A	
1182	Balance 4	Analytical	Mettler	AE100-240	L39952	
1183	Weight Set B	Weights, Certified		10 Piece	1000105495	
1184						
1888		Concentrator	Zymark	TurboVap II	TV0116N10262	
1186	BOD2	Refrigerator	Kenmore	29111	216584620	1997
1540	BOD3	Incubator	Kenmore	253.24082100	WB42923928	2004
1187	MS-9	GC	Agilent	6890N	US10133113	2000
1188	MS-9	MS	Agilent	5973	US10441238	2000
1189	MS-9	Auto Sampler	Agilent	G2614A	US12419350	2000
1190	Pipettor	Pipette	VWR-brand		105710154	
1191	Pipettor	Pipette	VWR-brand	0.1-1.0 ml	1294	
1861	Pipettor	Repipetter	Oxford Maxi Set	1-5 ml	7209	
1193	Pipettor	Pipette, graduated	Eppendorf	1-5 ml	N/A	
1902	Pipettor	Pipette, graduated	Eppendorf	0.1-1.0 ml	N/A	
1195	Pipettor	Repipetter	Oxford Maxi Set	1-10 Ml	N/A	
1196	Pipettor	Repipetter	Oxford Maxi Set	5-10 Ml	316	
1197	Pipettor	Pipette, graduated	Eppendorf	0.1-1.0 ml	N/A	
1198	Pipettor	Pipette, graduated	Rainen Pipetman	0.1-1.0 ml	N/A	
1199	Pipettor	Repipetter	Oxford Maxi Set	5-10 ml	9288	
1200	Hood #1	Hood	Kewaunee Scientific	48"x33"		
1201	Hood #2	Hood	Fisher Scientific	98"x23"	NA	
1202	Hood #5	Hood		98"x23"	NA	
1203	Hood #7	Hood	Airflow Supreme	48"x31"	NA	
1204	Hood #8	Hood	Airflow Supreme	48"x31"		
1205	Hood #9	Hood		70"x29"	NA	
1206		Extractor	Soxtherm		441262	2002
1207		Oven	Despatch	PRO-1		2002
1208		Oven	Lindberg / Blue M	Blue M		2002
1209		Oven	Fisher Scientific	649F		2002
1210	MS-10	GC/MS	Agilent	5973	US82311282	1998

1211	MS-10	GC/MS	Agilent	6890	US00024777	1998
1212	MS-10	Autosampler	Agilent	7683	US84302879	2001
1213	Balance 8	Toploader	Fisher Scientific	XL500	B028172	1997
1214	Balance 7	Analytical	AND	FR300	8400404	1998
1215	Balance 9	Analytical	Sartorius	1602	3405235	1997
1216	Turbidimeter	Turbidity Meter	Orbeco-Hellige	T-10	96510A	2002
1217	TOC-2	TOC	Rosemount	DC-190	L9408399	2002
1218	TOC-2	Auto Sampler	Rosemount	183	9401165	2002
1886	Shaker	Shaker	Eberbach		39707	
1220	AAGF	AAGF	Perkin Elmoer	4100 ZL	7022	
1221	FIMS	Auto Sampler	Perkin Elmer	AES-90		1999
1223	ICP	ICP	TJA Trace		5090	
1224	MS-11	Auto Sampler	Varian	Archon	12536	1999
1225	Autosampler	Auto Sampler	Varian	Archon	12535	
1226	MS-11	Sampler Concentrator	OI Corporation	4560	3515A10291	1999
1227	MS-11	GC	Agilent	5890	3336A56613	1994
1228	MS-11	MS	Agilent	5973	3435A01886	1994
1229	Concentrator	Concentrator	OI Corporation	4560	94284012	
1230	Sterili\zer	Sterilizer	National	9000D	10771117-185	
1231	pH Meter	pH Meter	Orion	EA 960	EQ 743480	2002
1232	Spectrophotometer	Spect	Milton Roy	301	3800121004	2000
1627	Spectrophotometer	Hach DR 3000	Hach	19600-00	900402416	1990
1233	Flame Photometer	Flame Photom	Bechmann	FP-124	3140	2002
1862	Pipettor	Pipette	VWR-brand	0.500ml	105700229	
1235	Pipettor	Pipette	Oxford	BenchMate	261862	
1236	Pipettor	Pipette	Eppendorf	0.1-1.0 ml	N/A	
1237	Pipettor	Pipette	Eppendorf	0.010-0.100 ml	N/A	
1238	Pipettor	Pipette	Eppendorf	0.10-1.0 ml		
1239	Pipettor	Pipette	Oxford	0.20-1.0 ml	100303	
1240	Pipettor	Pipette	Oxford	5-10 ml	5-10 ml	
1241	Pipettor	Pipette	Eppendorf	0.10-1.0 ml	N/A	
1242	Pipettor	Pipette	Eppendorf	0.10-1.0 ml	N/A	
1243	O2-3	Electrode, DO	ThermoOrion	9708		2002
1244	Weight Set	Weights, Certified	Fisher Scientific	10 Piece	12068	
1245	Weight Set	Weights, Certified	Fisher Scientific	10 Piece	12058	
1246	Paint Sprayer			Magnum RX5		2008
1247	4" Multipurpose Bench and Pipe		Northern Tools			2008
1248	ICP/MS-TJA	ICP/MS	Thermo Elemental	X-7	X0199	2002
1249		Autosampler	Cetas Technologies	510	120102 ASX	2002
1250	FAA (Hg)	Vapor Generator	Varian	VGA 77	95081021	

1251	ICP/MS-TJA	Chiller	Thermo NSLAB	M75	101365502-1	
1254		Respirator	MSA	Full Face, Medium		
1255		Respirator	Survivair	1/2 Face		
1256		Respirator	ERB Safety, Inc	1/2 Face, Medium		
1257		Respirator	ERB Safety, Inc	1/2 Face, Medium		
1258		Respirator	ERB Safety, Inc	1/2 Face, Medium		
1259		Respirator	3M	1/2 Face		
1260		Respirator	Wilson	Full Face, Medium		
1261		Respirator	RACAL H&S, Inc.	Full Face, Medium		
1262		Respirator	Survivair	1/2 Face		
1263	Balance 6	Toploader	Ohaus	G4000D	4303	1994
1264		Ultrasonic Convertor	Fisher Scientific	F550	F1643	
1265	Microscope	M2 LabScope	LW Scientific	LW 200	301473	
1303	Furnace					
1304	Hole Saw Cutting Tips		Ridgid Co			2008
1305	Microscope	Microscope	Nikon	Y52-T	159996	2002
1306	Microscope	Microscope				
1339	Cutoff Saw	Electric Power Tool	Harbor Freight Tools	1624		2008
1340						
1447	Fire Extiguisher	4-A:60-B:C (10 lb)	Kidde	460HCM	SJ-541115	
1448	Fire Extiguisher	3-A:40-B:C (5 lb)	Buckeye	5HI SA-40 ABC	RJ-891427	
1449	Fire Extiguisher	4-A:60-B:C (10 lb)	Kidde	460HCM	SJ-541264	
1450	Fire Extiguisher	2-A:10-B:C (5 lb)	Amerex	B500	VV-197997	
1451	Fire Extiguisher	1-A:10B:C (11 lb)	Amerex	397	W-609482	
1452	Fire Extiguisher	2-A:10-B:C (5 lb)	Amerex	B500	VV-197042	
1453	Fire Extiguisher	2-A:10-B:C (5 lb)	Amerex	B500	VR-591740	
1454	Fire Extiguisher	1-A:10B:C (11 lb)	Amerex	397	W-608377	
1455	Fire Extiguisher	2-A:10-B:C (5 lb)	Amerex	B500	VR-591795	
1456	Fire Extiguisher	3-A:40-B:C (5 lb)	Buckeye	5HI SA-40 ABC	RG-566914	
1457	Fire Extiguisher	20-A:80-B:C (15lb)	General	TGP-20D	EX-093393	
1458	Fire Extiguisher	4-A:80-B:C (10lb)	Buckeye	10HI SA 80 ABC	RX-328263	
1459	Fire Extiguisher	1-A:10B:C (2.5 lb)	Kidde	K110	JE-593071	
1460	Fire Extiguisher	2-A:10-B:C (5 lb)	Amerex	B500	VR-517253	
1461	Fire Extiguisher	1A:10B:C (2-5/8)	Kidde	Pro2-5/8 TCM-5	NS-438874	
1462	Fire Extiguisher	4-A:60-B:C (10 lb)	Kidde	460HCDM	SJ-535543	
1463	Fire Extiguisher	3-A:40-B:C (5 lb)	Badger	5MB-6H 2003	VY-672491	
1464	Fire Extiguisher	1-A:10B:C (2-5/8)	Kidde	Pro2-5/8 TCM-5	NS_-439056	
1465	Fire Extiguisher	4-A:60-B:C (10 lb)	Kidde	10 TAS-6	EX-722911	
1466	Fire Extiguisher	3-A:40-B:C (5 lb)	Badger	5MB-6H 2003	VY-652050	

1467	Fire Extiguisher		Extinguisher Svcs.	HMCL-11		
1468	Fire Extiguisher	1-A:10B:C (2.5 lb)	Buckeye	2.5 SA-ABC	RR-490753	
1469	Fire Extiguisher	4-A:80-B:C (10lb)	Badger	10 MB-8H 2003	VZ-169076	
1470	Fire Extiguisher	1-A:10B:C (2.5)	Buckeye	2.5 ABC-100	PS-30886	
1471	Incubator-3		Precision	51221033	601121210	2003
1472	Chromatography	Refrigerator	Forma Scientific	3791	786550420	
1473	Sterilizer (Autoclave)		Market Forge/Sterilmatic	STM-E	5185	
1474	Hood		Micro-bio Hood Scientific	Nuaire 4Ft		
1475	Hood #21		Fisher		93-809Q	
1476	Incubator-4		Forma Scientific	3327	33602-186	1996
1477	Dremel Tool and Buffing Tips				F013770003	2008
1478	Micropipettor	50-200 µl	VWR	821	13021146	
1479	Micropipettor	5-50 µl	VWR	821	12121230	
1480	Pipettor	Autoclavable 1-5 ml	Nichiryo	Nichipet EX	H35012181	
1481	Pipettor	Autoclavable 1-10 ml	Nichiryo	Nichipet EX	H27014361	
1482	R20	Refrigerator	WCI Commer. Ref	13-987-348G	93866590	
1541	R23	Refrigerator	Turbo Air	TGM-48R	GR48901137	2004
1500	Printer		Seiko	Smart Label Printer 100	E039645783	
1501	Pump	Vacuum Pump	_	2522B-01	_	
1502	Microscope	M2 LabScope	LW Scientific	LW 200	30H584	1998
1503	MS-12	5973	HP	5973	US81221559	2003
1504	MS-12	6890/GC	HP	6890	DE00020822	2003
1506	Balance 10	Analytical	Sartorius	BP110	60804869	2003
1505	MS-12	Sample Concentrator	OI Corporation	4660	A350466159	2003
1620	MS-13	GC	Agilent	6850N	US10506012	2005
1621	MS-13	MS	Agilent	5973N	US52047399	2005
1622	MS-13	Autosampler	Varian	Archon	14371	2005
1602	MS-13	Purge Press/4660	OI Analytical	4660	B421466132P	2004
1624	MS-13	Computer	HP Compaq	HP Compaq	2UA5160HR7	2005
1625	MS-13	Monitor	HP 9500	HP 9500	CNC44205ZC	2005
1626	MS-13	Printer	HP Laser Jet 2420d	HP Laser Jet 2420d	CNGK800775	2005
1506	Balance	Analytical	Sartorius	BP110	60804869	2003
1507	ICP-Varian	ICP-OES	Varian	VISTA-PRO	EL00123003	
1508	Thermometer	Alcohol	VWR	FRIO-TEMP	6645	
1509	Thermometer	Digital	VWR		21420702	
1510	Hydrometer Tester		Delmhorst Inst Co			2008
1511	HotBlock (1)	Digestor	CPI	MOD	05C0530-0029	2000
1512	HotBlock (2)	Digestor	CPI	MOD	05C0530-0029	2001

1513	Block Digestor	BD-46 Block Digester	Lachat	BD-46	1 800 703	2002
1514	Vortex	Genie	Scitific Industries	G-560	2-991986	
1515	Waterbath		Fisher Scientific			
1516	Stirrer	Fisher Stirrer	Fisher Scientific		61000838	
1517	pH Meter	pH Meter	Orion	410A	*	2002
1518	Dissolved Oxygen Meter	Dissolved Oxygen Meter	YSI	SI 5100	04C371	2003
1519	Lachat-2	YXZ	Lachat	ASX-500 Series	A81010-774	2003
1520	Lachat-2	Autoanalyzer	Lachat	QuickChem FIA+ 800 Series	A8300-2107	2003
1521	Lachat-2	Reagent Pump	Lachat	RP-100Series		2003
1522	Autosampler	YXZ	Lachat	ASX-500 Series	A81010-774	
1523	Autosampler	Autosampler	Varian	SPS 5	EL00043932	
1524	Thermometer	Mercury	VWR	76MM IMM	3S1550	
1525	Table Saw		Ridgid Co	TS3650	TH100085	2008
1526	Miter Saw		Ridgid Co	MS1250LZ1	225161100D	2008
1527	Jig Saw		Ridgid Co	R3121	BB055100340	2008
1528	Palm Sander		Ridgid Co	R2500	BB0604938389	2008
1529	Notebook		Toshiba	Satellite A45-S120	24055234H	
1548	Thermometer	Alcohol				
1549	Thermometer	Alcohol				
1550	Thermometer	Alcohol				
1551	Thermometer	Alcohol				
1552	Thermometer	Mercury				
1553	Skill Saw		Ridgid Co	R3200	BB073626798	2008
1554	Bit Set		Dewallt Co			2008
1555	4" Angle Grinder		NE Tool Co	151604		2008
1556	Wrech Socket Set		Home Depot			2008
1557	MAPP Gas Torch	Propane	Grainger			2008
1558	Thermometer	Digital	Fisher	15-077-941	51004331	
	Tile Cutter					2008
1559	Temperature Tester		Grainger	Fluke 62		2008
1560	Thermometer	Alcohol			F93881	
1561	Thermometer	Mercury			4464	
1562	Thermometer	Alcohol			T-1021	
1563	Thermometer	Mercury			Spare6	
1564	Thermometer	Mercury	`		14-983-10B	
1565	Thermometer	Mercury			3P9286	
1566	Thermometer	Alcohol			6753	
1567	Thermometer	Alcohol			F80593	
1568	Thermometer	Mercury			F94181	
1569	Thermometer	Mercury			F80408	

1570	Thermometer	Mercury			H64312	
1571	Thermometer	Mercury			F41082	
1572	Thermometer	Alcohol			F94132	
1573	Thermometer	Mercury			H64479	
1574	Thermometer	Alcohol			F94347	
1575	Thermometer	Alcohol			F33197	
1576	Thermometer	Mercury			F80310	
1577	Thermometer	Mercury			F80334	
1578	Thermometer	Mercury			H22275	
1579	Thermometer	Alcohol			F98363	
1580	Thermometer	Digital			21420735	
1581	Thermometer	Digital			21420731	
1582	Thermometer	Digital			21358523	
1583	Thermometer	Digital			21358306	
1584	Thermometer	Mercury			F56312	
1585	Thermometer	Mercury			61054	
1586	Thermometer	Mercury			9530	
1588	Thermometer	Mercury			14-983-10B	
1589	Thermometer	Mercury			F34375	
1590	Thermometer	Alcohol			6453	
1591	Thermometer	Alcohol			T-2560	
1592	Thermometer	Alcohol			T-1023	
1593	Thermometer	Mercury			Spare 7	
1594	Thermometer	Mercury			3676	
1595	Thermometer	Mercury			92-13219	
1596	Thermometer	NIST Traceable			HB/B 28199	
1597	Thermometer	NIST Traceable			NIST #1	
1598	Thermometer	NIST Traceable			NIST #2	
1599	Pipetter	1.0-5.0 mL	Oxford		5067	
1600	Compressor	12 gal Compressor	Sears	921-1664000	54547	2008
1603	SPE	Vacum Pump	Horizon Tech	DAAV716-EB	304506054	2004
1604	SPE	Speed-Vap III Evaporation Unit	Horizon Tech	Speed Vap III	42041	2004
1605	SPE-Extractor	SPE-DEX-4790 Extractor	Horizon Tech	SPE-4790	04-0485	2004
1606	SPE-Extractor	SPE-DEX-4790 Extractor	Horizon Tech	SPE-4790	04-0484	2004
1607	SPE-Extractor	SPE-DEX-4790 Extractor	Horizon Tech	SPE-4790	04-0483	2004
1608	SPE-DEX Controller	SPE-DEX Controller	Horizon Tech	SPC-100	04-0431	2004
1542	SPE-DEX Extractor	SPE-DEX 4790 Extractor 4	Horizon Tech	SPE-4790	04-0524	2004
1618	SPE-DEX Extractor	SPE Extractor #5	Horizon Tech	SPE-4790	04-0571	2005
1609	IC2	ICS 1000 Ion Chrom. System	Dionex	ICS-1000	5010499	2005
1610	IC2-Auto Sampler	AS 40 Automated Sampler	Dionex		4100492	2005

1611	IC2-Computer	Optiplex	Dell	GX2280	3GPMG61	2005
1612	IC2-Monitor	15" Monitor	Dell	LCD	MX-0D5421-46634-4C8-1CAT	2005
1633	Tray Seater	Quanti Tray Seater Model 2X	Idexx Labs	89-10894-02	3953	Aug-05
1634	Balance	Mettler Balance HL52	Lab Tech, Inc	HL52	731163	2000
1635	Balance 11	Toploader	Mettler	P2210	962061	2000
1657	Digital Reactor Block	Digital Reactor Block 200	Hach	LTG082.99.420 01	1147550	
1660	ICP-PE	Optima 4300DV	Perkin Elmer	Optima 4300 DV	077N0092302	2006
1661	ICP-PE/Chiller	Chiller	Perkin Elmer	N0772026	654704	2006
1670	Autosampler	Autosampler	Perkin Elmer	AS93 plus	1175	2006
1671	Water Purification System	Solution 2000	Solution Consultants, Inc.	2001A	930211A	
1669	Gas Regulator	Gas Regulator	March Inst, Inc.	GCM-200	1280	
1672	Plasma Asher	Vacuum Chamber	March Inst, Inc.	Plasmo D	1352	
1673	Vacuum Pump	Vacuum Pump	Leybold-Haraeus Vacuum	D4AC	128752007E	
1674	GC-8	GC-8	Agilent	6890N	CN 10609020	Mach 2006
1675	Injector	Injector (Tower)	Agilent	7683B	CN603330862	Mach 2006
1676	ALS Sampling Tray	ALS Sampling Tray	Agilent	G2614A	CN60638448	Mach 2006
1691	Hot Block	Hot Block #4	Environmental Express	SC154	4110CEC1933	8/1/2006
1692	Hot Block	Hot Block #5	Environmental Express	SC154	4110CEC1929	8/1/2006
1693	Fire Extinguisher	Fire Extinguisher	Cintas	A456	287752	10/1/2006
1694	Spectrophotometer	UV/VIS	Shimadzu	BIOSPEC-1601	50103R	10/12/2006
1699	Fire Extinguisher	CO2	Badger		Z-531491	11/1/2006
1700	Balance 12	Analytical	Mettler	AL104	1227330378	11/1/2006
1701	Pipettor	1-5 mL	Accumax		26741	12/6/2006
1702	Pipettor	0.1-1.0 mL	Accumax		101635	12/6/2006
1703	Pipettor	0.05-0.2 mL	Accumax		34053	12/6/2006
1704	Pipettor	0.1-1.0 mL	Accumax		101636	12/6/2006
1705	Freezer	3.5 Cubic feet	Haier	HNCM035E	605001120	12/1/2006
1706	Thermometer	Alcohol	FRIO-Temp		F-11	12/6/2007
1707	MS-14	MS	Inert	5975B	US62714424	12/1/2006
1708	MS-14	GC	Inert	6890 N	CN10631084	12/2/2006
1709	MS-14	Autosampler	Inert	7683B	CN63835818	12/1/2006
1710	Paint Sprayer			Magnum RX5		2008
1711	4" Multipurpose Bench and Pipe		Northern Tools			2008
1712	Saws All		Ridgid Co	R3000S	BB073512647	2008
1713	Thermometer	Alcohol	FRIO-Temp		F96194	

1714	Microscope	Vision Microscope	Lab Essentials, Inc.	Vision	505007	5/8/2006
1715	Microscope	Vision Microscope	Lab Essentials, Inc.	Vision	505019	5/17/2006
1716	Microscope	Vision Microscope	Lab Essentials, Inc.	Vision	505029	5/17/2006
1717	Balance 13	Analytical	Mettler	AL104	1227300041	2/15/2007
1720	Hood #11	Station 5				
1722	Stage Micrometer		Microscope Service, Inc.	L & W		Oct-05
1723	Pipettor	5ml	NICHIRYO	Benchmate II		
1724	Pipettor	5ml	NICHIRYO	Benchmate II		
1725	SPE-DEX Extractor	3 Place 'Oil & Grease Machine'	Horizon Tech	SPE 3000XL Plus-SS	07-2081	4/24/2007
1726	SPE-DEX Controller	3 Place SPE-DEX Controller	Horizon Tech	SPC-3000	07-1387	4/24/2007
1727	Thermometer	Traceable Sentry therm. w/probe	Fisher Scientific	15-077-941	72071657	2004
1728	MS-15	GC	Agilent	6850A	US10710001	2007
1729	MS-15	MS	Agilent	5973 Inert	US44610842	2007
1730	MS-15	Purge & Trap	OI Corporation	Eclipse 4660	D713466088P	2007
1731	MS-15	Autosampler	Varian	Archon	15099	2007
1732	MS-15	Computer	HP Compaq	ESO	USV3400DTH	2007
1733	MS-15	Monitor	Envision	H7124	A3871JA006986	2007
1819	F12/R28	Freezer/Refrigerator	Daewoo	FR-3503	KE071E2937 0137	6/8/07
1820	Freezer (F12) Thermometer	Alcohol	VWR	FRIO-TEMP	F99047	6/8/07
1821	Refrig (R28) Thermometer	Alcohol	VWR	FRIO-TEMP	T39733	6/8/07
1824	Refrig (R8) Thermometer	Alcohol	VWR	FRIO-TEMP	T40228	6/26/2007
1827	Pipettor	0.500 mL	VWR	VWR FE500	736900038	8/13/2007
1828	Thermometer	Alcohol	VWR	FRIO-TEMP		9/24/2007
1829	Thermometer	Traceable Ultra 8" stem	VWR	23226-658	72102502	10/10/2007
1830	Thermometer	Traceable Ultra 8" stem	VWR	23226-658	62008306	10/10/2007
1831	Thermometer	Traceable Ultra 8" stem	VWR	23226-658	72102439	10/10/2007
	Nailer Frame		Home Depot	FR350A	200748BA48741	2008
1832	Pipettor	1-5ml	Nichipet	Nichipet EX	EX H73006071	
1833	Pipettor	0.02-0..2ml	Nichipet	Nichipet EX	EX H07812651	
1834	Pipettor	1-5ml	Nichipet	Nichipet EX	EX H74006391	
1835	Air Compressor Regulators		Husky			2008
1836	Walking Tape Measurer		Lufkin	PS MW 18	0-37103-195272	2008

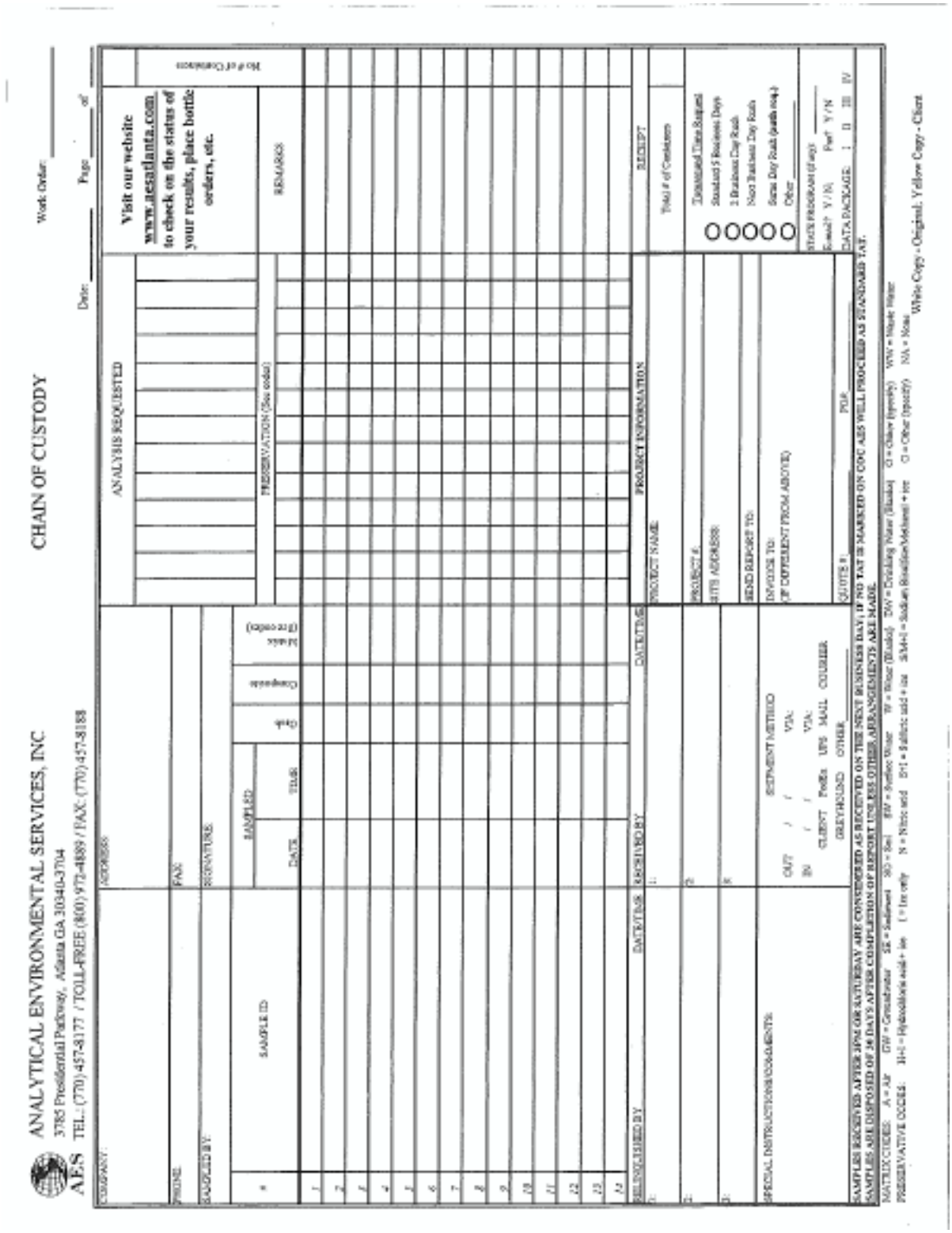
1837	Microscope	Meiji PLM Asbestos Microscope	MilesCo Scientific	ML6130	600091	2008
1838						
1839	Pipettor	1-5ml	Nichipet	Nichipet EX	H74006211	
1840	Pipettor	.500ul	Thermo Electron	4601100	AA96221	
1841	Balance 14	Analytical	Ohaus	AP3105	M52542	2003
1842	Thermometer	Infared Temperature Gun	VWR	7776-724	80170831	6/10/2008
1843	Thermometer	Alcohol	VWR	FRIO-TEMP		2008
1844	Dissolved Oxygen Probe	BOD Probe	YSI	5010	None (See 1518)	2003
1845	Conductivity Probe	Conductivity Meter Probe	Orion	14010	None (See 1153)	2001
1846	pH Probe	pH Meter Probe	Orion	Lot #JQ1	None (See 1119)	2000
1847	pH Probe	pH Meter Probe	Orion	Lot #MV1	None (See 1119)	2008
1848	Pipettor	5000ul	Nichiryo	Benchmate II	H71009871	2009
1849	Hot Block	Hot Block	Environmental Express			2005
1850	Rotator	Tumbler	Dayton	Motor 5k939A		2005
1851	Rotator	Tumbler	Dayton	Motor 1LPP4		2005
1852	Muffle Furnace	Furnace	Ney	6-160A		1999
1853	Distillation Unit	P/N 483-B000-01	WestCo Scientific	EASY DIST - triangular	1072	2008
1854	2 mg Weight	Aluminum, Class 1	Troemner	NVLAP Wt. Calibration	41931	2008
1855	2 mg Weight	Aluminum, Class 1	Troemner	NVLAP Wt. Calibration	41932	2008
1856	Dissolved Oxygen Probe	BOD Probe	YSI	5010	6F06E1720	2008
1857	COD Reactor	30 position; 120V; 200Watt	Bioscience	100 003	COD-B0203	2008
1858	Thermometer	Mercury for Flashpoint	VWR	57 MM IMM ASTM 9F-86	3236	2008
1859	Thermometer	Mercury for Flashpoint	VWR	57 MM IMM ASTM 9F-86	3239	2008
1860	Thermometer	Mercury for Flashpoint	VWR	57 MM IMM ASTM 9F	2C5474	
1863	Waterbath	KD Concentration	VWR	Primary in Prep		?
1864	Hood	Big 6	Fisher Scientific	Safety Flow Fume Hood	93-508Q	?
1865	pH Probe	pH Meter Probe	VWR	Lot #MO1, 1040113	ECN # 662-1051	2009
1866	pH Probe	pH Meter Probe	Thermo Scientific	Orion 9102BNWP	Lot Code LS1	2009
1867	pH Meter	pH Meter	Orion	940	QX117A-M	1994
1868	Hood	Hood	Fisher Scientific	93-508Q		?
1869	Pipettor	1-10mL	Nichiryo-Oxford	Benchmate II	H71013122	2009

1870	Thermometer	Alcohol (Red Spirit)	Fisher	14-997		2009
1871	Thermometer	Alcohol (Red Spirit)	Fisher	14-997		2009
1872	Thermometer	Alcohol			307058	
1873	Thermometer	Alcohol (Red Spirit)	Fisher	14-997		2009
1874	Pipettor	1-5mL	Nichiryo-Oxford	Benchmate II	H6Z004111	2009
1875	Pipettor	1-10mL	Nichiryo-Oxford	Benchmate II	H71013592	2009
1876	Pipettor	0.020-0.2mL	Nichiryo-Oxford	Benchmate II	H5X003711	2009
1877	Thermometer	NIST Traceable	ICL Labs	310-120-C	1018	2009
1878	Hygrometer-Thermometer	NIST Traceable	VWR	21800-066	90866528	2009
1879	Thermometer	Traceable Ultra 8" stem	VWR	23226-658	90850250	2009
1880	Sanyo Refrigerator	Refrigerator	Sanyo			2008
1881	Thermometer	Dial	HACH	Block Digestor-Ammonia		1998
1883	Thermometer	Mercury	VWR	Cat. No. 61027-205	8323	2009
1884	Thermometer	Mercury	VWR	Cat. No. 61222-550	8918	2009
1885	Thermometer	Digital	Fisher Scientific	Cat. #15-077-941	72071669	2007
1891	Vaccuum	14 Gallon Pro Vac	Ridgid Co / Home Depot	WD14500	08202 R1125	2009
1892	Hammer Drill	Drill	Dewallt Co		55751	2009
1893	Flow Meter	Hi Flow Sampler	Gilian	HFS 113A	3702	2001
1894	Refrigerator	Freezer/Refrigerator	Kenmore	253.745326	BA73115845	2007
1895	Waterbath	KD Concentration	Thermo Scientific	2843	208984	2009
1896	Thermometer	Digital	VWR	Cat. No. 61161-289	90806951	2009
1897	Thermometer	Red Alcohol	VWR	Cat. No. 89095-574		2009
1898	Thermometer	Blue Alcohol	Thermo Scientific	307059	4H9444	2009
1899	FIMS	Flow Injection Mercury System	Perkin Elmer	FIMS 100	101S9121001	2009
1900	Turbovap II	Concentration Workstation	Caliper Life Sciences	103187	TV0953N15641	2010
1901	Cordless Drill / Driver	C3 19.2 volt Diehard	Sears	315.11581	BD0828	2010
1903	Pipettor	1-10mL	Nichiryo-Oxford	Benchmate II	H71021912	2010
1904	Thermometer	Traceable Ultra 8" stem	VWR	23226-658		2004
1905	Thermometer	Traceable Ultra Waterproof	VWR	37000-414	101527348	2010
1906	Thermometer	Traceable Ultra Waterproof	VWR	37000-414	101527357	2010
1907	Thermometer	Digital	VWR	Cat. #21800-072	101494163	2010
1908	Incubator	Low Temperature	VWR	2020	100696	2010

1909	Dissolved Oxygen Probe	BOD Probe	YSI	5010	Lot #10C100275	2010
1910	Dissolved Oxygen Meter	BOD Meter	YSI	500-115V	07H101424	2010
1911	Pipettor	Reference Adjustable Volume	Eppendorf	Series 2000	3872438	2010
1912	Pipettor	Reference Adjustable Volume	Eppendorf	Series 2000	2754148	2010
1913	Pipettor	Reference Adjustable Volume	Eppendorf	Series 2000	2754218	2010
1914	Thermometer	Digital	VWR	Cat. #21800-072	101494179	2010
1915	Thermometer	Alcohol	VWR	61222-500	T 105668	2010
1916	Thermometer	Blue Spirit	VWR	89095-626	A27630	2010
1917	Thermometer	Blue Spirit	VWR	89095-626	A27136	2010
1918	Thermometer	Blue Spirit	VWR	89095-626	A27212	2010
1919	Thermometer	Blue Spirit	VWR	89095-626	A27343	2010
1920	Thermometer	Blue Spirit	VWR	89095-626	A27248	2010
1921	MS-16	Water/Soil Autosampler	OI Corporation	4552	MS1003W023	2010
1922	Conductivity Probe	Conductivity Meter Probe	Orion	11510	Lot OX7-10019	2010
1923	Pipettor	1-10mL	Oxford	Benchmate II	H71013692	2010
1924	MS-16	Sampler Concentrator	OI Corporation	4660	E008466762P	2010
1925	IC3	Ion Chromatograph	Dionex	ICS1500	1598656	2010
1926	Oven	Used for FOC	Ney	M-525	AKN 9238 166	1989
1927	Thermometer	Red Spirit	VWR	Cat. #89095-574	NA	2010
1928	Thermometer	Red Spirit	VWR	Cat. #89095-574	NA	2010
1929	Thermometer	Red Spirit	VWR	Cat. #89095-574	NA	2010
1930	MS-16	GC	Agilent	7820A	CN10202030	2010
1931	MS-16	MS	Agilent	5975	US10200403	2010
1932	MS-16	Computer	HP Compaq	dc7900 Small Form Factor	MXL0060YSL	2010
1933	MS-16	Monitor	HP Compaq	HPLE1901w	3CQ011NB9	2010
1934	Waterbath	Hexavalent Chromium	Lab Line	18007	0202-0011	2010
1935	Timer	Talking	VWR	Cat. 62379-565	101558247	2010
1936	Injector	Injector (Tower)	HP	18593B	3531A43472	2010
1937	Shaker Table	Environ Shaker	Lab Line	3527	9012000	2010
1938	Thermometer	Digital	Fisher	15-077-941	72071658	2010
1939	20 mg Weight	Aluminum, Class 1	CMI	NVLAP Wt. Calibration		2010
1940	Thermometer	Mercury	VWR	61013-072	M080750	2010
1941	Thermometer	Red Spirit	Fisher	15-041-13A		2010
1943	Hot Plate		Presto			2010
1944	Hot Plate		Sunbeam			2010
1945	Stir Plate	Yellow/White color	Barnstead/Thermolyne	S46725	776960817570	2002
1946	Hot Plate	Yellow/White color	Barnstead/Thermolyne	Model # HP47135	611950687583	2004

1947	Stir Plate	Burgundy color	Fisher	Cat. 11-500-7S	696920805574	2001
1948	Stirrer	Blue/White (BOD/Titrations)	Fisher	120M		2001
1949	Vaccuum Pump	for TSS	Gast	0522-V4F-G1800X	0191	1996
1950	UV Lamp		Spectroline	EA-160		2003
1951	Imhoff Cone	1 Liter Plastic				2002
1952	Imhoff Cone	1 Liter Plastic				2002
1953	Thermometer	Traceable Ultra Waterproof	VWR	37000-414	111472026	2011
1954	Thermometer	Traceable Ultra Waterproof	VWR	37000-414	111472020	2011
1955	MS-8	Autosampler	EST Centurion	Cents221031111	416080003183	2011
1957	Rotator	Tumbler	Dayton	Motor 1LPP4		2005
1958	Thermometer	Dial	HACH	Block Digestor-Ammonia		2011
1959	pH Probe	pH Meter Probe	Orion	Lot #PW1	None (See 1119)	2011
1960	100 g weight	ASTM E617-97 Class 1	Troemner	7017-1W	1000045228	
1961	10 g weight	ASTM E617-97 Class 1	Troemner	7021-1W	1000045229	
1962	1 g weight	ASTM E617-97 Class 1	Troemner	7025-1W	1000045230	
1963	1 mg	ASTM E617-97 Class 1	Troemner	7037-1W	1000045231	
1964	100 mg	ASTM E617-97 Class 1	Troemner	7029-1W	1000046206	
1965	Thermometer	Digital	Fisher	Cat. 15-077-941	72071677	
1966	Hot Plate	Hot Plate	Hamilton Beach			2011
1967	ICP-Varian	Chiller	Lytron			2010

Appendix IV - Chain of Custody



APPENDIX V

APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

Definition

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

Procedure

1. Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

(1) Obtain another sample with a lower level of analyte in the same matrix if possible.

(2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each

through the entire method, including blank measurements as described above in 4a. Evaluate these data:

(1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

(2) If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

EC15NO91.208

where:

X_i; i=1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from i=1 to n.

6. (a) Compute the MDL as follows:

$$MDL = T_{(n-1,1-\alpha=0.99)} \quad (S)$$

where:

MDL = the method detection limit

$t_{(n-1,1-\alpha=.99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (χ^2/df).

LCL = 0.64 MDL

UCL = 2.20 MDL

where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step 4.

(b) If this is the second or later iteration of the MDL calculation, use S^2 from the current MDL calculation and S^2 from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger S^2 into the numerator S^2_A and the other into the denominator S^2_B. The computed F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: if $S^2_A/S^2_B<3.05$, then compute the pooled standard deviation by the following equation:

EC15NO91.209

if $S^2_A/S^2_B>3.05$, respike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the S_{pooled} as calculated in 7b to compute The final MDL according to the following equation:

$$MDL=2.681\ (S_{pooled})$$

where 2.681 is equal to $t_{(12,1-\alpha=.99)}$.

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from precentiles

of the chi squared over degrees of freedom distribution.

LCL=0.72 MDL
UCL=1.65 MDL

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS' T VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of replicates	Degrees of freedom (n-1)	$t_{(n-1,.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
00	00	2.326

Reporting

The analytical method used must be specifically identified by number or title ald the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery.

If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

[49 FR 43430, Oct. 26, 1984; 50 FR 694, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986]

APPENDIX C TO PART 136—INDUCTIVELY COUPLED PLASMA—ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7

1. Scope and Application

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, and domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L. (See Section 5.)

1.3 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps must be taken to correct for potential interference effects. (See Section 5.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other elements may be added as more information becomes available and as required.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instruction provided by the manufacturer of the particular instrument.

2. Summary of Method

2.1 The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate corrections made.

APPENDIX VI
QUALITY ASSURANCE MANUAL TRAINING SUMMARY

Quality Assurance Manual Date and Revision Number:
Revision 15; February 21, 2011

Initial each section as reviewed. Please complete and return this form to Technical Director for placement in Employee's Training File:

_____ Section 3.0, Statement of Policy
_____ Section 4.0, Organization
_____ Section 5.0, Quality Assurance Program
_____ Section 6.0, Sample Bottle Preparation
_____ Section 7.0, Custody of Samples, Equipment and Supplies
_____ Section 8.0, Analytical Procedures
_____ Section 9.0, Calibration Procedures and Frequency
_____ Section 10.0, Preventative Maintenance
_____ Section 11.0, Quality Control Checks & Routines to Assess Precision, Accuracy & MDLs
_____ Section 12.0, Data Reduction, Review and Reporting
_____ Section 13.0, Corrective Action
_____ Section 14.0, Performance and System Audits
_____ Section 15.0, QA Reports to Management
_____ Section 16.0, Reagent Storage and Documentation
_____ Section 17.0, Waste Disposal
_____ Appendix I, Waste Disposal Procedures
_____ Appendix II, Lab Equipment Preventive Maintenance Schedule
_____ Appendix III, Lab Equipment List
_____ Appendix V, 40 CFR Part 136, Method Detection Limit
_____ Appendix VI, QA Manual Training Summary
_____ Appendix VII, Corrective Action Form
_____ Appendix IX, List of all methods under which lab is Accredited
_____ Appendix X, PCM Asbestos Program Specific Requirements

Comments:_____
_____
_____
_____

Print Name: _____ Date:_____

Signature: _____ Date:_____

Supervisor:_____ Date:_____

Technical Director:_____ Date:_____

Quality Assurance Manager:_____ Date:_____

APPENDIX VI
QUALITY ASSURANCE MANUAL TRAINING SUMMARY NON-TECHNICAL

Quality Assurance Manual Date and Revision Number:
Revision 15; February 21, 2011

Initial each section as reviewed. Please complete and return this form to Technical Director for placement in Employee's Training File:

_____ Section 3.0, Statement of Policy
_____ Section 4.0, Organization
_____ Section 5.0, Quality Assurance Program
_____ Section 6.0, Sample Bottle Preparation
_____ Section 7.0, Custody of Samples, Equipment and Supplies
_____ Section 13.0, Corrective Action
_____ Section 14.0, Performance and System Audits
_____ Section 16.0, Reagent Storage and Documentation
_____ Section 17.0, Waste Disposal
_____ Appendix I, Waste Disposal Procedures
_____ Appendix VII, Corrective Action Form
_____ Appendix IX, List of all methods under which lab is Accredited

Comments:_____
_____
_____
_____

Print Name: _____ Date:_____

Signature: _____ Date:_____

Supervisor:_____ Date:_____

Technical Director:_____ Date:_____

Quality Assurance Manager:_____ Date:_____

APPENDIX VII

Analytical Environmental Servs, Inc.

Corrective Action Report

Date Initiated: **Corrective Action Report ID:** 46616
Initiated By: **Department:** ad

Corrective Action Description

CAR Summary:

Description of Nonconformance:

Description of Corrective Action:

Performed By: **Completion Date:**

Client Notification

Client Notification Required: No **Notified By:**

Comment:

Quality Assurance Review

Nonconformance Type: Deficiency

Further Action required by QA:

Approval and Closure

Technical Director / Deputy Tech. Dir.: Close Date:

QA Manager Approval: QA Date:

APPENDIX IX - List of all methods under which lab is Accredited

TNI Methods			
Matrix	**Category**	**Method**	**Description**
Potable or Drinking Water (Safe Drinking Water Act - SDWA)			
PW	Microbiology	SM 9223 B	Total Coliforms & E. coli
Non-Potable Water (Clean Water Act - CWA)			
NPW	Vol Organics	EPA 8011	EDB & DBCP
NPW	Vol Organics	EPA 8015	Gasoline range organics (GRO)
NPW	Vol Organics	EPA 8015	Various Nonhalogenated Volatile Compounds
NPW	Ext Organics	EPA 8015	Diesel range organics (DRO)
NPW	Ext Organics	FL-PRO	Total Petroleum Hydrocarbons (TPH)
NPW	Ext Organics	EPA 610	Polynuclear Aromatic Hydrocarbons (PAHs)
NPW	Ext Organics	EPA 8310	Polynuclear Aromatic Hydrocarbons (PAHs)
NPW	Ext Organics	EPA 8315	Formaldehyde
NPW	Ext Organics	EPA 625	Semi-Volatile (Base-Neutral-Acid) Organics
NPW	Ext Organics	EPA 8270	Semi-Volatile (Base-Neutral-Acid) Organics
NPW	Gen Chem	EPA 1010	Ignitability
NPW	Gen Chem	EPA 110.2	Color
NPW	Gen Chem	EPA 120.1	Conductivity
NPW	Gen Chem	EPA 150.1	pH
NPW	Gen Chem	EPA 160.1	Residue-filterable (TDS)
NPW	Gen Chem	EPA 160.2	Residue-nonfilterable (TSS)
NPW	Gen Chem	EPA 160.3	Residue-total
NPW	Gen Chem	EPA 160.4	Residue-volatile
NPW	Gen Chem	EPA 160.5	Residue-settleable
NPW	Gen Chem	EPA 1664A	Oil & Grease
NPW	Gen Chem	EPA 1664A	Total Petroleum Hydrocarbons (TPH)
NPW	Gen Chem	EPA 180.1	Turbidity
NPW	Gen Chem	EPA 300.0	Ion Scan
NPW	Gen Chem	EPA 305.1	Acidity as CaCO3
NPW	Gen Chem	EPA 310.1	Alkalinity as CaCO3
NPW	Gen Chem	EPA 310.2	Alkalinity as CaCO3
NPW	Gen Chem	EPA 325.2	Chloride
NPW	Gen Chem	EPA 330.5	Residual free chlorine
NPW	Gen Chem	EPA 335.1	Amenable cyanide
NPW	Gen Chem	EPA 335.2	Cyanide
NPW	Gen Chem	EPA 350.1	Ammonia as N
NPW	Gen Chem	EPA 351.2	Kjeldahl nitrogen - total
NPW	Gen Chem	EPA 353.2	Nitrate as N
NPW	Gen Chem	EPA 353.2	Nitrate-nitrite
NPW	Gen Chem	EPA 353.2	Nitrite as N
NPW	Gen Chem	EPA 354.1	Nitrite as N
NPW	Gen Chem	EPA 360.1	Oxygen dissolved
NPW	Gen Chem	EPA 365.1	Orthophosphate as P
NPW	Gen Chem	EPA 365.1	Phosphorus total
NPW	Gen Chem	EPA 365.3	Orthophosphate as P
NPW	Gen Chem	EPA 370.1	Silica-dissolved

NPW	Gen Chem	EPA 375.4	Sulfate
NPW	Gen Chem	EPA 376.1	Sulfide
NPW	Gen Chem	EPA 377.1	Sulfite-SO3
NPW	Gen Chem	EPA 405.1	Biochemical oxygen demand
NPW	Gen Chem	EPA 410.4	Chemical oxygen demand
NPW	Gen Chem	EPA 415.1	Total organic carbon
NPW	Gen Chem	EPA 420.1	Total phenolics
NPW	Gen Chem	EPA 420.4 (420.2)	Total phenolics
NPW	Gen Chem	EPA 425.1	Surfactants - MBAS
NPW	Gen Chem	EPA 7196	Chromium VI
NPW	Gen Chem	EPA 9010/9012	Total cyanide
NPW	Gen Chem	EPA 9014	Total cyanide
NPW	Gen Chem	EPA 9030/9034	Sulfide
NPW	Gen Chem	EPA 9038	Sulfate
NPW	Gen Chem	EPA 9040	Corrosivity (pH)
NPW	Gen Chem	EPA 9040	pH
NPW	Gen Chem	EPA 9050	Conductivity
NPW	Gen Chem	EPA 9056	Ion Scan
NPW	Gen Chem	EPA 9060	Total organic carbon
NPW	Gen Chem	EPA 9065	Total phenolics
NPW	Gen Chem	SM 10200 H	Chlorophylls
NPW	Gen Chem	SM 2120 B	Color
NPW	Gen Chem	SM 2120 E	Color
NPW	Gen Chem	SM 2320 B	Alkalinity as CaCO3
NPW	Gen Chem	SM 2340 B	Hardness
NPW	Gen Chem	SM 2540 B	Residue-total
NPW	Gen Chem	SM 2540 C	Residue-filterable (TDS)
NPW	Gen Chem	SM 2540 D	Residue-nonfilterable (TSS)
NPW	Gen Chem	SM 2540 G	Total fixed and volatile residue
NPW	Gen Chem	SM 2540 F	Residue-settleable
NPW	Gen Chem	SM 2710 B	Specific Oxygen Uptake Rate (SOUR)
NPW	Gen Chem	SM 3500-Cr B	Chromium VI
NPW	Gen Chem	SM 3500-Fe B	Iron
NPW	Gen Chem	SM 5210 B	Biochemical oxygen demand
NPW	Gen Chem	SM 4500Cl G	Residual free chlorine
NPW	Gen Chem	SM 4500CN E (335.4)	Cyanide
NPW	Gen Chem	SM 4500CN G	Amenable cyanide
NPW	Gen Chem	SM 4500-H+B	pH
NPW	Gen Chem	SM 4500NO2 B	Nitrite as N
NPW	Gen Chem	SM 4500 S F	Sulfide
NPW	Gen Chem	SM 4500 SO3	Sulfite-SO3
NPW	Gen Chem	SM 5210 B	Carbonaceous BOD (CBOD)
NPW	Gen Chem	SM 5310 B	Total organic carbon
NPW	Gen Chem	TKN - AMMONIA	Organic nitrogen
NPW	Metals	EPA 200.7	Metals
NPW	Metals	EPA 6010	Metals
NPW	Metals	EPA 200.8	Metals
NPW	Metals	EPA 6020	Metals
NPW	Metals	EPA 245.1	Mercury

NPW	Metals	EPA 7470	Mercury
NPW	Microbiology	SM 9222 B	Total Coliforms
NPW	Microbiology	SM 9222 D	Fecal Coliforms
NPW	Pest-Herb-PCB	EPA 608	Pesticides & Polychlorinated Biphenyls
NPW	Pest-Herb-PCB	EPA 608.2	Methoxychlor
NPW	Pest-Herb-PCB	EPA 8081	Pesticides
NPW	Pest-Herb-PCB	EPA 8082	Polychlorinated Biphenyls
NPW	Pest-Herb-PCB	EPA 615	Herbicides
NPW	Pest-Herb-PCB	EPA 8151	Herbicides
NPW	Vol Organics	EPA 624	Volatile Organics
NPW	Vol Organics	EPA 8260	Volatile Organics
NPW	Vol Organics	AES SOP OA-11010	Oxygenates

Solids & Hazardous Materials (Resource Conservation & Recovery Act - RCRA)			
Solids	Vol Organics	EPA 8015	Gasoline range organics (GRO)
Solids	Vol Organics	EPA 8015	Various Nonhalogenated Volatile Compounds
NPW	Ext Organics	EPA 8015	Diesel range organics (DRO)
Solids	Ext Organics	FL-PRO	Total Petroleum Hydrocarbons (TPH)
Solids	Ext Organics	EPA 8310	Polynuclear Aromatic Hydrocarbons (PAHs)
Solids	Ext Organics	EPA 8315	Formaldehyde
Solids	Ext Organics	EPA 8270	Semi-Volatile (Base-Neutral-Acid) Organics
Solids	Gen Chem	EPA 350.1 in Soil	Ammonia
Solids	Gen Chem	EPA 351.2 in Soil	Kjeldahl nitrogen - total
Solids	Gen Chem	EPA 365.1 in Soil	Total Phosphorus
Solids	Gen Chem	EPA 1010	Ignitability
Solids	Gen Chem	EPA 1311	TCLP
Solids	Gen Chem	EPA 1312	SPLP
Solids	Gen Chem	EPA 7196	Chromium VI
Solids	Gen Chem	EPA 9010/9014	Total cyanide
Solids	Gen Chem	EPA 9014	Total cyanide
Solids	Gen Chem	EPA 9030/9034	Sulfide
Solids	Gen Chem	EPA 9040	pH
Solids	Gen Chem	EPA 9045	pH
Solids	Gen Chem	EPA 9050	Conductivity
Solids	Gen Chem	EPA 9056	Ion Scan
Solids	Gen Chem	EPA 9060	Total organic carbon
Solids	Gen Chem	EPA 9065	Total phenolics
Solids	Gen Chem	EPA 9071	Oil & Grease
Solids	Gen Chem	EPA 9081	Cation exchange capacity
Solids	Gen Chem	EPA 9095	Paint Filter Liquids Test
Solids	Gen Chem	Sec. 7.3 SW-846	Reactive cyanide
Solids	Gen Chem	Sec. 7.3 SW-846	Reactive sulfide
Solids	Metals	EPA 6010	Metals
Solids	Metals	EPA 6020	Metals
Solids	Metals	EPA 7471	Mercury
Solids	Pest-Herb-PCB	EPA 8081	Pesticides
Solids	Pest-Herb-PCB	EPA 8082	Polychlorinated Biphenyls

Solids	Pest-Herb-PCB	EPA 8151	Herbicides
Solids	Vol Organics	AES SOP OA-11010	Oxygenates
Solids	Vol Organics	EPA 8260	Volatile Organics
	AIHA Methods		
Air	Ext Organics	N1003	Hydrocarbons, Halogenated
Air	Ext Organics	N1300	Ketones
Air	Ext Organics	N1400	Alcohols I
Air	Ext Organics	N1450	Esters I
Air	Ext Organics	N1457	Ethyl acetate
Air	Ext Organics	N1500	Hydrocarbons, BP 36-126°C
Air	Ext Organics	N1501	Hydrocarbons, Aromatic
Air	Ext Organics	N1550	Naphthas in Air
Air	Ext Organics	N2000	Methanol
Air	Ext Organics	N2500	2-Butanone
Air	Ext Organics	N5506	Polynuclear Aromatic Hydrocarbons by HPLC
Air	Ext Organics	3M3520/SKC575	Organic Vapors on Passive Monitor
Air	Metals	N7300	Elements by ICP
Air	Metals	N6009	Mercury
Solids	Metals	N7082	Lead in Paint
Solids	Metals	SW3050B/7420	Total Lead in Solids
Solids	Metals	SW3050B/7000B	Total Lead in Solids
Air	Metals	N9102	Lead on Wipes
Air	Asbestos	N7400	PCM
Air	Gen Chem	N0500/0600	Particulates
Air	Microbiology	Fungal Air Culturable	MB - 15024
Air	Microbiology	Fungal Bulk Culturable	MB - 15023
Air	Microbiology	Fungal Surface Culturable	MB - 15023
Air	Microbiology	Fungal Air Direct Exam	MB - 15019, MB - 15022, MB - 15028
Air	Microbiology	Fungal Bulk Direct Exam	MB - 15020
Air	Microbiology	Fungal Surface Direct Exam	MB - 15020

Attachment 5

Quality Assurance Manual Acceptance Agreement

The information in this Quality Assurance Manual including its tables, appendices, figures, and / or attachments may be legally privileged and is confidential information intended for the use of reviewing Analytical Environmental Services Quality System policies and procedures. You are hereby notified that any dissemination, distribution, or copy of this manual or information therein including tables, appendices, figures, and / or attachments is strictly prohibited without written permission from a representative of Analytical Environmental Services Customer Service Department. If you have received this manual in error, please notify Analytical Environmental Services Customer Service by telephone at (770) 457-8177 for instructions on returning the document. If an electronic copy has been received in error by email, contact info@aesatlanta.com and delete the message. Thank you.

I have read, understood and agree to comply with the above statement.

_____ _____
Signature Date

_____
Printed Name

_____
Company

_____
Phone Number with extension

APPENDIX X

New Employee Initial Quality Assurance Manual Training

TRAINING: Initial Training on AES SOP #QA-01000, "SOP for the Quality Assurance Manual"

My signature confirms that I attended the initial training of the company's Quality Assurance Manual, which includes a discussion of the various sections contained within as well as responsibilities I have while performing my daily duties. I will be reading various sections of that document according to my job function. Upon completion I will sign-off on form 'Appendix VI – Quality Assurance Manual Training Summary'.

Supervisor:
_____

Section/area:
_____

Print Name:
_____

Employee Signature:
_____

Date: _____

APPENDIX XI
Outside Reference Documents

1. 2003 NELAC Standards, National Environmental Laboratory Accreditation Conference (NELAC), EPA 600/R-041-003, June 5, 2003, **www.nelac-institute.org**.

2. *AIHA Policy Modules for AIHA Laboratory Accreditation Programs*, current revisions posted to web, **www.aiha.org**.

3. American National Standard, *General Requirements for the Competence of Testing and Calibration Laboratories*, ANSI/ISO/IEC 17025:2005.

4. North Carolina Administrative Code, Title 15: Department of Environment, Health and Natural Resources; Chapter 2, Environmental Management Division; Subchapter 2H; Procedures for Permits, Approvals; Section .0800; Laboratory Certification, August 1, 2002, Environmental Management Commission, Raleigh, North Carolina, **www.esb.enr.state.nc.us/**.

5. *Groundwater Section Guidelines for the Investigation and Remediation of Soil and Groundwater*, NCDENR Division of Water Quality, Groundwater section, July 2000, **www.gw.ehnr.state.nc.us**/.

6. *Groundwater Section Guidelines for the Investigation of Soil and Groundwater contamination: Chlorinated Solvents and other Dense Non-Aqueous Phase Liquids, (An addendum to the "Groundwater Section Guidelines for the Investigation and Remediation of Soil and Groundwater*, NCDENR Division of Water Quality, Groundwater section, July 2000"), NCDENR Division of Water Quality, Groundwater section, July 2003, **www.gw.ehnr.state.nc.us**/.

7. *Analytical Methodology for Groundwater and Soil Assessment Guidelines*, SCDHEC UST Program Guidance document, August 24, 2005, http://www.scdhec.gov/environment/admin/htm/eqcguide.shtml.

8. Solutions to Analytical Chemistry Problems with Clean Water Act Methods, EPA 821-R-07-002 (revision to the "Pumpkin Document", EPA 821-B-93-001), March 2007, **www.epa.gov/waterscience/methods/**.

9. *Code of Federal Regulations, Title 40, Part 136*, U. S. Government Printing Office: Washington DC, current revision posted on the web, **www.epa.gov**.

10. Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition, EPA 815-R-05-004, January 2005, **www.epa.gov/safewater/methods/laboratorycertification.html**.

11. Supplement 1 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water, EPA 815-F-08-006, June 2008, **www.epa.gov/safewater/methods/laboratorycertification.html.**

12. Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March 1983.

13. Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA 600/R-94/111, May 1994.

14. Methods for the Determination of Inorganic Substances in Environmental Samples, EPA 600/R-93/100, August 1993.

15. HACH Procedures Manual, Seventh Edition, *Chemical Oxygen Demand, Method 8000*, HACH Chemical Company: Loveland, CO, 1999.

16. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition, American Public Health Association, Washington, DC, 1992.

17. Standard Methods for the Examination of Water and Wastewater, Nineteenth Edition, American Public Health Association, Washington, DC, 1995.

18. Standard Methods for the Examination of Water and Wastewater, Twentieth Edition, American Public Health Association, Washington, DC, 1998.

19. Standard Methods for the Examination of Water and Wastewater, Twenty First Edition, American Public Health Association, Washington, DC, 2005.

20. Methods and Guidance for Analysis of Water, *The Determination of Chlorinated Herbicides in Municipal and Industrial Wastewater, Method 615*, EPA 821-C-99-004, June 1999.

21. EASY DIST Manual of Environmental Methods, Rev. 9/5/1996.

22. Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-Polar Material) by Extraction and Gravimetry, EPA 821-R-98-002, February 1999, **www.epa.gov/waterscience/methods/method/oil**.

23. Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure, *Total Petroleum Hydrocarbons (TPH) Analysis - Gasoline and Diesel*, State of California Leaking Underground Fuel Tank Task Force, October 1989.

24. *Method for the Determination of Extractable Petroleum Hydrocarbons by GC/FID*, State of Tennessee Department of Environment and Conservation, Division of Underground Storage Tanks, current revision posted to web, **www.state.tn.us/environment/ust/groeph.shtml**.

25. *Method for Determination of Petroleum Range Organics, Method # FL-PRO*, Florida Department of Environmental Protection, Revision 1, November 1, 1995, **www.dep.state.fl.us/**.

26. Test Methods for Evaluating Solid Waste, Third Edition, SW-846 (including Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA, and IVB), US EPA Office of Solid Waste and Emergency Response: Washington, DC, December 1996 (Update IIIA, April 1998), **www.epa.gov**.

27. Methods of Soil Analysis, No. 5, Part 2, *Section 10-2 Saturation Extract and Other Aqueous Extracts, Chemical and Microbiological Properties, Second Edition*, American Society of Agronomy, Inc., 1982.

28. ASTM Standards, latest editions, **www.astm.org**.

29. NIOSH Manual of Analytical Methods, Fourth Edition, US Department of Health and Human Services, Cincinnati, Ohio, August 1994, **www.cdc.gov/niosh/**.

30. *Code of Federal Regulations, Title 40, Part 60 Appendix A, Test Method 18, VOC by GC*, U. S. Government Printing Office: Washington DC, current revision posted on the web, **www.epa.gov**.

31. Laboratory Guide to Common Aspergillus Species and Their Telemorphs, Klich, Maren, and John Pitt, CISRO Food Research Laboratory, 1988.

32. Identifying Filamentous Fungi, A Clinical Laboratory Handbook, St-Germain, Guy, and Richard Summerbell, 1996.

33. Environmental Monitoring Services Recommendations for Identification and Quantification of Airborne Fungal Spores, Hyphae, Skin Fragments, Pollen, Fibrous particulaes, and Arthropod (insect) Fragments, Revision 110402.

34. McCrone Research Institute of Chicago, IL, Recommendations for Identification and Quantification of Airborne Fungal Spores, Hyphae, and Pollen as instructed in Course 1630: Indoor air Quality: Fungal Spore Identification.

35. Environmental Monitoring Services Micro5 Analysis Standard Operating Procedure for Examining 100% of Total Trace, Revision 11/4/02.

36. Standards of Practice for the Assessment of Indoor Environmental Quality, Indoor Environmental Standards Organization, Volume 1, First Edition, April 2002.

APPENDIX XII

Environmental Microbiology Laboratory Accreditation Program (EMLAP) Specific Requirements

1.0 INTRODUCTION

Analytical Environmental Services, Inc. is dedicated to providing quality analytical services. Analytical Environmental Services, Inc. (AES) specializes in the analysis of microorganisms commonly detected in air (e.g., spore trapping), surface (e.g., tape lifts, swabs, wipes), and bulk (e.g., wallboard, carpet, building materials) samples collected from schools, hospitals, offices, industrial, agricultural, and other work environments. AES has implemented a quality assurance and quality control (QA/QC) program to establish quality control standards necessary for compliance to guidelines by The American Industrial Hygiene Association's (AIHA) Environmental Microbiology Laboratory Accreditation Program (EMLAP). In order to consistently maintain high standards of precision and accuracy in analytical testing, AES participates in AIHA's Proficiency Analytical Testing (PAT) program.

This quality assurance plan will establish the procedures that will be followed to ensure accuracy, precision, completeness, and representation of data obtained from the analysis of environmental microbiology samples.

2.0 PURPOSE

AES has implemented a quality assurance, quality control program for the purpose of providing a baseline of standards which will allow for a continuous surveillance quality performance for the benefit of AIHA EMLAP compliance, client satisfaction, and minimization of liability.

3.0 SCOPE

This QA/QC program provides the necessary guidelines to secure and maintain:

- High level of quality work
- Comprehensive accountability of all activities relevant to laboratory services.
- Continuous compliance with ISO/IEC 17025 and AIHA's EMLAP quality requirements.

This QA/QC program includes the following information:

- Comprehensive system of daily, weekly, monthly, and annual record keeping.
- Definition of routine monitoring activities.
- Sampling techniques for air, surface, and bulk collection.
- Sampling Equipment
- Calibration of Sampling Equipment
- Analysis of Air, Surface, and Bulk samples.
- Analytical Equipment
- Calibration of Analytical Equipment
- In-House training of analysts.
- QA/QC activities within lab.

4.0 FACILITIES

The laboratory has adequate facilities for the scope of services and meets the requirements for the most current and relative biosafety guidelines set forth by CDC, WHO, and AIHA. The lab has a documented routine monitoring program for the verification of adequate contamination control. The laboratory has the proper facilities for biological and chemical storage and disposal of refuse.

5.0 EQUIPMENT

Microscope/Magnification System

- Microscope/Magnification System consisting of Compound optical microscope with a high magnification (100x) oil immersion objective having a numerical aperture (n.a.) of at least 1.25.
- Alignment of each microscope shall be documented with each day of use.
- Each microscope shall have an ocular micrometer that shall be checked annually with a NIST traceable stage micrometer.
- The Field of View Diameter for each objective on the microscope shall be checked annually.

Class II Biological Safety Cabinet

- Performance certified annually according to NSF Standard 49.

Steam Sterilizer/Autoclave

- An autoclave with functioning temperature and pressure gauges for the disposal of potentially viable waste.
- Routine use of indicators to document successful sterilization with each use.
- Routine use of biological indicators to document the sterilization process.

Incubators and Refrigerators

- Temperature settings appropriate for the scope of testing.
- Temperatures recorded twice daily.

6.0 PERSONNEL

The laboratory conforms to the personnel requirements of the AIHA EMLAP guidelines. In all cases training records for degreed laboratory staff shall include a copy of transcript or diploma from an accredited college/university.

Technical Manager

- The laboratory shall be under the overall direction of an onsite, qualified person, who for the purposes of this document, is designated as the Technical Manager, and has the responsibility for the function, administration, and day-to-day operation of the laboratory. The Technical Manager or designee shall serve as the approved signatory.
- The Technical Manager shall have an earned microbiology or life science degree, minimally at the baccalaureate level, with the required combination of semester hours in microbiology and/or non-academic work experience as listed below. All non-academic work experience and coursework must be documented in the employee's training and personnel files.

(a) Microbiology degree and a minimum of two (2) years of full time equivalent documented environmental microbiological work experience (bacteriology and/or mycology).

(b) Life Science degree and:

i. Twenty (20) semester hours in Microbiology and a minimum two (2) years of full time equivalent documented environmental microbiological work experience (bacteriology and/or mycology).

ii. Sixteen (16) semester hours in Microbiology and a minimum three (3) years of full time equivalent documented environmental microbiological work experience (bacteriology and/or mycology).

iii. Twelve (12) semester hours in Microbiology and a minimum four (4) years of full time equivalent documented environmental microbiological work experience (bacteriology and/or mycology).

iv. Eight (8) semester hours in microbiology and a minimum of five (5) years of full time equivalent documented environmental microbiological experience (bacteriology and/or mycology).

(c) Experience must reflect the scope of work of the laboratory.

- The Technical Manager shall be experienced in the selection and the use of bioaerosol, surface, fluid, and raw material sampling methods and in sample processing for the quantification and identification appropriate to the FoTs of mesophilic and thermophilic bacteria, and mesophilic, xerophilic, thermo tolerant fungi (molds and yeasts), and fungi identified by spore trap collection methods.
- Training records for the Technical Manager shall include documentation of ability to identify genus/group of fungi from spore trap analysis and genus/species of fungi that are reported.

Laboratory Analytical Staff

The environmental microbiological program distinguishes two titles for those conducting analytical procedures within the laboratory. An analyst is one who has a bachelor's degree and a technician is one who does not have a degree.

Laboratory Technicians

- These staff members shall have a high school diploma or General Education Development (GED) During this required training period, the trainee shall perform work (and have work reviewed prior to release) under the direct supervision of a qualified technician, analyst and/or the Technical Manager.

- Technicians may function in the same manner as analysts for Air – Direct Examination (spore trap) analysis after completion of six (6) months documented on the job training and

demonstrated proficiency. For all other analyses, technicians may function in the same manner as analysts after one (1) year documented on the job training and demonstrated proficiency.

Laboratory Analysts

- These staff members shall have a bachelor's degree in a physical or biological science. Analysts shall have three (3) months of documented training for Air - Direct Examination (spore trap) and six (6) months of documented on-the-job training functioning for all other analyses as an analyst trainee. During the required analyst training period, the trainee shall be under the direct supervision of another qualified analyst and/or the Technical Manager. During this period, the trainee shall have all work reviewed prior to release by another qualified analyst and/or the Technical Manager. Training records for technicians and analysts shall include documentation of ability to identify genus/species of fungi and genus/group of fungi that are reported. Bacterial identification training records shall document training of relevant diagnostic procedures (e.g., gram stain, oxidase, biochemical reactions).

- All analysts and technicians shall have demonstrated ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples or in-house quality control samples. This demonstration shall be performed and documented at a minimum of every six (6) months.

Laboratory Quality Assurance Coordinator

- This Quality Assurance Coordinator (QAC) of the laboratory shall possess a bachelor's degree in an applicable basic or applied science and have six (6) months of non-academic relevant and documented microbiological laboratory analysis experience. In lieu of bachelor's degree, four years of non-academic analytical experience is acceptable.

- The QAC shall have documented training in statistics. Additional training may consist of quality control procedures.

7.0 ANALYTICAL METHODS

See SOP's

8.0 QUALITY ASSURANCE/QUALITY CONTROL

- Routine QA/QC procedures shall be an integral part of the laboratory procedures and functions. The laboratory is in compliance with APHA-AWWA-WPCF guidelines in *Standard Methods for the Examination of Water and Wastewater*, 20th Edition, APHA, 1998 for microbiology laboratories.

- Five (5) percent intra-analyst analysis shall be completed by each analyst to assess the precision of the analyst.

- Five (5) percent inter-analyst analysis shall be completed to assess the accuracy of the analysis performed within the laboratory.

- The laboratory shall use control charts or databases to compare intra- and inter-analyst analysis performance to established control charts.

- The laboratory shall ensure the quality control of culture media and analytical reagents per lot number for appropriate sterility, microbial growth, and/or analytical reactions. Records will be maintained and acceptance criteria will be documented.

- Acceptance Criteria on 5% replicate and duplicate analysis, daily reference slide analysis (spore traps) and monthly reference culture analysis will be documented and shall include the following:

  (a) Taxon identification acceptability
  (b) Taxon abundance ranking acceptability
  (c) Count of concentration acceptability determined statistically with use of control charts or databases (Spore Traps only).

- Laboratory will maintain routine records of temperature documentation for refrigerators and incubators. Acceptance criteria will be documented.

- The laboratory maintains a microbial culture collection of common organisms relevant to the methods performed. Cultures will be from recognized sources including EMPAT rounds. The culture collection will include the source and date of acquisition.

- The culture collection will be used monthly to prepare blind cultures to be used as part of the routine QC program to monitor accuracy in culture identification.

- The laboratory has a reference slide collection with various count levels and genera/groups of spores which is maintained and used as part of total spore analysis quality control.

- Each day of analysis, at least one slide from the collection shall be reviewed by each analyst. Slides are viewed on a rotational schedule so a different slide is viewed each day until the entire slide collection is examined. The analysis of these slides is incorporated into the daily QC plan. Acceptance criteria will be documented.

- Statistically derived control charts with control limits are used to assess performance.

- The laboratory participates and has documentation of a round robin slide exchange of real samples consistent with AIHA Policy 6A.3.2 *Requirements for Round Robin Programs*.

- Round robins include the participation of three (3) laboratories. Round robin program will consist of at least two (2) rounds per year, with each round completed within a 6-month timeframe.

- Each round will consist of four (4) samples at varying concentrations.

- Each analyst within the laboratory will analyze the samples independently and each of the analyst's results will be reported.

- The round robin analytical data will include raw counts and final concentrations for each fungal structure observed.

- Round Robin acceptance criteria shall include the organism identification, ranking, and quantification.

- A designated laboratory shall be responsible for data collection and distribution. The participating laboratories shall rotate this designation.

- A routine air monitoring program is used to verify adequate contamination control.

  (a) Two (2) spore trap samples are collected each month. One (1) inside sample and One (1) outside sample are collected and compared. Acceptance criteria will be documented.

SAFETY, HEALTH, ENVIRONMENTAL AND TRANSPORTATION REGULATIONS

Analytical Environmental Services, Inc. adheres to all applicable federal, state, and local regulations regarding safety, health, environment or transportation. Potentially viable microbial waste shall be collected in properly designated biohazard containers and disposed of properly through autoclaving.

Attachment 6

ANALYTICAL ENVIRONMENTAL SERVICES, INC.
ANNUAL MANAGEMENT REVIEW MEETING

REQUIRED ATTENDEES:

President	VP Operations
QA Manager	Technical Director
PCM Manager	Metals Lab Manager
Prep Lab Manager	Semi-Volatile Lab Manager
Sample Rec. Manager	Customer Service Manager
Micro Bio Lab Manager	Volatiles Lab Manager
H.R. Manager	Wet Chem Lab Manager
IC Manager	

The meeting will be conducted by the President or Vice President of Operations.

AGENDA

1. Follow Up-Actions from previous Management Review meetings.
    a. Changes in Policy and Procedures (QA)
    b. Facility Improvements (President/VP)

2. Quality Assurance Report:
    a. Accreditation Requirements (QA)
    b. Changes in Management Structure (President/VP)
    c. Changes/Expansion of laboratory Services (President/VP)
    d. New/Updates of Procedures/SOP's/Reference Materials (QA)

3. Review of Performance in Quality Areas
    a. Documentation (QA)
    b. Following SOP's (Technical Director)
    c. Calibration of Equipment (VP)
    d. Handling of failed QC Data (Each Department Supervisor provide an overall statement of finding these errors and how they are being handled in their department)
    e. Major PT Failure issues (QA)
    f. Calculations (QA)

g. Repeat and total number of deficiencies per department (Each Dept. Supervisor provide info. on repeat and total number of deficiencies related to a specific analysts or your dept. and how it is being handled, technical reprimands, etc.)

4. Managerial Reports
    a. Equipment Needs (Each Dept. Supervisor to provide info. on current equip. needs) (Mehmet update needs)
    b. Equipment Maintenance
        i. Calibration Information (VP)
        ii. Repair and maintenance data (VP)
        iii. Equipment downtime logs/review (Each Dept. Supervisor)
        iv. Resources
            1. Staffing Needs (VP)
            2. Department Training Needs (Technical Director)
            3. Facility and Equipment Needs (VP)

5. Internal Auditing
    a. Audit Results (QA)
    b. Audit Schedule (QA)
    c. Nonconformance by Department (HR)
    d. Results of Inter-Laboratory comparisons or proficiency (QA)

6. Corrective and Preventive Actions
    a. Type and source of issues (Each dept. Supervisor)
    b. Areas most commonly having problems (QA)
    c. Trends of root causes (QA)
    d. Reoccurring problems (QA)
    e. Summary and review of corrective action log (QA)
        i. Effectiveness
        ii. Cooperation
        iii. Closure

7. External Audit
    a. Performance Evaluation for Quality System and Technical Aspects (VP)
    b. Evaluation common weak areas from each auditing agency (QA)

8. Quality Planning
    a. Upcoming projects (Customer Service Manager)
    b. Status of ongoing projects (Customer Service Manager)
    c. Significant changes including staff/equipment/required accreditations (VP)

9. Customer Feedback (Customer Service Manager)
    a. Customer complaints
        i. Review of Customer Complaint Corrective Action Logs
            1. Repeated complaints
            2. Related/Unrelated issues
            3. Cause of issues identified and corrective measures followed
            4. Weekly meeting review
    b. Client satisfaction survey

10. Improvements (President/VP)
    a. Review of Quality Policy/Objectives
    b. Review of Quality Systems effectiveness and improvement of system and services
    c. Detail and assign responsible party time line for implementation of task.

APPENDIX XIII

PCM Asbestos Quality Program Requirements

1.0 INTRODUCTION

Analytical Environmental Services, Inc. is dedicated to providing quality analytical services. Analytical Environmental Services, Inc. (AES) specializes in the analysis of asbestos by Phase Contrast Microscopy (PCM) samples collected from schools, hospitals, offices, industrial, and other work environments. AES has implemented a quality assurance and quality control (QA/QC) program to establish quality control standards necessary for compliance to guidelines by The American Industrial Hygiene Association's (AIHA) Industrial Hygiene Laboratory Accreditation Program (IHLAP). In order to consistently maintain high standards of precision and accuracy in analytical testing, AES participates in AIHA's Proficiency Analytical Testing (PAT) program.

This quality assurance plan will establish the procedures that will be followed to ensure accuracy, precision, completeness, and representation of data obtained from the analysis of environmental microbiology samples.

2.0 PURPOSE

AES has implemented a quality assurance, quality control program for the purpose of providing a baseline of standards which will allow for a continuous surveillance quality performance for the benefit of AIHA IHLAP compliance, client satisfaction, and minimization of liability.

3.0 SCOPE

This QA/QC program provides the necessary guidelines to secure and maintain:

- High level of quality work
- Comprehensive accountability of all activities relevant to laboratory services.
- Continuous compliance with ISO/IEC 17025 and AIHA's IHLAP quality requirements.

This QA/QC program includes the following information:

- Comprehensive system of daily, weekly, monthly, and annual record keeping.
- Definition of routine monitoring activities.
- Analysis of air sample filters to determine concentrations of airborne fibers.
- Analytical Equipment
- Calibration of Analytical Equipment
- In-House training of analysts.
- QA/QC activities within lab.

4.0 FACILITIES

The laboratory has adequate facilities for the scope of services and meets the requirements for the most current and relative guidelines set forth by AIHA. The lab has a documented routine monitoring program for the verification of adequate contamination control. The laboratory has the proper facilities for biological and chemical storage and disposal of refuse.

5.0 EQUIPMENT

Microscope/Magnification System

- Phase Contrast Microscope – NIKON Alphaphot – 2, with 10x & 40x objectives; Walton – Beckett graticule with 100-mm diameter (Type –G-22); Green filter.
- Telescope ocular phase-ring centering
- Stage micrometer (0.01-mm divisions)
- HSE / NPL phase contrast test slide
- Alignment of each microscope shall be documented with each day of use.
- Each microscope shall have an ocular micrometer that shall be checked annually with a NIST traceable stage micrometer.
- The Field of View Diameter for each objective on the microscope shall be checked annually.

6.0 PERSONNEL

The laboratory conforms to the personnel requirements of the AIHA IHLAP guidelines. In all cases training records for degreed laboratory staff shall include a copy of transcript or diploma from an accredited college/university.

Technical Manager

- Qualifications of the laboratory Technical Manager are a bachelor's degree in an applicable physical or biological science, and a minimum of three (3) years relevant nonacademic analytical chemistry experience. A minimum of two (2) years experience must be in industrial hygiene analyses within the scope of accreditation. The remaining one (1) year can be from other laboratory analytical procedures. Relevant academic experience may be substituted for work experience. A relevant master's degree shall be considered equivalent to one (1) year of work experience. (Environmental, forensic, or similar microanalytical experience shall be reviewed to determine if the specific experience is a reasonable substitute.)

- The Technical Manager shall be available at least 50 percent of the laboratory operating hours to address technical issues for laboratory staff and customers. The Technical Manager shall authorize and document that all analyses for which the laboratory is accredited are completed by personnel with appropriate education and/or technical background. The Technical Manager shall ensure that adequate supervision is provided for all laboratory technical personnel.

Laboratory Analytical Staff

- The industrial hygiene program distinguishes two titles for those conducting analytical procedures within the laboratory. An analyst is one who has a bachelor's degree in chemistry or a related science. A technician is one who does not have a degree in chemistry or a related science.

- All analysts and technicians shall complete a training course (an in-house course is acceptable) for the applicable analysis prior to performing unsupervised analysis on

laboratory samples. Courses on sample preparation and instrument analysis may be taken separately or combined. The criteria and training requirements for laboratory personnel shall be clearly defined, documented and maintained on file. The laboratory must maintain a description of the training program content, duration of the training, qualifications of the trainer, and objective evidence that the analyst/technician has successfully analyzed unknown reference samples of the matrices/analytes of concern within specified acceptance criteria. The dates of authorization to perform specific tasks shall be recorded.

- All analysts and technicians shall have demonstrated ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples, or in-house quality control samples. This demonstration shall be done at a minimum of every six (6) months and documented.

- All analysts and technicians shall have a minimum of twenty (20) business days of hands-on experience conducting analyses in an industrial hygiene laboratory before initiation of independent work on customer samples.

7.0 ANALYTICAL METHODS

See SOPs

8.0 ASBESTOS TESTING

For asbestos testing, the lab shall adhere to the management system requirements as defined in Module 2A and this program specific module, Sections 2B.1 through 2B.4 (as applicable), in addition to the following management system requirements:

Phase Contrast Microscopy (PCM) Analysis

- The lab will comply with the quality assurance requirements of the Asbestos Standard Appendix A, CFR 1910.1001 and the most current revision of the NIOSH 7400 analytical method.

- The fiber counting microscopist will complete a NIOSH 582 course or an equivalent course. A description of the course as evidence of equivalent training will be submitted to AIHA. The description should include dates of training, course outline, contact hours, and record of examination.

APPENDIX XIV
PCM Asbestos QA Manual

Analytical Environmental Services, Inc.

QUALITY CONTROL/QUALITY ASSURANCE PROGRAM

FOR

AIR MONITORING

AND

PHASE CONTRAST MICROSCOPY

MEHMET YILDIRIM
LABORATORY MANAGER

1.0 INTRODUCTION

Analytical Environmental Services, Inc. is dedicated to providing quality analytical services throughout the U.S.

Analytical Environmental Services, Inc. (AES) specializes in the analytical aspects of asbestos testing. AES has implemented a quality control and quality assurance (QA/QC) program for air monitoring and phase contrast microscopy. This program was developed to establish quality control standards necessary for governmental compliance. In order to consistently maintain high standards of precision and accuracy in analytical testing, AES participates in the American Industrial Hygiene Association (AIHA) Proficiency Analytical Testing (PAT) program.

This quality assurance plan will establish the procedures that will be followed to ensure accuracy, precision, completeness and representation of data obtained from analysis of asbestos fibers. Laboratory and field operations will be conducted to comply with the methods referenced in this manual.

AES requires its technical staff members earn no less than a Bachelor of Science degree in a science related field. In addition, prior to formal task assignment, the employee is required to undergo extensive in-house training beyond the training and certification required by EPA Government standards.

2.0 PURPOSE

AES has implemented a Quality Control, Quality Assurance program for the purpose of providing a baseline of standards which will enable a continuous surveillance of Quality performance for the benefit of Government compliance, client satisfaction and minimization of liability.

3.0 SCOPE

This QA/QC program provides the necessary guidelines to secure and maintain:

- High level of quality work.
- Comprehensive accountability of all activities relevant to field and laboratory services.
- Continuous compliance with Government regulations.

A high level of employee and laboratory work performance is ensured by means of implementing an orderly system of record keeping which is able to track the sample from the moment that the sampling process started to the final reporting of the results.

Government Regulations mandate compliance and require:

- Accountability of work performed.
- Application of analytical methods.
- Observance of Health and Safety Regulations.

- Accreditation and certifications.
- Pre-requisite training to do work.

T his QA/QC document contains the following information:

- Comprehensive system of daily, weekly, and monthly record keeping.
- Definition of air monitoring activities.
- Air sampling techniques for PCM and TEM methods of analysis.
- Sampling equipment.
- Calibration of sampling equipment.
- Analysis of air samples by PCM.
- Calibration of analytical equipment.
- Procedures for measurement of fibers.
- QA/QC activities for fiber counts.

4.0 PERSONNEL RESPONSIBILITIES

4.1 Chain of Custody Officer

A. Requirements

Only an employee with training in NIOSH 582 will be allowed to perform as chain of custody officer.

B. Job Description

1. Preparation of chain of custody form for each project.

2. Checking the condition of the arriving packages.

3. Acceptance or rejection of each sample based on the criteria mentioned in the Quality Control/Quality Assurance Procedure Manual.

4. Sample preparation, including:

a. Checking the condition of each sample.

b. Verification of clients sample I.D. number with transmittal form.

c. Assign AES's I.D. number for each sample.

d. Preparation of Analytical Data Sheet.

5. Checking the typed reports and making the corrections.

4.2 Microscopist

A. Requirements

1. A degree in Natural Sciences.

2. Demonstration of proficiency analysis for ten (10) test samples submitted by the end of the training program.

3. Successful completion of NIOSH 582 course (above 70%).

B. Job Description

1. Checking the instrument performance.

2. Sample analysis (all steps must be conducted in accordance with published methodologies).

a. Transferring the samples to analytical dish under HEPA vacuum.
b. Visual fiber counting according to Method 7400.
c. Recording of analytical results.

3. Safety consideration.

a. Checking on HEPA vacuum system.
b. Checking on hood system.
c. Careful handling of the asbestos samples.
d. Using all facilities in a safe manner.

4. Contamination Consideration

a. Daily cleaning of analytical station.
b. Cleaning of glassware and sampling instruments.
c. Checking contamination of glass slides, coverslips and sampling instruments by running blank samples.

5. To determine the precision and accuracy of each microanalyst, he or she should analyze the internal standard samples as well as PAT samples.

6. Each microanalyst should reanalyze 10% blind samples of the total samples analyzed by him or her for that month.

4.3 Quality Control Officer

A. Requirements

1. At least six months of experience in analysis of air samples.

2. At least 1,000 samples analyzed by PCM technique.

B. Job Description

1. To develop a Quality Control/Quality Assurance program insuring satisfactory performance to governmental agencies and our clients.

2. Checking the performance of all instruments.

3. Quality control on the chain of custody form and the quality of the work that has been done by the chain of custody officer.

4. Instructing the chain of custody officer to make deficiency corrections.

5. Quality control on all samples analyzed by microanalyst and instruction of quality and quantity corrections of their analysis.

6. To instruct microanalyst about contamination and safety considerations. Also, to determine the precision and accuracy of the microanalyst.

7. Periodically checking the calibration of the analytical instruments.

8. Checking the laboratory report system.

5.0 RECORD KEEPING

Analytical Environmental Services has developed a state-of-the-art quality control program performed by its staff of Industrial Hygienists. For field tasks, our analysts are trained to perform at the highest quality as well as maintain a records system, available for internal auditing. Our field personnel maintain a variety of color designated working registers and journals, each dedicated to a specific area of activity:

I. QA/QC Register (red)

a. QA/QC manual for air monitoring and PCM analyses.
b. Log book for Quality Control of new filters (Laboratory Blanks).
c. Graphic calibration of rotameters for low and high flow rates.
d. Graph of the total coefficient of variation (CV).

II. Daily Activities Register (light brown)

a. Chain of Custody record.
b. Transmittal sheet.
c. Counting PCM form.
d. Daily form for PCM results.

III. Samples Log Book (gray)

a. Log book for PCM samples.

IV. Daily Journal Project Activities (black)

a. Log book for activities taking place during the working day.

V. Certificates/Accreditation (black/yellow)

a. Personal certificates (NIOSH 582, Supervision, Medical Test results, etc.).
b. Results for PAT intralaboratory analyses.
c. Results for PAT interlaboratory analyses.
d. Accreditation of the laboratories (NVLAP).

VI. Log Book of Blank Forms (green)

a. Log book for blank forms to be used for daily work.

VII. Proficiency Results

Every new job shall require a unique set of records. By the end of the job, the records are brought to the main laboratory and the analytical data is entered in the computer. The registers and journals are stored in our archives.

Analytical Environmental Services, Inc. maintains all the original records in its archives. Records are saved as hardcopies as well as backup files on tapes.

The record keeping system was developed by AES considering the following:

1. Records have to be continuously maintained during the data entry process.

2. Records must be kept in a database capable of providing QA/QC elements. Records shall be backed up once a week.

3. Final reports shall be reviewed by three (3) staff members. The last to review the final report shall be the laboratory director.

A flow chart for data entry and verification process is shown in the following Diagram 1.

Upon completion of the process, the original reports shall be kept in archives.

6.0 AIR MONITORING

6.1 General Activities

Air monitoring field activities include:

a. Site inspection to determine if preparation of work area is conducted in a safe manner.

b. Daily air monitoring during the removal of asbestos and subsequent cleaning operations.

c. Determination of worker exposure levels to airborne asbestos fibers.

d. Monitoring work activities to ensure that proper removal techniques are used.

e. Checking presence of asbestos material container to insure cleanliness and prevent outside exposure.

f. Ensuring enclosure system is well maintained.

g. Monitoring outside working areas for hazardous fibers.

h. Inspection of final cleaning operation to ensure compliance of job specifications.

i. Ensuring existing prevalent fiber level is at the specified limit before enclosure systems are removed.

j. Informing the owner's representative about air monitoring results and quality of work.

k. Reporting of final results, including work practice observation, daily air monitoring, & statistical interpretation of the results.

6.2 Sampling Equipment

The following items are necessary to perform sampling activities:

1. Personal Sampler Pumps

a. Calibrated pump whose flow rate can be determined to an accuracy of 5%. T he pump must be calibrated with a representative filter and holder in line.

b. The battery should be charged regularly. To maximize the life of the battery, periodically drain the battery's power and allow for a complete recharge. Failure to do so will result in a reduced capacity, which is a "battery memory", which is an irreversible process.

c. Check to make sure that the pump operates at a flow range between 1.5 liters/minute and 5.0 liters/minute. It has been found that a pump calibrated to a flow rate outside this range may not operate reliably.

d. Do not expose the pump to extreme heat. If pump is placed next to or on a hot piece of equipment, the (i.e., aluminum foil or a board which the pump can beset on.)

e. Do not expose the pump to excessive amounts of water. If the pump is placed in a wet environment, cover the pump with a piece of plastic. Be sure to allow some "breathing openings" when wrapping a pump with a piece of plastic.

f. Be sure to set pump on a stable surface when sampling. If necessary, secure the pump to a stationary object so that it does not tip over. Equipment vibration will produce enough motion to cause an unsecured pump to fall and break.

g. Clean the pumps before taking them out of a contaminated area after sampling. This will prevent you from being exposed to asbestos and help to maintain the equipment.

2. High Volume Pumps

a. Be sure that the pump operates correctly when it is turned on. If there is a grinding noise in the pump, do not use it.
b. Check the regulator to make sure the flow rate can be adjusted between 5.0 and 10.0 l/m.
c. Follow instruction (d) through (e) above in Low Volume pumps for handling the pump.
d. Ground the pump during operation to prevent electric shock.

3. Rotameter

High and low flow rate rotameter shall be used for calibration of area and personal pumps, respectively. The rotameter shall be calibrated against a primary standard.

a. Check rotameter to make sure that the center ball moves freely.

b. Check the fitting on the rotameter to make sure they are secure.

4. Cassettes

A cassette is composed of filter holder and filters.

a. Filter Holder contains the following:

· 3 section styrene plastic case for aerosol monitoring.
· Support Pad.
· Two (2) plastic sealing caps.

b. Filter

· 37mm or 25mm diameter plain white cellulose ester membrane filter (0.8 micron pore size).

Two types of sampling filters/cassettes are available:

a. For Phase Contrast Microscopy (PCM) testing, use 0.8 microns Mixed Cellulose Ester (MCE) filters loaded in 25-mm conductive cowl cassettes.

b. For Transmission Electric Microscopy (TEM) testing, use 0.45 Microns Mixed Cellulose Ester (MCE) filters loaded in 25-mm conductive cowl cassettes. Check for integrity of each cassette before setting up the sampling set to filter. Discard any damaged cassette.

Faulty or broken equipment should be taken out of service and given to the project supervisor(s) so it can be sent for repair.

5. Tubing

a. Check integrity of tubing; if it is cracked or if any piece is less than 18" long, discard it.

6.3 Calibration of Sampling Equipment

Calibrating equipment is a required function of the air monitoring process which needs to be documented in the equipment log as well as on the daily sample collection forms.

AES uses the rotameter as the calibrator for air sampling pumps. Since the rotameter is classified as a tertiary standard, it must be calibrated against a bubble buret, the primary standard.

1. A bubble buret system shall consist of:

a) buret stand with clamp
b) 1000 ml buret
c) plastic tubing
d) cassette with MCE 0.8 micron filter
e) rotameter
f) air sampler
g) dish with soapy water
h) stop watch

2. Calibration of rotometers:

Calibration shall be done as follows.

a. Use the Rotameter Calibration Chart (See form in Appendix I) to document all calibrating data. Record the rotameter I.D.# and estimate the temperature and pressure at time of calibrating.

b. Turn on air sampler and adjust flow rate to a desired level to be tested. Low flow rotameter should be calibrated at 1.0 l/m, 1.5 l/m, 2.0 l/m, 2.5 l/m, and 3.0 l/m. High flow rotameter should be calibrated at 6.0 l/m, 7.0 l/m, 8.0 l/m, 9.0 l/m, and 10.0 l/m.

c. Conduct three trials at each of the flow rate setting listed in "b". This is done by adjusting the pumping power so that the rotameter is read at the desired level and then recording the time it takes for the soap bubble to travel 1000 ml. Record the time of each trial. If a trial renders a traveling time significantly deviated from the others, discard the data and conduct the calibration again.

d. Calculate the average time of the three trials.

e. Calculate the calibrated flow rate determined from average time using the equation:

$$\text{Calibrated Flow Rate} = \frac{\text{(Volume bubble traveled (ml) / 1,000)}}{\text{(time (second) / 60)}}$$

f. Repeat steps c through e to generate calibrated flow rate for at least four flow rate levels.

g. Plot average point in graph of "Rotameter Reading" vs. the "Calibrated Flow Rate" and circle it. (See Appendix 1).

h. After points are plotted, draw a best-fitted line through the points. The best fit line can be drawn from visual estimation. The dashed line shall be drawn for extrapolation outside the measured range.

3. Frequency of Calibration

Rotameter must be calibrated prior to being placed into service and once per week in the beginning stage. When the flow rates have demonstrated a consistent pattern, the calibration can be performed once per month. Records from each calibration should be kept in the rotameter log book.

4. Air Samplers

a. Calibrate the sampling pumps using a calibrated rotameter before and after each sampling.

b. The rotameter should be placed in line between the cassettes and the pump.

c. With a rotameter in line adjust the pump to a desired flow rate according to the rotameter reading. Before recording the rotameter reading allow the pump to operate a few minutes in order to stabilize the air flow. Then use the rotameter's calibration curve to find the calibrated flow rate.

d. Correction to the flow rate for pumps with rotameter may be necessary if the pressure (elevation) and/or temperature where the samples are collected (actual flow rate) differs significantly from where the calibration was performed (calibrated flow rate). Actual flow rate at time of sampling may be calculated for a linear scale rotameter by using the following correction formula:

Actual Flow Rate =
Calibrated Flow Rate X ((Pcal/Pact)X(Tact/Tcal))1/2

Where both pressure (P) and temperature (T) are in absolute units such as:

Pressure in psi
Temperature in degree Rankin = degree Fahrenheit + 460

e. Use the actual initial and final flow rates to determine the average sampling flow rate, which will be used in calculating the sample fiber concentration.

1. Air sampling pumps must be calibrated before and after each sampling event.

2. Recalibrate a sampling pump if its flow rate is found drifting from the original setting significantly.

6.4 Techniques and Methods for Air Sampling

Two types of samples shall be collected and submitted for analysis:

1. BLANKS

Testing blanks provide an important quality control measure by detecting contamination during sampling and analysis in order to ensure the accuracy of testing results. Two types of blanks are used for air sampling:

a. Field Blanks: Submit at least two field blanks (or 10%, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.

b. Laboratory blanks: Laboratory blanks are used to detect if any contamination occurs during analytical process on each set of samples. They are also called sealed blanks. Laboratory blanks should be handled as a part of the regular samples throughout the entire analytical procedures.

2. AIR SAMPLES

There are two categories of air sampling techniques for asbestos testing:

a. Ambient Area Sampling: Air samples collected to determine average airborne fiber concentration levels of the tested area. Four types of purpose-oriented sampling use ambient area samples:

1) Exposure assessment
2) Inside abatement area test
3) Outside abatement area test
4) Final clearance

b. Personal Sampling: Air samples collected by attaching samplers to an individual in order to determine the personal exposure levels to airborne fibers.

Sampling purposes and strategy, applicable regulations or

recommendations, sampling period, flow rate, volume of air, and placement of pumps of each type of sample are discussed as follows:

1. Exposure Assessment

Sampling Purposes and Strategy: Exposure assessment sampling is usually used in the survey stage or research purposes.

No standard or reference level is used in order to reach an answer of passing or failure. The intention is to find the exact airborne fiber concentration level, no matter how low it is. Therefore contamination of samples, either from new filters, sampling techniques, or analytical procedures becomes critical to the accuracy of test, since most of exposure assessment tests are conducted in low concentration environments. To reveal the real situations, exposure assessment should be conducted under normal occupancy conditions, i.e. normal work hours, normal traffic, and normal ventilation. Aggressive sampling techniques are not recommended unless it is intended to discover the worst scenario of exposure.

Applicable Recommendations: EPA "purple book" : Guidance for Controlling Asbestos-Containing Materials in Buildings, EPA 560/5-85-024, June 1985; EPA "silver book": Measuring Airborne Asbestos Following an Abatement Action, 1985.

Sampling Period: 6.5 hours recommended. Shorter sampling duration may be necessary because of time and/or work conditions. Sampling periods may be represented by one (1) or more samples collected as time weighted average (TWA).

Flow Rate:

a. High flow: 6.0 1/m to 10.0 1/m recommended
b. Low flow: 2.0 1/m to 2.5 1/m recommended

Flow rates will vary depending on the volume of air which is needed to be collected.

Volume of Air:

a. TEM testing: minimum 1,500 liters, recommended 3,000 liters.
b. PCM testing: minimum 1,000 liters, recommended 3,000 liters.

Placement of Pumps: Exposure assessment samples should be placed at levels which are representative of a worker's breathing zone (approximately 4 feet to 6 feet above the floor) and at locations which have representative air quality (i.e. avoid dead corners or dirty air exhaust).

2. Inside Abatement Area Samples

Sampling Purposes and Strategy: These samples are collected during abatement activities inside a controlled area to determine the worker's average exposure levels. They also serve as indicators to the quality of abatement work performance. High fiber concentration results, from the inside abatement area samples indicate inadequate engineering controls of fiber release from poor removal techniques. This increases the risks of worker's over exposure. Some project specifications require the maintenance of airborne fiber concentration, inside the controlled area, to be lower than a certain level, for example, 0.1 f/cc by PCM. The inside abatement area sample results can also be used as the reference for proper respirator selection.

Applicable Recommendations: EPA "purple book": Guidance for Controlling Asbestos-Containing Materials in Buildings, EPA 560/5-85-024, June 1985; EPA "silver book": Measuring Airborne Asbestos Following an Abatement Action, 1985.

Sampling Period: Minimum 80% of working time recommended. Shorter sampling duration may be necessary because of time and/or work conditions. Sampling periods may be represented by one (1) or more samples collected as time weighted average (TWA).

Flow Rate: 2.0 1/m to 2.5 1/m recommended.

Volume of Air: Determined by sampling duration and flow rate.

Placement of Pumps: Inside abatement area samples should be placed at levels which are a representative of a worker's breathing zone (approximately 4 feet to 6 feet above the floor) and at locations which have representative air quality (i.e. avoid dead corners or dirty air exhaust).

3. Outside Abatement Area Samples

Sampling Purposes and Strategy: These samples are collected during the abatement activities outside the controlled area in order to detect if there is any leaking from the containment. Outside abatement area samples are very important when an abatement is conducted in an occupied building. Elevated airborne fiber concentrations indicate a possible leakage from the controlled area, which require an immediate response to solve the problem. These types of samples can be considered as the exposure assessment in the outside abatement area.

Applicable Recommendations: EPA "purple book": Guidance for Controlling Asbestos-Containing Materials in Buildings, EPA 560/5-85-024, June 1985; EPA "silver book": Measuring Airborne Asbestos Following an Abatement Action, 1985.

Sampling Period: Minimum 80% of working time recommended. Shorter sampling duration may be necessary because of time and/or work conditions. Sampling periods may be represented by one (1) or more samples collected as time weighted average (TWA).

Flow Rate:

a. High flow: 6.0 1/m to 10.0 1/m recommended
b. Low flow: 2.0 1/m to 2.5 1/m recommended

Flow rates will vary depending on the volume of air which is needed to be collected.

Volume of Air:

a. TEM testing: minimum 1,500 liters, recommended 3,000 liters.

b. PCM testing: minimum 1,000 liters, recommended 3,000 liters.

Placement of Pumps:

Outside abatement area samples should be at levels which are representative of a worker's breathing zone (approximately 4 feet to 6 feet above the floor). In order to

detect any leakage from the containment area, samples should be collected from the negative pressure machine exhausts, decontamination unit entrance, and leeward side of containment (at critical barriers).

4. Final Clearance Test

Sampling Purposes and Strategy: Final clearance testing is conducted following the abatement activities to determine if air quality is maintained below a predetermined level. Aggressive sampling techniques are required in order to disclose the worst scenario of airborne fiber levels. Two protocols are available for final testing:

a. EPA Level II TEM testing: Samples are collected inside the controlled area to determine if airborne asbestos fiber concentration is below the clearance level. The most common standard used in this industry is 0.01 structures per cubic centimeter and/or 10.0 nanogram per cubic meters.

b. EPA PCM testing: Samples are collected inside the controlled area to determine if airborne fiber concentration is below the clearance level of 0.01 fibers per cubic centimeter.

c. AHERA Protocol TEM testing: Five samples inside the controlled area and five samples outside the controlled area are collected, attached with one inside field blank, one outside field blank, and one sealed blank. See AHERA regulations for specific procedures for clearance testing.

d. AHERA Protocol TEM testing: Although TEM method is chosen to be used for final testing, AHERA regulations do allow an interim PCM clearance procedure that from October 8, 1989 to October 7, 1990 PCM testing can be used for final clearance testing if the abatement is conducted in a containment less or equal to 1,500 square feet or 500 linear feet. Five samples inside controlled area are collected. The area is cleared if each of these five samples is below 0.01 fibers per cubic centimeter.

Applicable Regulations: EPA 40 CFR Part 763 Asbestos-Containing Materials in Schools; Final Rule and Notice (AHERA); EPA "silver book":

Measuring Airborne Asbestos Following an Abatement Action, 1985.

Sampling Period: Recommended. Shorter sampling duration may be necessary because of time and/or work conditions. Sampling periods may be represented by one (1) or more samples collected as time weighted average (TWA).

Flow Rate:

1. EPA Level II TEM testing: 2.0 to 12.0 liters per minute
2. EPA PCM testing: 2.0 to 12.0 liters per minute
3. AHERA TEM testing: 1.0 to 10.0 liters per minute
4. AHERA PCM testing: 0.5 to 16.0 liters per minute

Flow rates will vary depending on the volume of air which is needed to be collected.

Volume of Air:

1. EPA Level II TEM testing: minimum 1,500 liters, recommended 3,000 liters
2. EPA PCM testing: minimum 1,000 liters, recommended liters
3. AHERA TEM testing: minimum 1,200 liters, recommended 1,500 liters
4. AHERA PCM testing: minimum 3,000 liters

Placement of Pumps: Inside abatement area samples should be placed at levels which are representative of a worker's breathing zone (approximately 4 feet to 6 feet above the floor) and at locations which have representative air quality. For AHERA TEM testing, outside abatement area samples should be placed at locations representative of air entering the abatement site.

5. Personal Samples:

Sampling Purposes and Strategy: Personal sampling is the direct measurement of the exposure level of the sampled individual. OSHA regulations require that employers should determine employee's asbestos exposure level under construction using personal sampling techniques. Therefore,

in an abatement project, the removal contractor (the employer) is required to collect personal samples from representatives of the workers (the employees). OSHA regulations also require that personal sampling be conducted in an occupational environment if asbestos exposure is suspected. It is recommended to collect personal samples from at least 20% of the total staff.

Applicable Regulations: OSHA 29 CFR Part 1910 and 1926, June 20, 1986.

Period of Sampling: Sample no less than 85% of time workers are in work area. If required, collect 8 hour time weighted average samples during first exposure to asbestos inside the work area. Multiple samples may be needed to represent an 8 hour TWA for personal exposure.

Flow Rates: Low flow: 2.0 1/m to 2.5 1/m Volume of Air: Determined by total time the samples are collected.

Placement of Pumps:

a. Personal sampling pumps should be placed on workers by using a belt or a duct tape. Cassettes should be located in a worker's breathing zone.

b. Precaution should be taken to prevent pump damage, e.g. covering the pump with plastics to prevent water damage.

6. Excursion Limit

Excursion Limit is defined by OSHA as 1.0 f/cc of air averaged over a 30 minute sampling period. The Excursion Limit is intended to provide for a reduction in health risk when used in conjunction with the 0.2 f/cc Permissible Exposure Limit (PEL).

The test for Excursion Limit is performed under the following circumstances:

a. When asbestos concentration will not be uniform throughout the shift.

b. When asbestos concentration may reasonably be expected to exceed PEL.

c. For each shift, each job classification, each work area in which operations are most likely to produce exposure above the PEL.

Testing for Excursion Limit:

The procedure for Excursion Limit testing is similar to the procedures of personal sampling. Sampling is performed for 30 minutes.

7. Time-Weighted Average (TWA) Exposure Monitoring

6.5 PURPOSES OF TWA SAMPLING

OSHA asbestos standard for construction industry 29 CFR Part 1926.58 entitled Asbestos, Tremolite, Anthophyllite, and Actinolite of June 20, 1986 specifies in section (f)(1)(ii), page 22757, that "Determinations of employee exposure shall be made from breathing zone air samples that are representative of the 8-hour TWA of each employee." OSHA delineates further in section (f)(1)(iii) that "Representative 8-hour TWA employee exposure shall be determined on the basis of one or more samples representing full-shift exposure for employees in each work area."

6.5.1 TWA SAMPLING

Personal samples shall be collected for the determination of OSHA 8-hour TWA employee exposure following the protocols below:

* An air flow rate between 0.5 liter/min. and 2.5 liter/min. shall be selected for 25-mm cassettes.

* Samples shall be taken in the "breathing zone" of the employee (i.e. attached to or near the collar or lapel near the worker's face).

* For each personal sample collected, complete and accurate information of the worker's name, social security number, work area, and tasks performed shall be included in a sampling data sheet.

6.5.2 TWA SAMPLING STRATEGY

* One or a few representative workers, depending on the size of the working crew, shall be selected for personal samples collection. Samples shall be taken from the same person continuously, if possible, throughout the entire shift in order to determine this individual's TWA exposure.

* Samples shall be collected to cover at least 80% period of the time of a work shift so that the results are representative.

* TWA results shall be determined for at least 25% of the staff of a shift to represent the entire staff's exposure of the shift. For example, for a crew of 8 workers working 8 hours, personal samples shall be taken from at least two representative workers for at least six and a half hours in order to obtain TWA results for each of these two individuals.

* The ideal situations for TWA monitoring are: one individual working for 8 hours in one work area performing several tasks. However:

  - If the work/shift lasted less than 8 hours, samples shall be taken to cover as much working time as possible. In such a case, TWA results will be based on the actual work hours, not 8 hours.

  - If samples were collected from Worker X working area A,B, and C of a working shift, the TWA results from these samples will represent the overall, not specific to any work area, exposure of Worker X of that shift.

  - If samples were collected from Workers X, Y, and Z in area A of a shift, the TWA results from these samples represent the overall, not specific to any worker, exposure of workers of work area A.

CALCULATION OF TWA

$$\text{TWA (f/cc)} = \frac{C1xT1 + C2xT2 + ... + CnxTn}{T1 + T2 + ... + Tn}$$

where Cn = Fiber concentration of each sample in f/cc.
Use reported fiber concentration (RFC) value. If RFC is of the format as "<x.xxx" (less than...), use the value without the less than sign.

Tn = Sampling duration of each sample in minute.

For example, four personal samples were taken from one individual in one work area:

Sample ID	Sampling duration (min.)	Concentration (f/cc)
#1	125	0.021
#2	110	0.050
#3	130	0.022
#4	100	<0.009

Then TWA of this individual should be:

$$TWA = \frac{0.021x125 + 0.050x110 + 0.022x130 + 0.009x100}{125 + 110 + 130 + 100} = 0.026 \text{ f/cc}$$

6.5.3 REPORTING OF TWA RESULTS

TWA results shall be prepared using the Time-Weighted Average Exposure Monitoring Report form and submitted with the Daily Air Monitoring PCM Analysis Reports.

To prepare the TWA report using the Time-Weighted Average Exposure Monitoring Report form, adhere to the following steps:

* Fill out the sampling date, project name, and AES Job Number.

* For each sample to be used in calculating the TWA result, write the sample ID number, sampling duration, reported concentration, and worker's name and social security number.

* Describe the work area, tasks performed, and the total work hours by the worker from whom personal samples were taken.

* Calculate the total duration of all samples. The total sampling duration should cover at least 80% of the total work hours specified in the previous column.

* Calculate the TWA and report it to the third decimal place.

* Extend the bottom line of the last sample entry row to across the last three columns for clarifications of a TWA set.

* Make necessary comments for other important information.

* Sign at the bottom right.

7.0 AIR SAMPLE ANALYSIS

AES analyzes air sample filters to determine concentrations of airborne fibers utilizing Nationals Institute of Occupational Safety and Health (NIOSH 7400 - A" rules) Analytical Method.

In order to consistently insure high standards of analysis, AES participates in the American Industrial Hygiene Association (AIHA) Proficiency Analytical Testing (PAT) Program (I.D. #9096-001). See Appendix B for our PAT results.

7.1 Chain of Custody

For each batch of samples received in the lab, a PCM Chain of Custody form is filled out (Exhibit #1). The client and project names are recorded and a job number is assigned. The date and the person receiving the samples are noted, as well as the condition of the package and the condition of the samples. The person preparing the samples for analysis verifies that the information on the transmittal sheet is consistent with the numbers on the samples and then assigns AES's own number to each of the samples and records the number on both the lid and container in permanent ink. A data analysis sheet (Exhibit #2), on which the AES number and the original identification number are recorded, is prepared for each sample. It is required to "log in" all of the received air samples into a central log book (Exhibit #3).

The PCM Chain of Custody form also records the date of analysis, the person who analyzed the samples, and the quality control officer. The report is entered into a data base, checked, signed; and mailed to the client. The original lab report, the original typed report and the Chain of Custody form are placed in our permanent files.

7.2 Criteria for Acceptance or Rejection of PCM Samples

Upon the receipt of the package in the laboratory, the package is examined to see if it has been damaged in such a way that any cassette of the air sample has been broken or deformed, the lid has been opened, or the flow rate time is missing. In any of the above cases, some of the samples may not be acceptable for testing. The client I.D. number on each sample container must be legible and not be ambiguous, and verified against the transmittal form. In the case of any discrepancy, the sample will be rejected for analysis.

7.3 PCM Analytical Procedures

All air samples are analyzed according to Method NIOSH 7400 for fiber content. This method requires the use of Phase Contrast Microscopy.

In order to perform the analysis, samples are mounted from their collecting devices to the glass slides. This transfer takes place under a negative air environment provided by a Nilfisk HEPA vacuum. This procedure protects our microscopist from being exposed to asbestos during the transfer. The inside of the cassette must be checked to insure that all parts of the sample are placed under the hood, also equipped with a HEPA negative air system. This negative air system pulls all fibers released during analysis away from the microscopist, trapping them in the HEPA filter. If, for any reason, the container is open or falls on the lab floor or table, and the sample spills in the laboratory during sample preparation or sample analysis, AES will have the contaminated area cleaned.

The microanalyst will record if the sample is wet, or damaged. The purpose of this action is to avoid erroneous results.

Once the sample is mounted and dissolved according to the following procedures, if necessary, an initial gross stereomicroscope examination will be performed. Sample analysis should proceed by using the 40x magnification.

7.4 Contamination Control

A cleaning procedure for all analytical stations is sustained. However, it remains the responsibility of the individual analyst to insure that the glassware and sampling instruments used in the performance of the analytical procedure are properly cleaned. To control any contamination of glass slides, cover slips and sampling instruments, blank samples are included frequently, and the results are recorded on a contamination control testing sheet (Exhibit #5) for further consideration.

When a particular sample is to be analyzed by an outside laboratory for quality assurance, the lab manager will insure that a representative sample is taken and mounted according to standard procedures. Contamination has to be checked daily prior to any analysis. If a contaminated blank is found, it is to be disposed of after thoroughly checking that the source of the contamination is not on the slides or cover slips.

7.5 Sample Preparation

Following is the standard procedure to remove a filter from a cassette and prepare it for analysis:

1. Clean working area.

2. Label a clean sample slide, include both AES lab # and Field #.

3. Open cassette, place filter surface up.

4. Collapse the filter and make it transparent by using acetone evaporator Model by Wonder Makers. This technique is a faster more permanent mounting technique than the Dimethyl Phthalate/Diethyl Oxalate method. Avoid making any "fiber immigration" or "fiber redistribution" on filter. This may be caused by an excess of acetone. Immediately place 3 to 3.5 ìl ultriacetin on wedge using a micropipet. Gently lower a clean cover slip onto wedge at a slight angle and cover filter. Finally, glue the edges of the cover slip by using nail polish. Prior to the analysis, the microscope must be set up as follows:

a. Microscope on, eyepieces in place, clean the Walton Beckett reticle. This

must be mounted on eye piece.

b. Focus on slide: place slide on the stage, right side up - use the 10 x objective to focus first if necessary. Be sure the 40x phase objective is in place and in focus. DO NOT CHANGE THE FOCUS AFTER THIS.

c. Adjust the field iris.

d. Align the phase rings.

7.6 Analytical Equipment

The following equipment must be available to perform analytical work:

1. PCM microscope with 10x & 40x objectives & Walton Beckett graticule.
2. Telescope-phase alignment.
3. Stage micrometer.
4. Slides.
5. HSE/NPL slide.
6. Cover slips.
7. Lens paper.
8. Markers and pens.
9. Acetone.
10. Triacetin.
11. Syringe.
12. Blades.
13. Tweezers.
14. Hot Block.

7.7 Calibration of Analytical Equipment

7.7.1 Microscope Calibration

Daily and monthly calibrations are performed for AES's Nikon microscopes. The daily calibration includes phase ring alignment and adjustment of the light source. The monthly calibration includes calibration of Walton-Beckett graticule and calibration of phase shift detection limit.

1. Calibration of phase rings:

   a. Adjust the light source for even illumination across the field of view at the condenser iris.
   b. Focus on the particle to be examined.
   c. Make sure field iris is in focus and centered.
   d. Use the telescope ruler supplied by Nikon to ensure that the phase rings are concentric.

e. Use front screws to adjust the rings until the best fit is achieved.

2. Calibration of Walton Beckett graticule:

a. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
b. Set the appropriate interpupillary distance, and if applicable, reset the binocular head adjustment so that the magnification remains constant.
c. Install the 40 to 45 X phase objective.
d. Place a stage micrometer on the microscope object stage and focus t the microscope on the graduate lines.
e. Measure the magnified grid length. L(mm) using the stage micrometer.
f. Remove the graticule from the microscope and measure its actual grid length L(mm). This can best be accomplished by using a stage fitted with verniers.
g. Calculate the circle diameter, d(mm) for the Walton Beckett graticule.

$$d = \frac{L_a * D}{L_o}$$

Example: If L_o = 108 um L_a = 2.93 mm and D = 100 um then D = 2.71 mm.

h. Check the field diameter.
D - (acceptable range 100 ìm ± 2) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (mm^2).

3. Calibration of Phase Shift Detection Limits

Check the phase-shift detection limit of the microscope periodically.

a. Remove the HSE/NPL phase-contrast test slide from its shipping container and center it under the phase objective.
b. Bring the blocks of grooved lines into focus.

NOTE: The slide consists of seven sets of grooves (ca. 20 grooves to each block) in descending order of visibility from sets 1 to 7. The requirements for counting are that the microscope optics must resolve the grooved lines in set 3 completely. Although they may appear somewhat faint, the grooved lines in sets 6 to 7 must be invisible. Sets 4 and 5 must be at least partially visible but may vary lightly in visibility between microscopes. A microscope which fails to meet these requirements has either too low or too high a resolution to be used for asbestos, tremolite, anthophyllite, and actinolite counting.

c. If the image quality deteriorates, clean the microscope optics and, if the problem persists consult the laboratory director.

7.8 Procedure for Measurement of Fibers.

A. Place the slide on the mechanical stage.

B. Focus the microscope.

C. Regularly check phase ring alignment.

D. Counting.

Select "A" rules of NIOSH 7400 and start counting. The "A: RULES ARE REQUIRED FOR MONITORING PURPOSES UNDER OSHA OR NIOSH STANDARDS. The "A" rules are as follows:

1. Count only fibers longer than 5 um. Measure the length of curved fibers along the curve.

2. Count only fibers with a length to width ratio equal or greater than 3:1.

3. For fibers which cross the boundary of the graticule field, do the following:

a. Count any fiber longer than 5 um which lies entirely within the graticule area.

b. Count as 1/2 fiber any fiber with only one end lying within the graticule area.

c. Do not count any fiber which crosses the graticule boundary more than once.

d. Reject and do not count all other fibers.

e. Count all bundles or fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.

8.0 QUALITY CONTROL/QUALITY ASSURANCE PROGRAM FOR FIBER COUNTS

8.1 INTRALABORATORY PROGRAM

The following procedures are implemented to ensure the accuracy and precision of fiber counts:

8.1.1 Field Blanks

Field blanks are submitted with every batch of samples; with each set of samples, 10% or at least 2 field blanks are submitted. The blank results are averaged and subtracted from the analytical results before reporting. Any sample represented by a blank, having a fiber count in excess of 7 fibers/100 fields is rejected, and possible contamination is reported.

8.1.2 Laboratory Blank

A laboratory blank is analyzed from every box of new cassettes.

8.1.3 Blind Recounts

Two types of blind recounts are performed by AES personnel.

1. Blind recounts on a set of reference slides including a range of fiber loadings. The QA/QC officer maintains a set of reference slides which are supplied to the microanalyst for periodic counting. From blind recounts on reference slides, the laboratory intracounter Sr is estimated for each microanalyst. (See Tab 9).

2. Blind recounts performed on 10% of the total samples counted.

    a. Blind recounts are performed by the same microanalysts on 10% of filters counted. (See Tab 5).

    b. The QA/QC officer verifies that identity of the samples is unknown to the counter.

    c. The sample is discarded if the difference between two counts exceeds

$$|\_AC_2 - \_AC_1| > 2.78 \times (\_AC_{AVG}) \times CV_{FB},$$

where:
AC1=lower estimated airborne fiber concentration
AC2=higher estimated airborne fiber concentration
ACavg=average of the two concentrations estimates
CVfb=CV for the average of the two concentration estimates..

d. A blind counting form for the same count is attached in Appendix 1.

An intralaboratory program is in effect to assess the accuracy of each analyst. The program is based on the samples submitted to PAT program. Each microanalyst reports the results for 4 samples submitted with each proficiency round. The results are statistically analyzed and plotted on a summary graph. If the results of a certain microanalyst fall outside the control limits, a training program for that particular load of fibers is initialized, and proficiency has to be proven. AES has chosen control limits to be two Standard Deviations from the mean, despite the fact that for proficiency in the PAT program, three Standard Deviations from the mean are acceptable. Attached are the results for a few rounds of PAT programs. (See Appendix 1).

8.2 INTERLABORATORY PROGRAM

AES participates in two inter-laboratory programs:

1. Proficiency Analytical program directed by NIOSH (PAT).
2. Round Robin program together with an additional laboratory.

The results for various rounds in these programs are attached in Appendix 1. AES has been rated as proficient with 100% of the results being within one standard deviation.

8.3 Quality Control/Quality Assurance program for PCM mobile laboratories.

Mobile laboratories are defined as those working stations outside the main laboratories where analytical work for fiber counting is performed. In order to assure the highest standard of performance AES has developed a program to conduct periodical QA/QC audits.

These audits are conducted monthly and contain the following areas.

I. Auditing of air sampling and analytical procedures.
II. Auditing of mobile laboratory condition and equipment.
III. Auditing of calibration procedures.
IV. Auditing of project record keeping.
V. Auditing of sample storage method.
VI. Auditing of reference materials present at the site.

The auditing shall be performed by regional QA/QC officers and the results shall be communicated to the operation managers. Corrective action shall be taken within 3 days of the auditing completion. If training is necessary, the microanalyst shall be put into a comprehensive and intense program of training.

9.0 PROCEDURE FOR CORRECTING MICROANALYST DEFICINCIES

The following procedures shall be followed for correction of microanalysts deficiencies:

1. Transferring Sample I.D. Numbers

   Errors made by the microanalyst in the process of transferring the Sample I.D. number designated by the client or, the lab number assigned by AES, from the sample container to the analytical sheet, will be corrected by the Chain of Custody Officer, with the assistance of the microanalyst.

2. Sample Results

   The Quality Control Officer analyzes the sample and checks the sample results to see if it has been correctly calculated, with special emphasis on Time Weighted Average (TWA), Measured Detection Limit (MDL), Measured Fiber Concentration (MFC) and Reported Fiber Concentration (RFC).

3. Blind Counting

   If during QC summaries blind counting test is failed, or proficiency in PAT sample analysis was not demonstrated, the analyst shall be put in a training program in order to correct deficiencies.

The Quality of Fiber Count Data

Martin T. Abell, Stanley A. Shulman, and Paul A. Baron
National Institute for Occupational Safety and Health, 4676 Columbia Parkway, Cincinnati, Ohio 45226

Optical fiber counts are used to determine asbestos exposure, so it is important to assess, control, and document the quality of those counts. These functions are the responsibility of the quality assurance (QA) coordinator in each laboratory. The QA coordinator must recognize that, compared to the analytical results for other substances, fiber count data are much more variable and have different statistical properties. These data, therefore, warrant special treatment. This article discusses the need to recount some samples, the procedures for determining bias and variability from these recount data, and the use of these statistics to test analytical results or assign confidence limits to them. Three kinds of bias and variability must be considered: intracounter, intralaboratory, and interlaboratory. As data pairs (count and recount) are obtained, the first consideration is whether bias is present. If bias is detected in a set of data, that data should not be used for any purpose until the source of bias is investigated. Bias can be difficult to detect, and when not detected, the differences in the data are assumed to be variability. The procedures recommended in this article for determining variability are based on the relatively simple calculations of NIOSH Method 7400, but alternative calculations are also discussed. Variability is expressed as s_r, which is an estimate of relative standard deviation.

An example calculation of intracounter s_r is given, along with an example of how to use this s_r to test the quality of a fiber count for an individual sample. This test, which does not have great power, is meant to detect differences between a counter's historically established s_r and the s_r for the test sample. Such a difference indicates a problem with the sample or the analytical procedure, and as a guideline, fiber counts that fail the test should not be used to evaluate exposure. In an extension of the test given in NIOSH Method 7400, a guideline is given for deciding whether an entire sample set should be rejected based on the number of individual fiber counts rejected. Intralaboratory s_r is an indicator of differences among counters within a laboratory, but these differences should be investigated for identifiable biases due to differences in training, visual acuity, or equipment. Interlaboratory s_r can be calculated if samples are exchanged between laboratories. Interlaboratory s_r is used to calculate a confidence interval about each analytical result, and that confidence interval should be reported as the analytical result. Analytical results reported for other substances do not include a confidence interval, but the analytical methods for other substances can be calibrated with readily available reference materials, thereby reducing the differences among laboratories. The nature of the fiber counting method and the lack of reference materials means that biases between laboratories are not easily corrected, or even identified. The confidence interval informs the person reading an analytical report about the differences among laboratories.

Abell, M. T.; Shulman, S. A.; Baron, P. A.: The Quality of Fiber Count Data. Appl. Ind. Hyg. 4:273–285; 1989.

Definitions

The following acronyms are defined here and are not defined again in the text.

AAR	Asbestos Analysts Registry program (a quality assurance program for individuals who count fibers)
ACGIH	American Conference of Governmental Industrial Hygienists
AIHA	American Industrial Hygiene Association
HSE/NPL	UK Health and Safety Executive/UK National Physical Laboratory
NIOSH	National Institute for Occupational Safety and Health
OSHA	Occupational Safety and Health Administration
PAT	Proficiency Analytical Testing program (a laboratory quality audit program)
PEL	Permissible Exposure Limit (issued by OSHA)
REL	Recommended Exposure Limit (NIOSH)
TLV	Threshold Limit Value (ACGIH)

The following words and symbols are defined as used in this article. Most are more thoroughly described in the text.

bias: systematic difference between two sets of measurements.

confidence interval: an interval which contains the parameter under study with high probability; a range of fibers/mm^2 values (or fibers/cc) that includes the true mean for the sample with high probability.

component of variance: one of several sources of total variability of a measurement.

count: (verb) to determine the number of fibers/mm^2 on a filter by phase contrast microscopy and by following the counting rules of a specific method such as NIOSH Method 7400.

count (fiber count): (noun) the datum produced as a result of counting the fibers on a single filter sample.

data pair: two numbers; the count and recount data for a sample.

fiber: for the purpose of this paper, any particle counted according to the rules of NIOSH Method 7400.[1]

interlaboratory RSD: the true RSD pertaining to measurements made by a randomly chosen laboratory.

interlaboratory s_r: statistic that describes the variability between/among laboratories (see s_r); an estimate of interlaboratory RSD.

intracounter RSD: the true RSD pertaining to counts by a given counter.

intracounter s_r: statistic that describes the variability of data pairs obtained by a single counter (see s_r); an estimate of intracounter RSD.

intralaboratory RSD: the true RSD pertaining to measurements made by a randomly chosen counter in a given laboratory.

intralaboratory s_r: statistic that describes the variability between/among counters in a laboratory (see s_r); an estimate of intralaboratory RSD.

lognormal: refers to data that, after transformation to the log scale, conforms to the normal distribution.

Poisson component: that part of the variability of count data due to the random distribution of fibers on a filter surface.

pooled: data that are combined into one set because they can be considered to measure the same thing.

power: the ability of a statistical test to detect differences between two groups of data; the probability of rejecting the null hypothesis of a statistical test, as a function of the value assumed by the parameter under study.

QA Coordinator: person in each laboratory responsible for assessing, controlling, and documenting the quality of fiber count data.

recount: (verb) to count the fibers on a sample filter for the second (or third, etc.) time.

recount: (noun) the datum produced as a result of performing a recount.

reference sample: samples that have already been prepared and counted more than once and are recounted for the purpose of calculating or testing a statistic.

reference value: statistic based on the best available data for reference samples.

RSD: the true value of the relative standard deviation, also known as coefficient of variation, or CV; the true standard deviation divided by the true mean.

s_r: estimate of relative standard deviation[2] (see RSD); a statistic that indicates the variability of a set of data.

$s_{r,s}$: subjective component of s_r, that is, s_r with the Poisson component of variability removed. Applied only to interlaboratory variability in this article.

sample categories: groups of samples judged by the counter to be similar for quality control purposes (i.e., the count data are expected to exhibit homogeneous variability). A sample, or the data pair for it, is usually assigned to a category on the basis of fiber loading (fibers/mm^2), but other characteristics should be considered.

sample set: incoming samples grouped together for quality control purposes. Samples are usually assigned to a set based on the fact that they came from a similar source and will be analyzed by one counter.

stopping rule: A rule for counting fibers, stated in NIOSH Method 7400 as follows: "Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count."

transform: a mathematical operation (e.g., taking a log or square root) performed on all the elements of a set of data.

variability: random differences among numbers in a set, where all the numbers in the set are presumed to measure the same quantity (such as the fibers/mm^2 on a filter.) It is measured by the variance.

Introduction

Many decisions regarding worker exposure to asbestos or other fibrous materials are made on the basis of fiber count data. Therefore, it is important to know the quality of these data. Each laboratory should have a Quality Assurance (QA) Coordinator who is responsible for assessing, controlling, and reporting the quality of the laboratory's fiber count data. This means that the variability and bias of count data should be determined and reduced when possible and that the data should be reported with some indication of its variability.

NIOSH Method 7400[1] outlines procedures for determining variability and reporting it. Methods for other analytes[3] do not discuss such procedures, and the procedures are not necessarily the same as would be used for those other methods. However, optical fiber counting is sufficiently different to warrant special treatment.[4] It is more subjective than other methods, involving complex decisions by the analyst, as well as depending on the visual acuity, training, motivation, and experience of the analyst.

The primary objective of this article is to explain in greater detail the quality control procedures and calculations outlined in NIOSH Method 7400. These recommended procedures are summarized in the "Conclusions and Recommendations." The authors believe that widespread use of the practices described will ultimately improve the quality of fiber count data being reported. However, the simple procedures of NIOSH Method 7400 will not be appropriate for all situations, and some alternative statistical procedures are discussed briefly in the appendix. The investigation of the procedures described in the appendix, and several others, failed to find any for general use that were clearly superior to those presented in the text and in NIOSH Method 7400. Although this article is meant as a practical guide, it concerns research questions still open for discussion.

Variability and Bias

The quality of fiber count data is measured by the variability and bias of that data. There are many elements in the procedure used for determining a fiber count that can contribute to variability and bias. For example, the samples brought into the laboratory for analysis may be of poor quality (poor filter quality, poor sampling conditions, etc.) without anyone being aware of it, causing the variability of the fiber counts to increase. This section discusses some of the sources of variability and bias, particularly those which may be quantified.

One source of variability is the random distribution of fibers over the filter surface, only part of which is analyzed. Random distribution affects even high quality samples. It means that if a filter surface is divided into small, equal areas, there is equal probability of a fiber depositing on any given area, but areas selected at random will not necessarily have the same number of fibers. This distribution of fibers on the filter surface can be described by Poisson statistics.[5] However, it has also been shown that the distribution can be more variable than Poisson, being better described by a negative binomial[6] or lognormal[7] distribution. In NIOSH Method 7400 and in this article, the variability

of a fiber count due to this random distribution is called the Poisson component of variance.

Another source of variability is dependent on the actions of the individual counter. As a counter gains experience, that person will usually adopt routines that decrease variability.[8] On the other hand, counting is still somewhat subjective, and each result is influenced by the "mind-set" of the counter at the time. A statistic that indicates a single counter's ability to reproduce previous results is the intracounter s_r, which is an estimate of intracounter RSD. This is obtained from repeat counts of samples by a counter; the procedure for calculating this statistic is given below.

Repeat counts produced by one counter usually match each other more closely than they match those produced by some other counter for the same sample. This additional source of variation, the difference among counters in a single laboratory, is included in the intralaboratory s_r, an estimate of intralaboratory RSD. Finally, laboratories differ from each other, a source of variation included in interlaboratory RSD. A laboratory is defined in this context as a group of counters who participate in a single QA program. Note that in this discussion, each kind of variability includes those previously mentioned. That is, the Poisson component of variance is included in the intracounter RSD, which is included in intralaboratory RSD, which is included in interlaboratory RSD.

Bias means systematic difference, but the word is used in different ways. Usually, bias is thought of as the difference between a measured result and some "true" value. However, there is no reference method for counting fibers that gives the true value, so this is not the meaning of bias that will be used here. Fiber counting can be "calibrated" only to the extent of using a test slide (HSE/NPL Phase Contrast Test Slide, PTR Optics, Waltham, Massachusetts) to check the microscope or counting reference samples, as discussed below, to compare results with those obtained in the past (usually by other laboratories). While these steps are highly recommended, they are not comparable to the calibration that can be achieved in methods for other analytes.

Bias is used in another way which deserves brief mention here and is discussed at length elsewhere.[9] This use of the word has to do with the linearity of the method over the range for which it is used. NIOSH Method 7400 recommends that loadings be in the range of 100 to 1300 fibers/mm^2 of filter surface area so that reported fiber counts will be proportional to loading. It has been found that loadings below this range often result in concentration estimates that are positively biased, while loadings above this range result in concentration estimates that are negatively biased.[10] It is recognized that measurements are often made at low concentrations in order to document compliance with established exposure limits (e.g., a PEL, REL or TLV). Even if the results of these measurements are positively biased, they are useful indicators of compliance if their upper confidence limits are below these exposure limits.

In the rest of this article, the word bias means consistent differences between the results obtained by a single counter or laboratory and the reference values for those samples. A reference value is usually based on the average result obtained by a group of competent counters. The use of reference samples and reference values for various purposes is discussed throughout remainder of this article.

In practice, bias may not be easy to distinguish from variability. If one counter always reports much higher counts than the other counters in a laboratory, there is clearly a bias and something should be done about it. But if a consistent difference is smaller, it may not be noticed so easily and becomes a component of the intralaboratory RSD. Each of the three components of variability mentioned above, along with a related kind of bias, is covered in a separate subsection of the "Calculations and Discussion" section below.

The different types of variability, or RSD, are listed in the center column of Figure 1. To the left, some possible sources of variability or bias that contribute to each type are shown. To the right, the uses of each type of RSD are listed. In the "Use" column, an "Indicator" is a statistic that can be included in an analytical report. A better use for these indicators is to identify problems to be corrected, linking the "Use" column back to the "Source" column.

Quality Assurance Program

Determining the different kinds of variability and bias defined above can help document the quality of fiber counts and is the first step to identifying the source of poor quality fiber counts. It is recommended that the QA Coordinator in each laboratory assume the responsibility of determining and tracking these measures of variability and bias. To accomplish this, the QA Coordinator supplies each counter with QA samples in addition to the routine workload. These additional samples are provided blind to the analysts to ensure that they have no idea of the expected answers for the samples. As illustrated in Figure 2, QA samples are of two types: recount samples and reference samples.

Recount samples are the most frequently counted QA samples introduced into the sample stream. These are samples from a set that are counted a second time and constitute at least 10 percent of each set. They provide a check on both sample quality and analyst performance. The count and recount data for the samples are called data pairs and are used to calculate various statistics. Further discussions of recounts are given under the "Intracounter Bias and s_r" and "Sample Quality Test" sections below.

Reference samples should constitute an additional 5 percent of the workload of each counter. These samples come from a previously prepared bank of samples for which the mean and variability have been historically established. These samples are taken from the normal workload and are selected to uniformly cover the range of concentrations normally encountered by the laboratory. Note that samples mounted by the recommended acetone–triacetin technique are not permanent and have been observed to degrade in time periods ranging from six months to several years.[11] Reference samples should therefore be checked for filter integrity and replaced at least every two years.

Reference sample data are used to determine several statistics. First, the data for reference samples already counted by the same counter can be used for determining intracounter bias. This is mentioned in the "Intracounter Bias and s_r" section, although that section is primarily concerned with recount samples and variability. Next, the data for reference samples counted by other counters in the same laboratory can be used to determine the statistics discussed in the "Intralaboratory Bias and s_r" section. Finally, samples that have been counted by analysts from other laboratories in an exchange program can be used to estimate biases between laboratories and interlaboratory s_r. If sufficient data are collected to provide a good estimate of interlaboratory s_r, this value can be used by the laboratories in the exchange group to calculate confidence limits on reported results. The calculation of these statistics is described under the "Interlaboratory Bias and s_r" section.

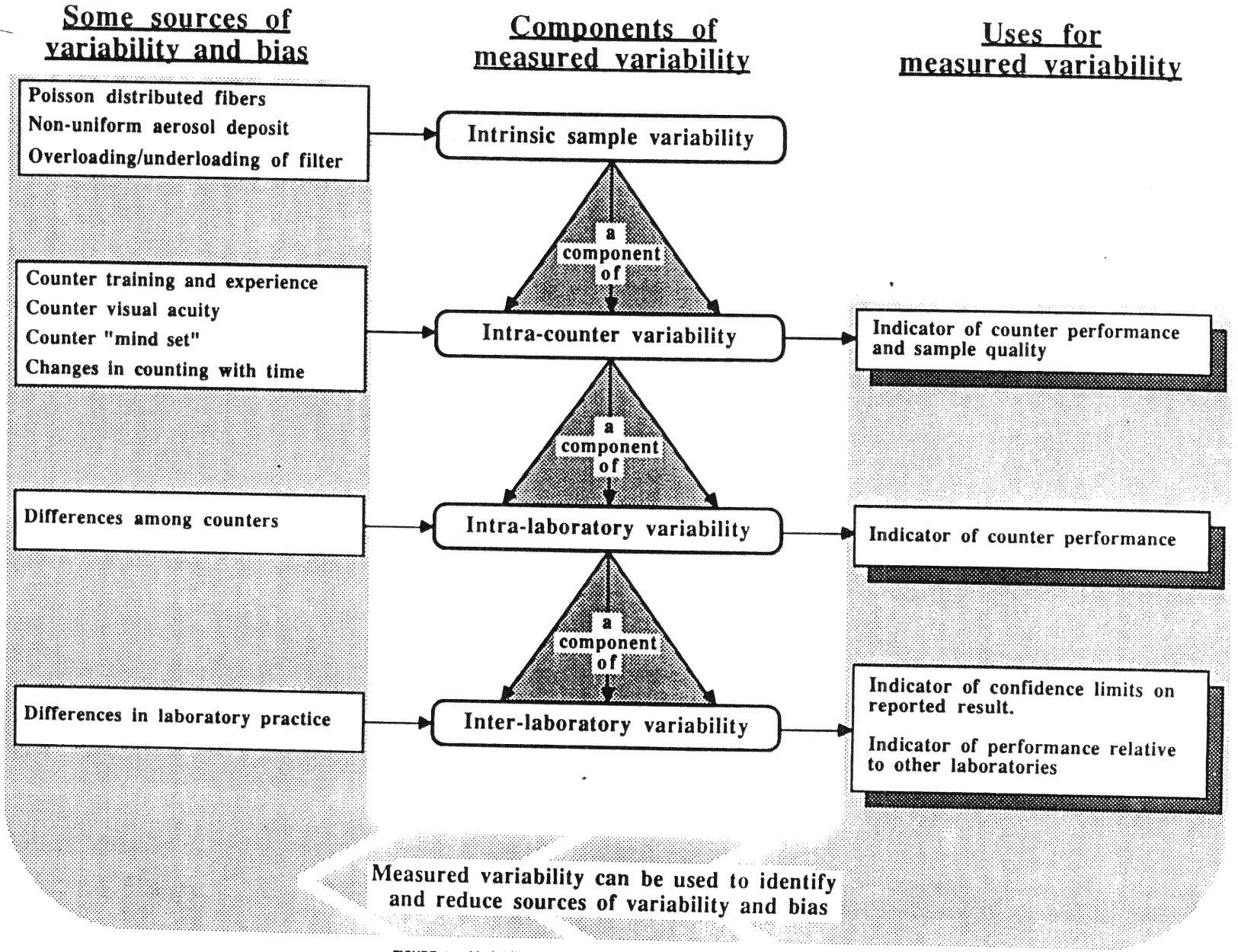


FIGURE 1. Variability components, their sources, and their uses.

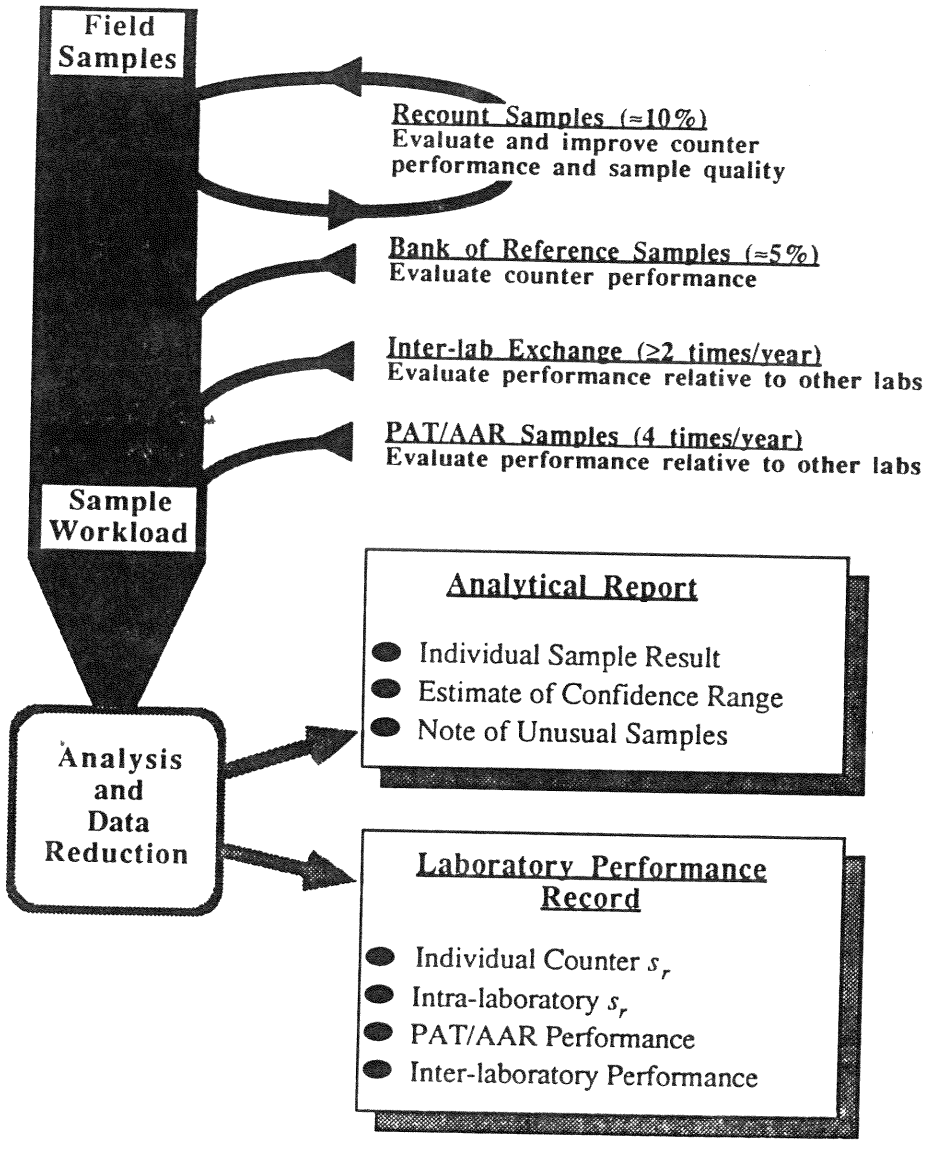


FIGURE 2. The total asbestos sample workload includes quality assurance samples so that data quality can be determined.

Calculations and Discussion

In this section, the terms "sample set" and "sample category" are used frequently. A sample set is any number of samples arriving in a laboratory which are grouped together for quality control purposes. The quality control elements of concern are the assignment of blank and recount samples and some of the calculations discussed in the rest of this article. Ordinarily, the assignment of samples to a set is based on the fact that they came from a similar source and will be analyzed by one counter. For example, samples collected in one building, where there is only one source of fibers, could constitute a set. However, if a laboratory must report results on a daily basis, the samples analyzed in a day may be taken as a set, even if they represent only a subset of the samples from a source.

A sample category refers to samples with similar characteristics as judged by the person counting them. The data pairs obtained from the recounted samples in each category are used to calculate an intracounter s_r for the category; a counter will achieve different s_r's for different categories of samples. The characteristics that can affect variability are primarily fiber loading, as well as fiber type and nonfibrous-particulate loading. The number of categories needed will depend on the variety of samples received by the laboratory, but a good starting point is to differentiate samples into three categories based on loading (total fiber count): 5 to 20 fibers, 20.5 to 50 fibers, and > 50.5 fibers. A laboratory may decide to split these three categories into six, based on whether the particulate loading is considered low or high, or to differentiate categories by fiber type, etc.

The above descriptions of set and category point up several differences. Samples are assigned to the same set only if they are judged to be similar based on sampling conditions; samples are assigned to a category based on what the analyst observes about them through a microscope. Samples are assigned to sets as they arrive, and the set is usually of little interest to the laboratory after the data from the set have been reported; sample data are assigned to categories and may be the basis of statistical calculations. Sample sets may be divided into smaller sets, usually to accommodate other than quality considerations; sample categories are subdivided primarily in the interest of refining quality control calculations.

Intracounter Bias and RSD

This section is primarily concerned with the variability in the

fiber count data of individual counters, as estimated by intracounter s_r. However, if fiber count data include some differences due to bias, those differences should not be incorporated into s_r. Bias for a single counter means bias over time. That is, intracounter bias is the difference in the true fiber counts produced by one counter when counting the same sample at two different times. Of course, bias cannot be distinguished from variability for a single sample, but if enough data are available, bias can be detected.

It is even possible to detect intracounter bias within a single sample set if enough filters are recounted and the bias is great enough. When these conditions apply, bias can be tested using a two-tailed t-test that compares an average difference to a true mean of zero. The procedure is described in the "Bias" section of the Appendix. If bias is detected, its cause should be determined and eliminated before the data are used in any other way.

Intracounter bias may also occur over a longer period of time, analogous to "drift" in an instrumental result. Such bias can be detected from recounts of past reference samples introduced into the sample stream by the QA Coordinator. It is possible to do a t-test, as before, or to simply plot the count differences for reference samples as a function of time (date). If the differences are calculated by subtracting the original count from the recount, positive differences indicate a drift toward higher counts and negative differences toward lower counts. If a plot indicates that drift is occurring, it may be more instructive to simply proceed with an investigation of the possible causes for such a drift instead of testing for significant bias. This is particularly true if the counter's results are diverging from reference values established externally, e.g., in the PAT program.

If bias is not detected in the fiber count data produced by an individual counter, the remaining differences in the data are considered to be variability, specifically, intracounter RSD, or its estimate, intracounter s_r. Theoretically, the minimum intracounter RSD for optimally loaded samples is approximately 0.1, based solely on the distribution of fibers on the filter.[6,12] In practice, other sources of variability increase this minimum. The intracounter s_r statistic should be calculated by the QA Coordinator for each counter in a laboratory so that the quality of sample data can be determined by the "Sample Quality Test" below.

The following is a suggested scheme for initially determining an analyst's intracounter s_r. If available, field samples should be used to determine intracounter s_r since they will match the samples analyzed in the future better than laboratory generated samples would. Otherwise, samples from another source, such as the PAT program, can be used. Based on the initial fiber count, randomly select 15 or more representative field samples in each of three ranges of total fiber count: 5 to 20 fibers, 20.5 to 50 fibers, and >50.5 fibers. (The procedures described here may not be applicable for counts less than 10, but they are recommended unless the applicability of an alternative can be established. The appendix describes some alternative procedures.) Recount each of these (already mounted) samples and record the result as fiber/mm² and total fibers. Use the average of the total fibers counted to make the final assignment to a category. Use the fibers/mm² value for the calculation of intracounter s_r and in the "Sample Quality Test" below. The calculation of intracounter s_r can only be done in terms of fibers counted if all the counts were done on the same number of fields, a condition that may not be met because of the stopping rule given under "Definitions" above. Calculate the average and standard deviation for each sample. In this case, where there are only two measurements:

$$\bar{x} = \frac{(x_1 + x_2)}{2} \quad \text{and} \quad s = 0.707 \cdot |x_1 - x_2| \tag{1}$$

where:

x_1, x_2 = independent counts by 1 counter (fiber/mm²)
$\bar{x}$ = average fiber count (fibers/mm²)
s = estimate of standard deviation (fibers/mm²)

Divide the estimate of the standard deviation, s, by the average, $\bar{x}$, to obtain s_r for each sample:

$$s_r = \frac{s}{\bar{x}} \tag{2}$$

Square the s_r values. The square root of the average of these squared s_r values is the pooled intracounter s_r:

$$s_r \text{ (pooled)} = \sqrt{\frac{s_r^2 + s_r^2 + \ldots}{n}} \tag{3}$$

where:

n = the number of s_r values pooled

Values are pooled within each category. The more values that are pooled, the better the pooled s_r estimates the true RSD value for that category. The final step is to convert the pooled s_r value to a pooled s_r value on the square root scale for use in the "Sample Quality Test" (Equation 5) below. This is accomplished by simply dividing it in half:

$$s_r \text{ (pooled, sq root scale)} = \frac{s_r \text{ (pooled, original scale)}}{2} \tag{4}$$

If the true RSD of a measurement is less than 0.3, dividing by 2 gives a good approximation to the true RSD on the square root scale. Thus, Equation 4 is a good approximation for the estimate of RSD, s_r, if enough values of s_r are pooled together.

Table I shows a sample calculation of intracounter s_r using artificial data. The data are in two categories, differentiated by

TABLE I. Determination of Intracounter s_r

Low Range Example: 5 to 20.5 Total Fibers Counted

Orig. Count (f/mm²)	Recount (f/mm²)	Average (f/mm²)	Average* (total)	Std. Dev. (f/mm²)	s_r
18	32	25	20	9.90	0.396
10	5	7.5	6	3.54	0.471
18	9	13.5	11	6.36	0.471
9	21	15.0	12	8.48	0.566

$s_r = \sqrt{\Sigma (s_r^2)/4} = 0.48$

s_r (pooled, square root scale) = 0.48/2 = 0.24

High Range Example: ≥ 50.5 Total Fibers Counted

Orig. Count (f/mm²)	Recount (f/mm²)	Average (f/mm²)	Average* (total)	Std. Dev. (f/mm²)	s_r
318	253	285.5	100	46.0	0.161
90	118	104.0	82	19.8	0.190
68	97	82.5	65	20.5	0.249
108	84	96.0	75	17.0	0.177
83	61	72.0	57	15.6	0.216

s_r (pooled) = $\sqrt{\Sigma (s_r^2)/5} = 0.20$

s_r (pooled, square root scale) = 0.20/2 = 0.10

*Data are assigned to ranges based on the average of total fibers counted

loading range. There are four data pairs given under "Low Range" and five pairs under "High Range." Actual calculations should be based on about 15 or more pairs of counts since values of s_r based on too few counts may be quite biased.[13] The third column of Table I lists the averages of the pairs of results in fibers/mm². The fourth column gives the averages in terms of total fibers, which is the basis for assigning the data to the loading categories. The fifth column lists the standard deviations for the count pair. The sixth column gives an s_r for each filter, the result of dividing the average into the standard deviation. When the values in this last column are squared, averaged, and the square root of that result calculated, the result is the pooled relative standard deviation (0.48 for the low-range example). On the square root scale, the pooled s_r is approximately 0.48/2, or 0.24. The calculations for the high range are the same. The first sample in the high range differs from the rest in that only 40 fields were counted instead of 100, which is why the ratio of total fibers counted to fibers/mm² is not the same.

The initial estimate of pooled, intracounter s_r may be based on a limited amount of data. However, the "Sample Quality Test" given below requires that 10 percent of the field samples in each set be recounted, so the number of data pairs available for the s_r calculation constantly increases. Using this data to frequently recalculate s_r for each counter in the laboratory is a valuable quality assessment tool in itself. The pooled s_r value obtained from Equation 3 should have immediate meaning to most analysts since it is a relative standard deviation on the original scale.

There are only three reasons for not including all of these new data pairs in a recalculation of the intracounter s_r. The first reason is that there are so many new data pairs (more than one-fifth of all the available data pairs) that the value of s_r will depend primarily on that data. In that case, a randomly selected subset of data may be chosen. The second reason applies when there are definite reasons for suspecting that the samples actually belong in a new category. In that case, a new category could be created. The third reason is that individual values of s_r differ greatly from the others in a category even though the samples seem to fit in that category. An s_r that differs greatly from the others is sufficient reason to exclude it from the pooled data. There are formal tests for poolability of standard deviations[14] (which are approximately applicable for relative standard deviations), but they will not be discussed here.

As additional data pairs from the "Sample Quality Test" become available, that data can be used either to increase the amount of data in each sample category or to create more sample categories. Several decisions need to be made. A first consideration is that, since data generated by a counter in the past may not represent the current capability of that counter, a balance should be struck between eliminating older data and having enough data to determine s_r accurately. When there are 100 data pairs for a sample category, they should be assigned to smaller ranges (5 to 15, 15.5 to 25 fibers counted, etc.). The top range should be 80 to 100 fibers, even if the 100 fibers are counted in less than 100 fields, since the s_r values throughout this range are approximately equal. And when the number of ranges for a given sample type exceeds 5, it is advisable to establish a curve of s_r as a function of sample loading and to estimate s_r values from this curve.

Sample Quality Test

Samples and the procedures used to collect, transport, store, mount, and analyze them should be examined often to detect any problems. If there is reason to suspect a problem for any sample, the data for that sample should not be reported or used in any further calculations. Otherwise, sample data should only be rejected if they fail the test given here.

The intracounter s_r is needed to test the quality of the results obtained for a sample set. Also needed are data pairs for some of the samples in the set. NIOSH Method 7400 states that 10 percent of the samples in every set should be randomly selected for recounting. The QA Coordinator, or someone designated by the coordinator, should select and relabel the samples. The samples may then be combined with other relabeled samples, e.g., those from other counters for determining intralaboratory s_r, and given to the counter (or counters) for recounting. The data for each of the samples that have been counted twice by the same counter are then tested as follows:

$$\text{If } |y_1 - y_2| > 2.8\,\bar{y} \cdot s_r \text{ (pooled, sq root scale), reject sample} \qquad (5)$$

where:

$$y_1 = \sqrt{\text{original count}}$$

$$y_2 = \sqrt{\text{recount}}$$

$$\bar{y} = \frac{y_1 + y_2}{2}$$

This test differs from that given in earlier versions of NIOSH Method 7400 primarily in that the data are converted to the square root scale first. Revision 3 of NIOSH Method 7400 gives the test in the form given here. The derivation of Equation 5 is explained in "Remarks on Equation 5" in the appendix, and alternative tests are also described in the appendix. Table II gives two examples of how to evaluate data pairs using the values of s_r from Table I. Since the test is done on the square root scale, the intracounter s_r [pooled, square root scale] from Equation 4 is used.

The justification for converting to the square root scale is given in the "Distribution of Fiber Count Data" section of the Appendix. The conclusion given there is that Equation 5, which assumes normality, will be more appropriate on the square root scale for most loadings. If the data are left on the original scale, as in Revisions 1 and 2 of NIOSH Method 7400, the test results will only be different when the samples are lightly loaded and the counts differ widely. Even if the data are better described as lognormally distributed, as long as the intracounter RSD is less than 0.3, there is little difference in the power of the test in Equation 5 and an analogous test based on log-normality as described under "Test on the Log Scale" in the Appendix.

TABLE II. Evaluating Recounts Using Intracounter s_r

Low Range	
Square Root of First Count: (10 fibers = 13 f/mm²)	3.57
Square Root of Second Count (29 fibers = 37 f/mm²)	6.08
Average ($\bar{y}$)	4.82
Difference:	2.51
Historical s_r (low range):	0.24
$2.8 \cdot \bar{y} \cdot s_r$	3.24
Difference < 3.24:	accept
High Range	
Square Root of First Count: (65 fibers = 83 f/mm²)	9.10
Square Root of Second Count (46 fibers = 59 f/mm²)	7.65
Average ($\bar{y}$):	8.38
Difference:	1.44
Historical s_r (low range):	0.10
$2.8 \cdot \bar{y} \cdot s_r$	2.35
Difference < 2.35:	accept

TABLE III. Determining the Need for 100% Recounts

Number of Samples in 10% Recount	Number of Rejected Samples (Equation 5) that Indicate Need for 100% Recount
2– 7	≥ 2
8–16	≥ 3
17–28	≥ 4
29–40	≥ 5

If 15 samples are recounted, there is an 80% chance of deciding that the RSD for the set is greater than the established (historical) intracounter RSD, when the true set RSD is 75% greater than the established intracounter RSD.

The factor 2.8 in Equation 5 provides an upper limit for $|y_1 - y_2|$ that will be exceeded not more than 5 percent of the time when the actual RSD for the sample set equals the counter's established RSD. When that limit is exceeded, the sample is rejected. About 5 percent of the samples recounted will be rejected even when all the samples are of good quality. However, when too many of the recounted samples are rejected by Equation 5, then the RSD for this sample set may be higher than the established intracounter RSD. That conclusion can be drawn when the number of rejected samples match that listed in Table III. This test, based on all the count-recount data pairs that have been determined for a sample set, is an extension of the test in NIOSH Method 7400, which is performed for each recounted sample.

It should be noted that this test does not have high power to detect differences between the sample set RSD and the established intracounter RSD when the number of recounted samples is small. For example, even if as many as 15 samples are recounted from a sample set that has an actual RSD that is 75 percent greater than the established intracounter RSD, there is only an 80 percent chance that this test will detect a difference. The chance of detecting a difference drops for fewer recounts, becoming 40 percent when five samples in a set are recounted. Although the Appendix gives some alternatives to Equation 5, the alternative tests have similar power. Equation 5 is simple to apply, once the recounts have been performed, and it will confirm that there is a problem with the worst data.

When a sample set has high variability as indicated by Equation 5 and Table III, there are several courses of action, which depend primarily on how the data are to be used. In some cases, it is desirable to identify and eliminate the source of variation. If the microscope is functioning properly and the counter has not changed procedures, then it may be necessary to obtain new samples. The samples may have been collected under unusual circumstances, for example, with a highly charged cassette, or they may have a matrix that is very difficult to count, or they may have been improperly mounted. In other cases, it may be sufficient to report that the variability was high. Often, it is advisable to recount all the samples and perform the "sample quality test" for each. When a data pair is rejected by Equation 5, both numbers should be reported with the note that the data are more variable than normal for that kind of sample. The reason for reporting the data is that the data may still be useful for decision making. For example, when the count and recount differ greatly but both are below the exposure limit, a decision may still be made using these data.

Intralaboratory Bias and RSD

A laboratory is defined here as a number of analysts grouped together for quality assurance purposes. In a laboratory with only one counter, intralaboratory bias and intralaboratory s_r are the same as intracounter bias and intracounter s_r, so the following discussion assumes that the laboratory has two or more counters.

Intralaboratory bias can be detected by having all the counters in the laboratory count the same reference samples. As mentioned, these reference samples can be already mounted and counted samples from the laboratory's sample stream. If the average result obtained by one counter for the samples in one or more categories is somewhat higher or lower than the average for the other counters, then there may be bias. Instead of determining if the difference is significant, it may be just as simple, and much more informative, to investigate the possible causes. There may be one counter who is doing something obviously different, such as using a different graticule or misadjusting the microscope. At other times, the problem will be more subtle, and it may be necessary to statistically design an experiment to determine the cause.

Intralaboratory s_r must be calculated based on reference samples that have been counted by all the counters in the laboratory. The calculation is the same as for intracounter s_r except that the formula for standard deviation given in Equation 1 cannot be used when there are more than two counters. For three or more counters, use the standard formula:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2} \qquad (6)$$

Then s_r for each sample is calculated as in Equation 2 and pooled as in Equation 3.

The expected level of performance by a competent laboratory may depend on the types of samples. For occupational samples in the range 100–1300 fibers/mm^2, intralaboratory s_r values of 0.17–0.22 have been achieved by laboratories with good quality assurance programs.[12,15] If all the samples evaluated by a laboratory are from low concentration levels, then the s_r estimate may be larger simply due to the higher intrinsic (relative) variability of the samples.

The intralaboratory s_r is used to track the differences between the counters in the laboratory. If intralaboratory s_r changes significantly, the cause should be investigated. Although increases in intralaboratory s_r are of most concern, the reasons for decreases are also of interest. Increased intralaboratory s_r may indicate the presence of bias.

Interlaboratory Bias and RSD

Most analytical methods, including those in the *NIOSH Manual of Analytical Methods*,[3] do not address differences between laboratories. Such differences are considered to be interlaboratory biases, and it is assumed that they are negligible because of the ready availability and widespread use of highly accurate calibration standards and reference materials. Such is not the case for fiber counting. As mentioned in "Variability and Bias" above, calibration standards for fiber counting are not readily available, and the results for fiber counting as performed by human counters are not easily adjusted.

In theory, it should be possible to eliminate fiber count differences among laboratories. In practice, these differences are difficult to eliminate and the reasons for them difficult to identify. Ideally, a reference sample counted by the OSHA laboratory in Salt Lake City, Utah, would be available since that laboratory determines regulatory compliance. In practice, one way a laboratory can approach this ideal is by participating in the PAT program; the OSHA laboratory consistently reports PAT asbestos results close to the reference value. However, PAT samples are laboratory-generated, are not identical, and are not, therefore,

ideal reference samples.

Another way to approach this ideal would be to exchange slides containing mounted field samples among laboratories. This is less than ideal because the OSHA laboratory cannot participate in each of these round robins, and it is logistically difficult for more than six laboratories to participate in such a round robin. Nevertheless, OSHA now requires laboratories counting fibers for compliance purposes to exchange samples from their workload with other laboratories and to analyze and post the results.[16] Each laboratory is required to exchange samples at least twice a year with at least two other laboratories.

A quality assurance program for fiber counting should use both types of reference sample: mounted samples exchanged with other laboratories and the audit samples set quarterly to participants in either the AAR or the PAT program. These samples provide a means of detecting biases. In the case of the PAT program, a laboratory can compare its result with the average result of the reference laboratories, a group of approximately 80 highly qualified laboratories. Note that the analysis of PAT or AAR samples does not fill the OSHA requirement to exchange field samples with other laboratories.

Although these procedures bring laboratories into closer agreement, differences can persist, and NIOSH Method 7400 refers to these differences as interlaboratory s_r. These differences may be considered as residual biases by some, but the differences may not be constant, and finding an assignable cause is difficult. This is not meant to imply that these differences should be accepted; the purpose of the interlaboratory s_r is to quantify differences and to make them known by using them to assign confidence limits to reported fiber counts.

The determination of interlaboratory s_r is based on recounts done by persons in different laboratories. To obtain sufficient data to calculate interlaboratory s_r, a group of laboratories will want to exchange samples at a greater rate than that required by OSHA. A simple plan calls for approximately 20 samples to be counted in each laboratory in the group. The samples will already have been mounted and counted in one of the laboratories. Each laboratory can contribute equally to the number of samples, say five from each of four laboratories. The samples contributed should be representative of the laboratory's workload as far as loading and other characteristics. As the already-mounted samples arrive in a laboratory, each is counted by a different (randomly chosen) counter for that laboratory. The samples are then sent to the next laboratory and the count data sent to the coordinating laboratory.

The calculation of interlaboratory s_r is performed by the coordinating laboratory as follows. For each sample, the results of the participating laboratories should be averaged, and a standard deviation (Equation 6) and s_r (Equation 2) calculated. The calculations are done in fibers/mm^2. Before pooling these inter-laboratory s_r (total) values, the Poisson component of variability is removed to obtain the subjective inter-laboratory component, $s_{r,s}$, as follows:

$$s_{r,s} = \sqrt{s_r^2(\text{total}) - s_r^2(\text{Poisson})} \qquad (7)$$

$$= \sqrt{s_r^2(\text{total}) - 1/\text{count}}$$

where: count = the average of the counts reported by the laboratories for the sample.
(If $S_r^2 < 1/\text{count}$, let $s_{r,s} = 0$)

These $s_{r,s}$ values are then pooled in the same way as was done for intracounter s_r in Table I, but the result does not need to be converted to the square root scale. Note that the removal of the Poisson component in Equation 7 means that the samples used for determining interlaboratory s_r need not be put into categories based on fiber loading. Some groups of laboratories may wish to put especially difficult samples, e.g., asbestos cement dust, into a separate category and calculate an interlaboratory $s_{r,s}$ just for use with that kind of sample.

The simple plan just given for arriving at interlaboratory $s_{r,s}$ should serve the needs of most groups. There may be questions about how to pick samples, how to assign counters, or whether the data from samples mounted in different laboratories are poolable. If these are a concern, the advice of a statistician should be sought before data are collected or analyzed. Note that it is worthwhile to reduce differences among laboratories[17] since data that are biased will increase the interlaboratory $s_{r,s}$ and negate the advantage of determining it. If a laboratory is not required to exchange samples under the OSHA rule, or if not enough data have been collected since forming the laboratory group, or if there appear to be unresolved problems with the data collected, the conservatively high estimate of variability given in NIOSH Method 7400 can be used, as discussed below.

For most purposes, the interlaboratory $s_{r,s}$, not the intracounter or intralaboratory s_r, is used to calculate confidence limits on reported data. Intracounter s_r may be used for special studies where the purpose is to measure relatively small differences in fiber counts and for which all analyses are performed by one counter. Similarly, when counts for a study are all produced by one laboratory, but not necessarily one counter, it is appropriate to use the intralaboratory s_r. When comparisons are being made between laboratories or when results are to be compared to the PEL or other criterion (REL, TLV®), the interlaboratory $s_{r,s}$ should be used. In general, when comparing a fiber count to the OSHA PEL, one needs to be confident that a fiber count made by any other laboratory, including the OSHA laboratory, will produce a similar result. The following discussion uses interlaboratory $s_{r,s}$ to estimate confidence limits.

When the interlaboratory $s_{r,s}$ has been established in a sample exchange program, it is appropriate to use it to calculate confidence limits for each sample instead of using the graph in NIOSH Method 7400. The following formulae recombine the Poisson component of variability with $s_{r,s}$ to give the 90 percent confidence limits:

$$\text{UCL} = \frac{2x + 2.25 + \sqrt{(2.25 + 2x)^2 - 4\,(1 - 2.25 s_{r,s}^2)\,x^2}}{2(1 - 2.25 s_{r,s}^2)} \qquad (8)$$

$$\text{LCL} = \frac{2x + 4 - \sqrt{(4 + 2x)^2 - 4(1 - 4 s_{r,s}^2)\,x^2}}{2(1 - 4 s_{r,s}^2)} \qquad (9)$$

where:

$s_{r,s}$ = subjective interlaboratory s_r
x = total fibers counted on sample
UCL = upper confidence limit
LCL = lower confidence limit

These formulae were derived from the work of Ogden[12] by extrapolating to higher $s_{r,s}$ and to different values of x. The UCL formula is not valid for values of $s_{r,s}$ greater than 0.67, and the LCL formula is not valid for values of $s_{r,s}$ greater than 0.5. The confidence limits calculated for a fiber count provide a range of values within which the mean count of a group of competent laboratories is expected to fall 90 percent of the time.

These confidence limits are in units of total fibers to be consistent with NIOSH Method 7400. Any conversions to concentration units are performed on the confidence limits in the same way as would be done for x, the original fiber count. As an example, if the interlaboratory $s_{r,s}$ is 0.25 and 24 fibers have been counted on a sample, the above equations give 13.8 fibers and 42.8 fibers as the confidence limits. If these fibers were counted in 100 fields of 0.00785 mm^2 on a 25-mm filter and the air volume of the sample was 500 liters, then the confidence limits on the air concentration are 0.014 and 0.042 fibers/cc. Since the primary use of a fiber count is to compare with an exposure limit such as the OSHA PEL, the upper confidence limit is of most interest. For this example, the upper confidence limit on the air concentration is 0.042 fibers/cc.

NIOSH Method 7400 gives an example calculation of confidence limits when 24 fibers, the same as in the example just given, have been counted. Based on the graph in the method, the resulting confidence limits are 0.011 and 0.077 fibers/cc. We can no longer be confident that the fiber concentration, nominally 0.024 fibers/cc, is less than 0.05, 0.06, or even 0.07 fibers/cc. The confidence band is wider because the graph is based on an $s_{r,s}$ of 0.45.[1,15] The graph was produced by substituting 0.45 for $s_{r,s}$ and substituting various values for x in Equations 8 and 9, then plotting UCL and LCL as a percentage of the substituted x. The graph, or the conservatively high estimate of $s_{r,s}$ (0.45), can be used when the data for calculating interlaboratory $s_{r,s}$ are not available. When using the graph in NIOSH Method 7400, note that it gives the percent differences to be added to and subtracted from the original fiber count. These percent differences are relatively constant (+213% and −49%) for fiber counts above 30 because the Poisson component of variability becomes less important. Thus, the confidence limits for 50 fibers counted are 25 and 157 fibers.

A group of laboratories exchanging samples may achieve an interlaboratory $s_{r,s}$ less than the estimate given in NIOSH Method 7400. It is then reasonable for each laboratory in the group to use confidence limits based on that $s_{r,s}$ when reporting their sample results. The lower $s_{r,s}$ indicates that these laboratories have successfully lowered their variability. The laboratories with smaller $s_{r,s}$ values can report results with tighter confidence limits, thereby increasing the number of definitive results (those definitely above or below a given level). It is possible that all the laboratories in a given exchange group, especially a small group, agree well with each other, but that they are all biased. However, participation of laboratories and counters in audit programs such as the PAT program and the AAR reduces the likelihood of that.

Usually, intracounter s_r is about one-half of the interlaboratory s_r.[15] If the intracounter s_r for a given set of samples is a little higher than the historically established s_r, the effect on interlaboratory s_r and sample confidence limits is negligible, particularly if the graph in NIOSH Method 7400 is being used. However, if many of the samples in a set fail the criterion in Equation 5, the confidence limits given by Equations 8 and 9 are not applicable.

Samples are taken from an environment in order to provide an estimate of the airborne concentration in that environment. The above discussion has treated the analytical variability of individual samples at length. Since the environmental variability can often overshadow the analytical variability, strategies have been developed by others to improve the estimates of environmental concentrations.[18] One important strategy is to take multiple samples. If K samples are taken, the relative standard deviation due to environmental variability is reduced by the factor $1/\sqrt{K}$. Multiple samples taken from the same environment and analyzed by a laboratory can also reduce the intralaboratory s_r by the same $1/\sqrt{K}$ factor. However, the largest component of the analytical variability is the between laboratory variability, which is not reduced by the use of multiple samples. Therefore, multiple samples are useful for improving the estimate of airborne concentration from an environment but are not generally useful for reducing the analytical variability. This emphasizes that reducing the interlaboratory s_r through sample exchanges is of primary importance in improving the confidence limits that a laboratory can report for any given sample.

Conclusions and Recommendations

Each laboratory should have a Quality Assurance Coordinator responsible for assessing the quality of fiber count data. The coordinator will include blind reference samples into the laboratory's sample stream, obtain the recount data, and use it to calculate intracounter s_r and intralaboratory s_r. Interlaboratory s_r can also be calculated based on sample exchanges with other laboratories. There are several ways to determine these components of variance, but the simple calculations discussed in this article and outlined in the current version of NIOSH Method 7400 will suffice for most laboratories.

To summarize:

- Calculate a Student's t-statistic for (recount–count) differences in the sample set in order to detect statistically significant bias. If bias exists, determine its cause and correct it.
- Calculate an intracounter s_r (Equations 1–4, Table I) for each counter and each sample category (i.e., loading range, type of interference, etc.) based on recounts by the same counter on recently counted sample slides. Plot s_r as a function of loading if enough data are available. Recounting older samples provides a method of detecting drift in a counter's performance.
- Apply the "Sample Quality Test" (Equation 5, Table II) to data pairs obtained by recounting 10 percent of the samples in each set. The recount is performed by the same counter on slides counted within the last day. If the number of samples "rejected" exceeds the number in Table III, reject the sample set or recount all the samples and apply the test to each sample individually.
- Calculate intralaboratory s_r based on recounts by all counters. Check for changes in s_r and for biases between counters.
- Calculate interlaboratory $s_{r,s}$ based on recounts of mounted field samples already counted in two or more other laboratories (Equation 7). Be alert to bias and, if detected, correct it and recalculate interlaboratory $s_{r,s}$. If recount data are not yet available, an $s_{r,s}$ of 0.45 is assumed.
- Use the interlaboratory $s_{r,s}$ and the total number of fibers counted on a sample to calculate confidence limits (Equations 8 and 9) for that fiber count. If the $s_{r,s}$ of 0.45 has been assumed, use the graph in NIOSH Method 7400. Report these confidence limits with the fiber count.

Intracounter s_r for properly loaded samples that have low background can theoretically approximate a value as low as 0.1. An intralaboratory s_r of 0.17 – 0.22 has been achieved by competent laboratories. NIOSH Method 7400 gives a conservatively high estimate of 0.45 for the subjective component of interlaboratory s_r.

Appendix

In this appendix, some topics mentioned in the main text are

further explained. The topics are: how to test for bias; background on Equation 5 (the "Sample Quality Test"); the distribution of fiber count data and justification for the square root transform; and mention of several alternatives to the use of intracounter s_r and Equation 5 for testing data.

Bias

The Student's t distribution can be used to test for bias in count data. Specifically, we are interested in seeing if the true average difference (recount–count) is equal to zero. If there are k data pairs, the first step is to compute the k differences:

$$d = \sqrt{x_2} - \sqrt{x_1}$$

where:

x_2 = the recount
x_1 = the original count

The reason for using the square root is discussed in the "Distribution of Fiber Count Data" section below. The next step is to calculate

$$t = \frac{\bar{d}}{s_d/\sqrt{k}} \qquad (10)$$

where:

$\bar{d}$ = the average of the differences
s_d = the estimated standard deviation of the differences

The value obtained for the t statistic should be compared with the value that is located in the row and column of a Student's t table corresponding to $k-1$ degrees of freedom and the 0.05 significance level (for the typical two-tailed table). If the value obtained in Equation 10 is greater than the table value, then bias can be assumed to be responsible.

Remarks on Equation 5

For a normal random sample of size n, the probability density function has been given for $s/\bar{x}$,[19] where s denotes the sample standard deviation and $\bar{x}$ is the sample mean. Although the function was based on a sample standard deviation defined as:

$$s = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(x_i - \bar{x})^2} \qquad (11)$$

it is easy to modify the result for s defined as:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2} \qquad (12)$$

If Prob ($\bar{x} < 0$) is very small, the noncentral t distribution describes the distribution of $\sqrt{n} \cdot \bar{x}/s$. To arrive at Equation 5 in the text, the 95 percent upper confidence limit on the sample s_r was determined as a multiple of the true s_r value.[19,20] This multiplier depends somewhat on the value of the true s_r. However, for $s_r < 0.3$ on the original scale, the multiplier is less than or equal to 2.8, the value used in Equation 5. That is:

$$\Pr\left(\frac{\left(\frac{1}{\sqrt{2}}|y_1 - y_2|\right) \div \bar{y}}{s_r \text{ (pooled, sqrt scale)}} < \frac{2.8}{\sqrt{2}}\right) \geq 0.95 \qquad (13)$$

Distribution of Fiber Count Data

Equation 5 assumes that, for fibers/mm² data, $\sqrt{x_2} - \sqrt{x_1}$ data are approximately normally distributed and have an RSD corresponding to that computed for the category in which the average for the two fiber counts falls. The facts associated with Poisson deposition of the fibers and Poisson distribution of the actual counted fibers are as follows: 1) if the actual count is at least 15 fibers in 100 fields, the counts are approximately normal on both the original and square root scales and 2) the absolute standard deviation of the counts is approximately constant on the square root scale. Thus, even if the counts themselves (which reflect both deposition of fibers on the filter and the counting process) are Poisson distributed, square root transformation of those counts does not assure constant RSD. Also, the data used in the test are in fibers/mm², and these do not, in general, follow a Poisson distribution, even if the deposition itself is Poisson. Furthermore, even if the deposition is Poisson, the fact that a particular person counts fibers and uses a particular microscope, introduces variability beyond that expected from the Poisson. These concerns are discussed next: first with regard to normality, then with regard to constant RSD.

The first issue is normality. Assuming Poisson deposition of fibers and Poisson counts of these fibers, what is the degree of non-normality of the fibers/mm² data on the original, square root, and log scales? Application of the stopping rule to samples with different loadings results in variable numbers of fields being counted as well as variable numbers of fibers being counted. When loadings are low, a variable number of fibers in a constant area of the filter (100 fields) is counted. Likewise, when loadings are high, a variable number of fibers in a constant area of the filter (20 fields) is counted. When loadings are intermediate, approximately 100 fibers are counted in a variable area (20–100 fields) of the filter. A fiber density of ≤ 0.8 fibers/field corresponds to low loading since the probability is high that fewer than 100 fibers will be counted in 100 fields. A fiber density of > 8 fibers/field corresponds to high loading since > 100 fibers will have been counted in 20 fields or less. Fiber densities in the range of 0.8 to 8 fibers/field can be taken as the intermediate range.

Thus, in the cases of low and high loadings as just defined, the area of the filter examined is constant, and all the variability is in the number of fibers counted. Since the fibers counted on the filter are assumed to be Poisson, the fiber/mm² measurement is also from a Poisson distribution whose mean is $100 \cdot \mu$ ($20 \cdot \mu$ for high loadings), where μ is the true mean count for one field. For sufficiently high Poisson counts (a count of at least 15 fibers in 100 fields), as stated above, the square roots and the original counts are approximately normally distributed, as are the fiber/mm² values for the low and high loadings. The logarithms of these counts, computer simulations indicate, are not normally distributed for low loadings, but are for high loadings.

For intermediate loadings, the complication of having variable numbers of both fibers and fields counted, makes it difficult to make any statement about the correct transformation to normality of the fiber/mm² values based on theory alone. Computer simulations suggest that the transformation that is best at producing a normal distribution in this loading range depends on the loading. For 1 fiber/field, the untransformed data are not normally distributed, the square root transformed data are marginally normal, and the log transformed data are nearly normal. At 2 fibers/field, only the log transformed data can be said to be normally distributed. At 4 fibers/field, the log transformed data are normal, and the square root transformed data are normal again, but the original data are not normal.

For the entire range under consideration, provided at least 15 fibers are counted, the square root and log transformations in-

duce a normal distribution of the fiber/mm² data at most loadings. Thus, for statistical tests that assume normality, such as Equation 5, the result will be accurate on the square root scale for most loadings. However, for loadings of about 2 fibers/field, where the log transformation is more nearly normal than the square root, a test based on the log transform seems more reasonable than one based on the square root. Such a test is also discussed below. The above remarks assume that the counted fibers are approximately Poisson distributed. The authors have not studied the sensitivity to non-normality of the test in Equation 5.

The second issue, after normality, is that of constant RSD within each sample category. For Poisson counts, the square root transform induces approximately constant variance, whatever the loading. Thus, if the categories cover a relatively wide range of loadings and most of the variability is Poisson variability due to counts, a version of Equation 5 based on absolute standard deviation rather than s_r might be useful. This approach is mentioned below. On the other hand, if the sample categories are relatively narrow loading ranges, for counts that have considerable variability in excess of Poisson variability, the relative standard deviation will be slightly more constant than the absolute standard deviation. For moderately excessive variability and narrow bands, the two measures are about equally variable.

Test on the Log Scale

The preceding section argues that fiber count data are normal on the square root scale. However, as mentioned in "Variability and Bias," the distribution has also been described as lognormal in the literature. Thus, it is reasonable to compare the test in Equation 5, which assumes square root normality, with an analogous test based on log normality. A test analogous to Equation 5, but based on the log scale, is:

$$\text{If } |\ln(x_1) - \ln(x_2)| > 2.77 \cdot s_r, \text{ reject the sample} \qquad (14)$$

where:

s_r = the established intracounter s_r on the original scale

Table IV provides a comparison of the power of the tests in Equations 5 and 14, assuming an historical s_r of 0.30 on the original scale. In Table IV, the first column is the ratio of the true RSD of the count–recount data pairs being tested to the established intracounter RSD. The table shows that neither the square root test of Equation 5 (column 2) nor the log test of Equation 14 (column 3) has great power, even when the test being used matches the distribution of the data. For example, when the true RSD of the data pairs being tested is 2.92 times the established intracounter RSD of 0.30, the tests have a 43 percent (square root) and 56 percent (log) chance of accepting the data. The aggregate test using Table III has more power.

The fourth column of Table IV shows the probability of accepting the data when the square root test is used on lognormally distributed data, again assuming an historical RSD of 0.30 on the original scale. The table shows, however, that never is there an appreciable difference in power from the test based on the logs of the data. We conclude that the square root test can be used even when the data are lognormally distributed. However, as stated above, there could be instances, for example, when the true filter deposition is about 2 fibers/field, that use of Equation 14 would be more appropriate.

TABLE IV. Comparison of Outlier Tests Assuming Square Root and Lognormal Distributions

RSD(true)* / RSD(hist)	Probability of Accepting Samples		
	Sq Root Test & Sq Root Dist.	Log Test & Log Dist.	Sq Root Test & Log Dist.
1.0	0.95	0.95	0.95
1.5	0.80	0.83	0.83
2.92	0.43	0.56	0.57
3.74	0.29	0.48	0.51
4.70	0.11	0.42	0.45

*RSD(hist) = 0.3

Test Based on Absolute Standard Deviation

A test can be based on the historical standard deviation instead of the historically established intracounter s_r. The data pairs to be used for determining the standard deviation should be grouped into categories just as was done to determine intracounter s_r. For these calculations, the fibers/mm² data are converted to their square roots (y_1 and y_2) immediately since there is no simple way to convert the standard deviation to the square root scale as there is for s_r. The calculation of standard deviation for each sample is similar to Equation 1 ($s = 0.707\,|y_1 - y_2|$), and the standard deviations are pooled as in Equation 3. As an example, the low-range data in Table I yield an s[pooled, square root scale] of 0.929. The data to be tested are also converted to the square root scale:

$$\text{If } |y_1 - y_2| > 2.77 \cdot s \text{ (pooled, sqrt scale)}, \text{ reject sample} \qquad (15)$$

The factor 2.77 provides an upper limit for $|y_1 - y_2|$ that will be exceeded not more than 5 percent of the time. Factors of 3.64 and 4.65 correspond to 1 percent and 0.1 percent upper limits.[21] This test is more powerful (will correctly reject more samples) than the s_r test in Equation 5,[22] but for most cases the power difference is relatively small. The test based on Equation 15 is appropriate when most of the variability of the counts is due to the Poisson distribution of counts.

Test Based on Pooled Sample Set s_r

Another way to use the data when every sample in a set has been recounted is to calculate a pooled s_r for each sample category for comparison to the established intracounter s_r for that category. If it can be established that the RSD corresponding to the pooled s_r is no greater than the RSD corresponding to the established intracounter s_r, then the results can all be reported. If the former RSD is appreciably greater than the established intracounter s_r, it may be necessary to widen the reported confidence limits for the samples. This test has some difficulties. If the samples in the set fall into different loading categories there will be more than one pair of s_r values to compare, and for categories with only a few samples, there will be little power to detect differences in s_r. Also, this test is not as simple or flexible as the sample-by-sample test given in Equation 5.

Test Based on Means

Tests of sample quality could be based on means rather than variability. One such test is a chi-square test that seeks to determine if means μ_1 and μ_2 are equivalent, where μ_1 and μ_2 are the true means as estimated by the count and recount, x_1 and x_2. This test also requires that the intracounter s_r be determined. If a minimum intracounter RSD (Poisson component only) is assumed, the true means of a pair of counts with a true average of 20 would have to differ by 11 or more for the test to have 95 percent power to reject the data. For an average of 40 counts, the difference of the true means would have to be 13. The differences can be decreased to 8.5 and 10 fibers, respectively, if

80 percent power is acceptable. These differences would have to be even greater for larger, more realistic, intracounter RSD. Thus, as with the tests based solely on variability, this test does not have great power to detect differences.

In fact, this is the same test as that given in Equation 15. In using this as a test of means, we are assuming a different alternative, namely, that the true means corresponding to the two counts may differ. In using it as a test of variances, we are testing whether the absolute variances differ. Since the fields counted on a slide are a random selection, it may make more sense to view the alternative hypothesis for the test in Equation 15 as inequality of variances associated with two random samples from the same source.

Acknowledgment

The authors thank those who contributed to the evolution of this article, including the following: M. Attfield, P. Bierbaum, L. Bloomfield, K. Busch, M. Carmel, D. Crane, T. Dinh, L. Doemeny, P. Eller, R. Hartle, R. Zumwalde, and an anonymous reviewer for the journal.

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Received 7/26/88; review decision 8/29/88; revision 6/12/89; accepted 6/15/89

Errata for "Quality of Fiber Count Data" (AIH, Nov., 1989).

In the section on interlaboratory s_r (relative standard deviation), the article states "...the calculations are done in total fibers, not fibers/mm^2." This does not always work in practice because of the rule that says to stop counting after the field in which the 100th fiber is counted. This means that for the more heavily loaded samples, the fiber counts obtained by the participating laboratories will all be near 100, with little variability.

Most of the variability will be in t number of fields counted, which is n taken into account. We therefo recommend that fibers/mm^2 data be us to calculate interlaboratory s_r. T result can then be used to calcula (Equation 7) the subjective compone of interlaboratory s_r, designated s_r, Even though the other term in th equation is 1/count, any discrepan due to the change will be small.

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To Mike Underhill	From Marc Hunt
Co. Trident Env Services	Co. Enviro Investigations
Dept.	Phone # (919) 544-7500
Fax # (803) 821-1767	Fax # (919) 544-2199

The following block of text can be substituted for that at the bottom of the left hand column on page 281:

The calculation of interlaboratory s_r is performed by the co-ordinating laboratory as follows. For each sample, the results of the participating laboratories should be averaged, and a standard deviation (Equation 6) and s_r (Equation 2) calculated. The calculations are done in fibers/mm2. Before pooling these inter-laboratory s_r (total) values, the Poisson component of variability is removed to obtain the subjective inter-laboratory component, $s_{r,s}$, as follows:

$$s_{r,s} = \sqrt{s_r^2(\text{total}) - s_r^2(\text{Poisson})} \qquad (7)$$

$$= \sqrt{s_r^2(\text{total}) - 1/\text{count}}$$

where: count = the average of the counts reported by the laboratories for the sample.
(If $S_r^2 < 1/\text{count}$, let $s_{r,s} = 0$)

These $s_{r,s}$ values are then pooled in the same way as was done

Received 3/97

ASBESTOS and OTHER FIBERS by PCM 7400

Various MW: Various CAS: Various RTECS: Various

METHOD: 7400, Issue 2 **EVALUATION: FULL** **Issue 1: Rev. 3 on 15 May 1989**
Issue 2: 15 August 1994

OSHA : 0.1 asbestos fiber (> 5 μm long)/cc; 1 f/cc/30 min excursion; carcinogen
MSHA: 2 asbestos fibers/cc
NIOSH: 0.1 f/cc (fibers > 5 μm long)/400 L; carcinogen
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline, anisotropic

SYNONYMS [CAS #]: actinolite **[77536-66-4]** or ferroactinolite **[15669-07-5]**; amosite **[12172-73-5]**; anthophyllite **[77536-67-5]**; chrysotile **[12001-29-5]**; **serpentine [18786-24-8]**; crocidolite **[12001-28-4]**; tremolite **[77536-68-6]**; amphibole asbestos **[1332-21-4]**; refractory ceramic fibers **[142844-00-6]**; fibrous glass.

SAMPLING

SAMPLER: FILTER (0.45- to 1.2-μm cellulose ester membrane, 25-mm; conductive cowl on cassette)

FLOW RATE*: 0.5 to 16 L/min

VOL-MIN*: 400 L @ 0.1 fiber/cc
-MAX*: (step 4, sampling)
*Adjust to give 100 to 1300 fiber/mm^2

SHIPMENT: routine (pack to reduce shock)

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: see EVALUATION OF METHOD

OVERALL PRECISION ($\hat{S}_{rT}$): 0.115 to 0.13 [1]

ACCURACY: see EVALUATION OF METHOD

MEASUREMENT

TECHNIQUE: LIGHT MICROSCOPY, PHASE CONTRAST

ANALYTE: fibers (manual count)

SAMPLE PREPARATION: acetone - collapse/triacetin - immersion method [2]

COUNTING RULES: described in previous version of this method as "A" rules [1,3]

EQUIPMENT:
1. positive phase-contrast microscope
2. Walton-Beckett graticule (100-μm field of view) Type G-22
3. phase-shift test slide (HSE/NPL)

CALIBRATION: HSE/NPL test slide

RANGE: 100 to 1300 fibers/mm^2 filter area

ESTIMATED LOD: 7 fibers/mm^2 filter area

PRECISION ($\hat{S}_r$): 0.10 to 0.12 [1]; see EVALUATION OF METHOD

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The method gives an index of airborne fibers. It is primarily used for estimating asbestos concentrations, though PCM does not differentiate between asbestos and other fibers. Use this method in conjunction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. Fibers < ca. 0.25 μm diameter will not be detected by this method [4]. This method may be used for other materials such as fibrous glass by using alternate counting rules (see Appendix C).

INTERFERENCES: If the method is used to detect a specific type of fiber, any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

OTHER METHODS: This revision replaces Method 7400, Revision #3 (dated 5/15/89).

REAGENTS:

1. Acetone,* reagent grade.
2. Triacetin (glycerol triacetate), reagent grade.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically conductive extension cowl and cellulose ester filter, 0.45- to 1.2-μm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use to check for clarity and background. Discard the filter lot if mean is ≥5 fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.
   NOTE 2: The electrically conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling.
   NOTE 3: Use 0.8-μm pore size filters for personal sampling. The 0.45-μm filters are recommended for sampling when performing TEM analysis on the same samples. However, their higher pressure drop precludes their use with personal sampling pumps.
   NOTE 4: Other cassettes have been proposed that exhibit improved uniformity of fiber deposit on the filter surface, e.g., bellmouthed sampler (Envirometrics, Charleston, SC). These may be used if shown to give measured concentrations equivalent to sampler indicated above for the application.
2. Personal sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow-rate requirements (see step 4 for flow rate), with flexible connecting tubing.
3. Wire, multi-stranded, 22-gauge; 1", hose clamp to attach wire to cassette.
4. Tape, shrink- or adhesive-.
5. Slides, glass, frosted-end, pre-cleaned, 25 x 75-mm.
6. Cover slips, 22- x 22-mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
7. Lacquer or nail polish.
8. Knife, #10 surgical steel, curved blade.
9. Tweezers.

EQUIPMENT:

10. Acetone flash vaporization system for clearing filters on glass slides (see ref. [5] for specifications or see manufacturer's instructions for equivalent devices).
11. Micropipets or syringes, 5-μL and 100- to 500-μL.
12. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
13. Graticule, Walton-Beckett type with 100-μm diameter circular field (area = 0.00785 mm^2) at the specimen plane (Type G-22). Available from Optometrics USA, P.O. Box 699, Ayer, MA 01432 [phone (508)-772-1700], and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].
    NOTE: The graticule is custom-made for each microscope. (see APPENDIX A for the custom-ordering procedure).
14. HSE/NPL phase contrast test slide, Mark II. Available from Optometrics USA (address above).
15. Telescope, ocular phase-ring centering.
16. Stage micrometer (0.01-mm divisions).

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape. For personal sampling, fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward.
    NOTE: The cowl should be electrically grounded during area sampling, especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (i.e., cold water pipe).
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [6]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E, of 100 to 1300 fibers/mm^2 ($3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area A_c = 385 mm^2) for optimum accuracy. These variables are related

to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. The collection efficiency does not appear to be a function of flow rate in the range of 0.5 to 16 L/min for asbestos fibers [7]. Relatively large diameter fibers (>3 μm) may exhibit significant aspiration loss and inlet deposition. A sampling rate of 1 to 4 L/min for 8 h is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes ($\leq$400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If $\geq$ 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement, and a maximum sampling rate of 2.5 L/min [3].

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.
   NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index $\leq$1.46. This method collapses the filter for easier focusing and produces permanent (1 - 10 years) mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400 - revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7,8,9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9,10,11].

NOTE 2: Excessive water in the acetone may slow the clearing of the filter, causing material to be washed off the surface of the filter. Also, filters that have been exposed to high humidities prior to clearing may have a grainy background.

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [2].
   NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
   a. Cut wedges of ca. 25% of the filter area with a curved-blade surgical steel knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.
      NOTE: Static electricity will usually keep the wedge on the slide.

b. Insert slide with wedge into the receiving slot at base of "hot block". Immediately place tip of a micropipet containing ca. 250 µL acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger-button while holding pipet firmly in place. After waiting 3 to 5 sec for the filter to clear, remove pipet and slide from their ports.
   CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous use of this device in an unventilated space may produce explosive acetone vapor concentrations.

c. Using the 5-µL micropipet, immediately place 3.0 to 3.5 µL triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass.
   NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.

d. Mark the outline of the filter segment with a glass marking pen to aid in microscopic evaluation.

e. Glue the edges of the cover slip to the slide using lacquer or nail polish [12]. Counting may proceed immediately after clearing and mounting are completed.
   NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturers instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings and major servicing.
    a. Each time a sample is examined, do the following:
       (1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.
       (2) Focus on the particulate material to be examined.
       (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.
    b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:
       (1) Center the HSE/NPL phase-contrast test slide under the phase objective.
       (2) Bring the blocks of grooved lines into focus in the graticule area.
          NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when centered in the graticule area. Blocks 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.
       (3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Document the laboratory's precision for each counter for replicate fiber counts.
    a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [13]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field

and reference samples (e.g., PAT, AAR, commercial samples). The Quality Assurance Officer should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter precision. Obtain separate values of relative standard deviation (S_r) for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, and >50 to 100 fibers in 100 graticule fields. Maintain control charts for each of these data files.

NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [9]

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm^2) exceeds 2.77 $(X)S_r'$, where X = average of the square roots of the two fiber counts (in fiber/mm^2) and $S_r' = \frac{S_r}{2}$, where S_r is the intracounter relative standard deviation for the appropriate count range (in fibers) determined in step 11. For more complete discussions see reference [13].

NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data [13].

NOTE 2: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

14. The analyst is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. An ergonomically designed chair should be used, with the microscope eyepiece situated at a comfortable height for viewing. External lighting should be set at a level similar to the illumination level in the microscope to reduce eye fatigue. In addition, counters should take 10-to-20 minute breaks from the microscope every one or two hours to limit fatigue [14]. During these breaks, both eye and upper back/neck exercises should be performed to relieve strain.

15. All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program for asbestos and routinely exchange field samples with other laboratories to compare performance of counters.

MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.

17. Adjust the microscope (Step 10).

NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 μm) [4].

18. Counting rules: (same as P&CAM 239 rules [1,10,11]: see examples in APPENDIX B).

a. Count any fiber longer than 5 μm which lies entirely within the graticule area.
   (1) Count only fibers longer than 5 μm. Measure length of curved fibers along the curve.
   (2) Count only fibers with a length-to-width ratio equal to or greater than 3:1.

b. For fibers which cross the boundary of the graticule field:

(1) Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule a above.
(2) Do not count any fiber which crosses the graticule boundary more than once.
(3) Reject and do not count all other fibers.

c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.

d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.

19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.

NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 seconds per field is appropriate for accurate counting.

NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters >1 μm), polarized light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [15].

NOTE 3: Do not count at edges where filter was cut. Move in at least 1 mm from the edge.

NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [5].

NOTE 5: Counts are to be recorded on a data sheet that provides, as a minimum, spaces on which to record the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, average count, fiber density, and commentary. Average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/mm^2) is defined as the average count (fibers/field) divided by the field (graticule) area (mm^2/field).

CALCULATIONS AND REPORTING OF RESULTS

20. Calculate and report fiber density on the filter, E (fibers/mm^2), by dividing the average fiber count per graticule field, F/n_f, minus the mean field blank count per graticule field, B/n_b, by the graticule field area, A_f (approx. 0.00785 mm^2):

$$E = \frac{\left(\frac{F}{n_f} - \frac{B}{n_b}\right)}{A_f}, \text{ fibers/mm}^2.$$

NOTE: Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of filter area covered with particulate should be reported as "uncountable" or "probably biased." Other fiber counts outside the 100-1300 fiber/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased."

21. Calculate and report the concentration, C (fibers/cc), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (approx. 385 mm^2 for a 25-mm filter):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}.$$

NOTE: Periodically check and adjust the value of A_c, if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.

NOTE: Precision depends on the total number of fibers counted [1,16]. Relative standard deviation is documented in references [1,15-17] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Fig. 1).

EVALUATION OF METHOD:

A. This method is a revision of P&CAM 239 [10]. A summary of the revisions is as follows:
   1. Sampling:
      The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m^3 full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [10].
   2. Sample Preparation Technique:
      The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,10]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.
   3. Measurement:
      a. The Walton-Beckett graticule standardizes the area observed [14,18,19].
      b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [4,14].
      c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm^2 filter area (a total of 78.5 fibers counted in 100 fields, each with field area = .00785 mm^2.) Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [20]. The recommended loadings should yield intracounter S_r in the range of 0.10 to 0.17 [21,22,23].

B. Interlaboratory comparability:
   An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [9]. The relative standard deviations (S_r) varied with sample type and laboratory. The ranges were:

	Intralaboratory S_r	Interlaboratory S_r	Overall S_r
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)**	0.11 to 0.29	0.20 to 0.35	0.25

* Under AIA rules, only fibers having a diameter less than 3 μm are counted and fibers attached to particles larger than 3 μm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.
** See Appendix C.

A NIOSH study conducted using field samples of asbestos gave intralaboratory S_r in the range 0.17 to 0.25 and an interlaboratory S_r of 0.45 [21]. This agrees well with other recent studies [9,14,16].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory S_r. (NOTE: The following discussion does not include bias estimates and should not be taken to indicated that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give an S_r that depends on the number, N, of fibers counted:

$$S_r = 1/(N)^{1/2} \quad (1)$$

Thus S_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual S_r found in a number of studies is greater than these theoretical numbers [17,19,20,21].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [15] found this subjective component of intralaboratory S_r to be approximately 0.2 and estimated the overall S_r by the term:

$$\frac{[N + (0.2 \cdot N)^2]^{1/2}}{N} \quad (2)$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were +2 S_r and - 1.5 S_r. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association also found that the variability had both a constant component and one that depended on the fiber count [14]. These results gave a subjective interlaboratory component of S_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [21]. This value falls slightly above the range of S_r (0.25 to 0.42 for 1984-85) found for 80 reference laboratories in the NIOSH PAT program for laboratory-generated samples [17].

A number of factors influence S_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. It is hoped that the laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the S_r.

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [16].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm^2 counting field area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the S_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory S_r is smaller, then it is more correct to use that smaller S_r. However, the estimated S_r of 0.45 is to be used in the absence of such information. Note also that it has been found that S_r can be higher for certain types of samples, such as asbestos cement [9].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e., 0.16 + 2.13 x 0.16 = 0.5.

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and -49% as the upper and lower confidence values of the mean for a 100-fiber count.

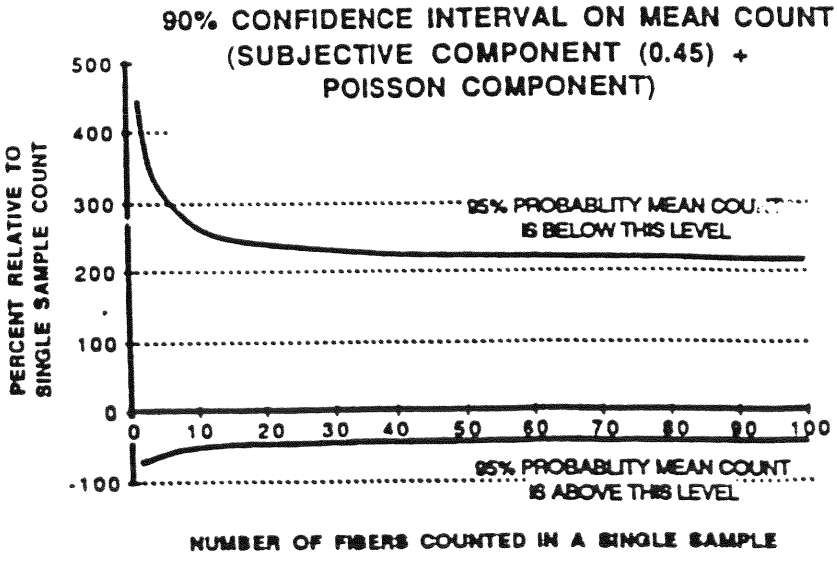

Figure 1. Interlaboratory Precision of Fiber Counts

The curves in Figures 1 are defined by the following equations:

$$UCL = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25 S_r^2) X^2]^{1/2}}{2(1 - 2.25 S_r^2)} \quad (3)$$

$$LCL = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4 S_r^2) X^2]^{1/2}}{2(1 - 4 S_r^2)} \quad (4)$$

where S_r = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory S_r when approximately 100 fibers are counted.
X = total fibers counted on sample
LCL = lower 95% confidence limit.
UCL = upper 95% confidence limit.
Note that the range between these two limits represents 90% of the total range.

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METHOD WRITTEN BY:

Paul A. Baron, Ph.D., NIOSH/DPSE.

APPENDIX A: CALIBRATION OF THE WALTON-BECKETT GRATICULE:

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (D) 100 μm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40 to 45X phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_o (μm), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_o} \times D. \qquad (5)$$

Example: If L_o = 112 μm, L_a = 4.5 mm and D = 100 μm, then d_c = 4.02 mm.

8. Check the field diameter, D (acceptable range 100 μm ± 2 μm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range 0.00754 mm^2 to 0.00817 mm^2).

APPENDIX B: COMPARISON OF COUNTING RULES:

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. The rules will be discussed as they apply to the labeled objects in the figure.

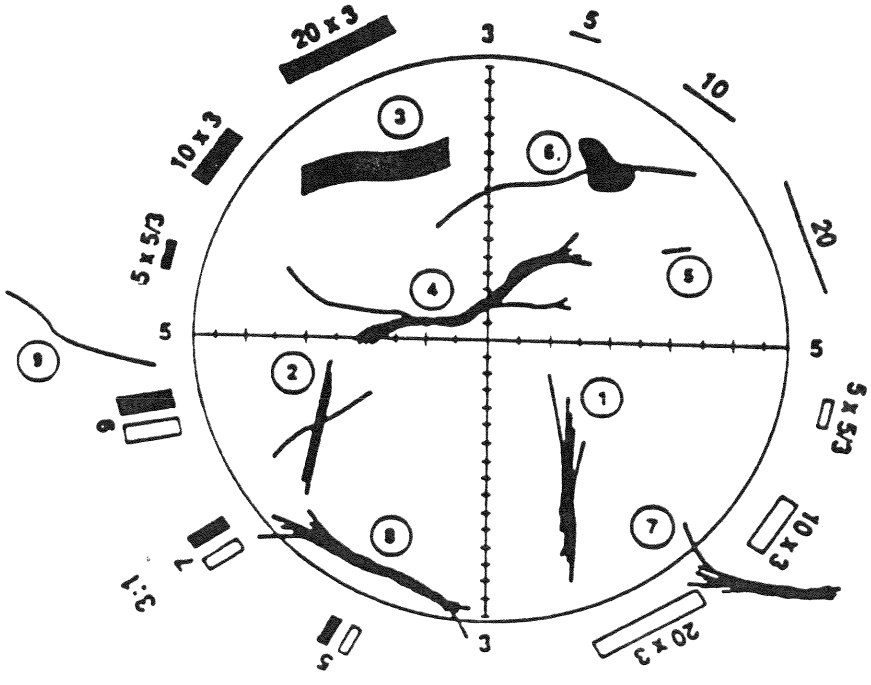

Figure 2. Walton-Beckett graticule with fibers.

These rules are sometimes referred to as the "A" rules.

FIBER COUNT

Object	Count	DISCUSSION
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fiber	If fibers meeting the length and aspect ratio criteria (length >5 μm and length-to-width ratio >3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (>3 μm), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is ≤5 μm long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once. count
9	Do not count	Ignore fibers that lie outside the graticule boundary.

APPENDIX C. ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the "B" rules given below (from NIOSH Method 7400, Revision #2, dated 8/15/87), the World Health Organization reference method for man-made mineral fiber [24], and the NIOSH fibrous glass criteria document method [25]. The upper diameter limit in these methods prevents measurements of non-thoracic fibers. It is important to note that the aspect ratio limits included in these methods vary. NIOSH recommends the use of the 3:1 aspect ratio in counting fibers.

It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

"B" COUNTING RULES:

1. Count only ends of fibers. Each fiber must be longer than 5 μm and less than 3 μm diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 μm in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

APPENDIX D. EQUIVALENT LIMITS OF DETECTION AND QUANTITATION

fiber density on filter*		fiber concentration in air, f/cc	
fibers per 100 fields	fibers/mm^2	400-L air sample	1000-L air sample
200	255	0.25	0.10
100	127	0.125	0.05
LOQ.....80.....	.....102.....	.....0.10.....	.....0.04
50	64	0.0625	0.025
25	32	0.03	0.0125
20	25	0.025	0.010
10	12.7	0.0125	0.005
8	10.2	0.010	0.004
LOD.....5.5.....	.....7.....	.....0.00675.....	.....0.0027

* Assumes 385 mm^2 effective filter collection area, and field area = 0.00785 mm^2, for relatively "clean" (little particulate aside from fibers) filters.

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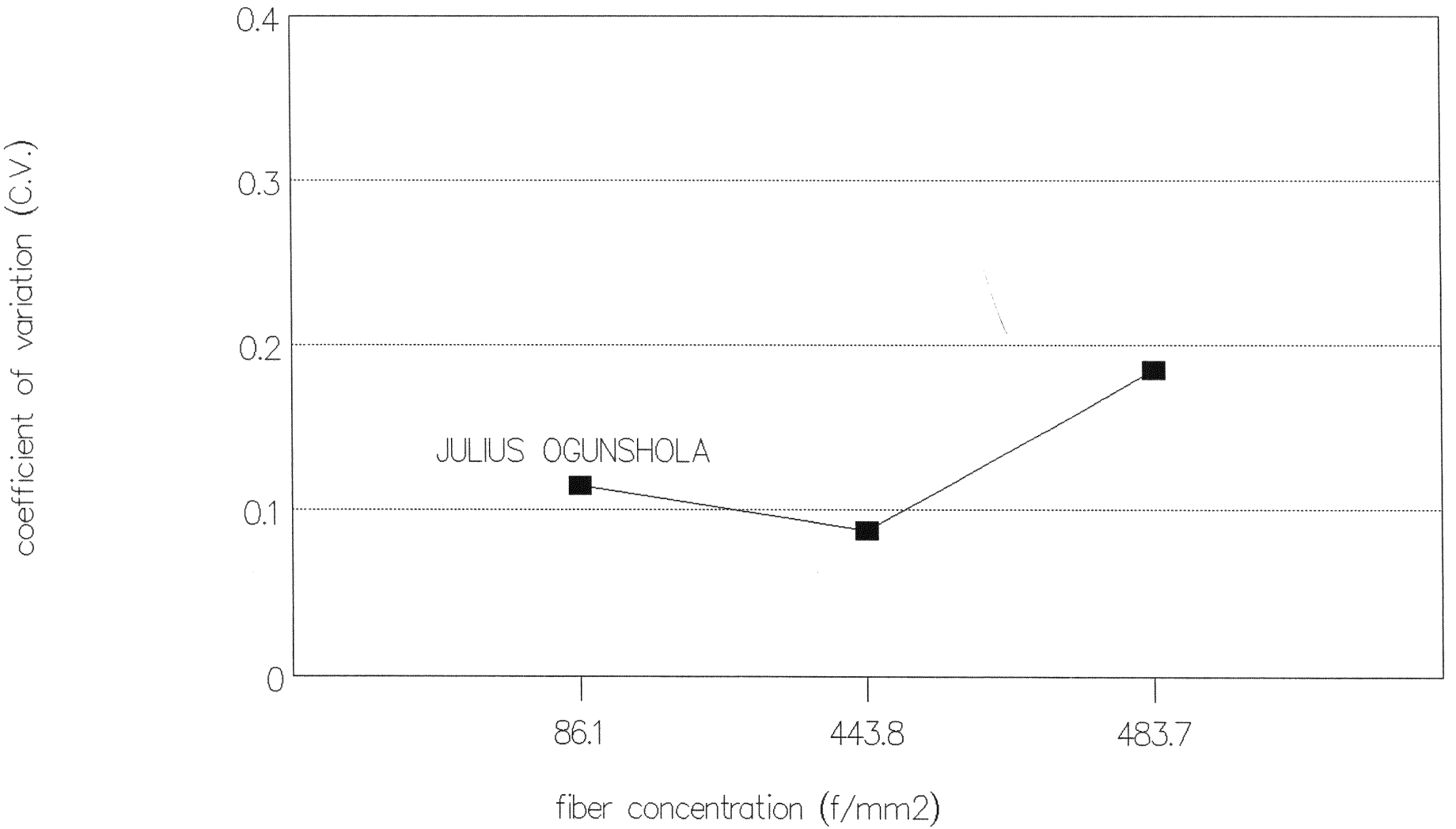
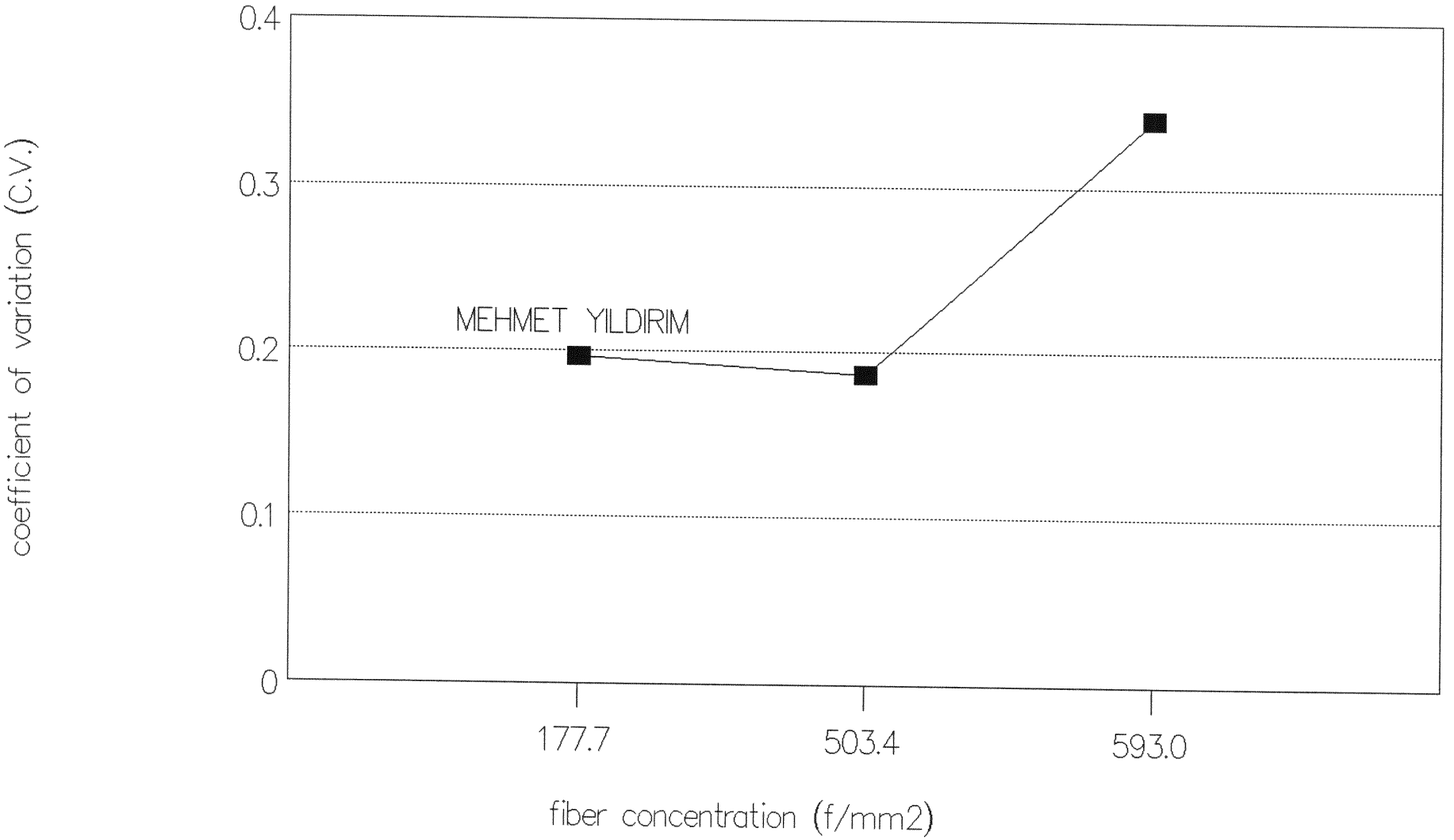
PRECISION COUNT FOR VARIOUS FIBER LOADS



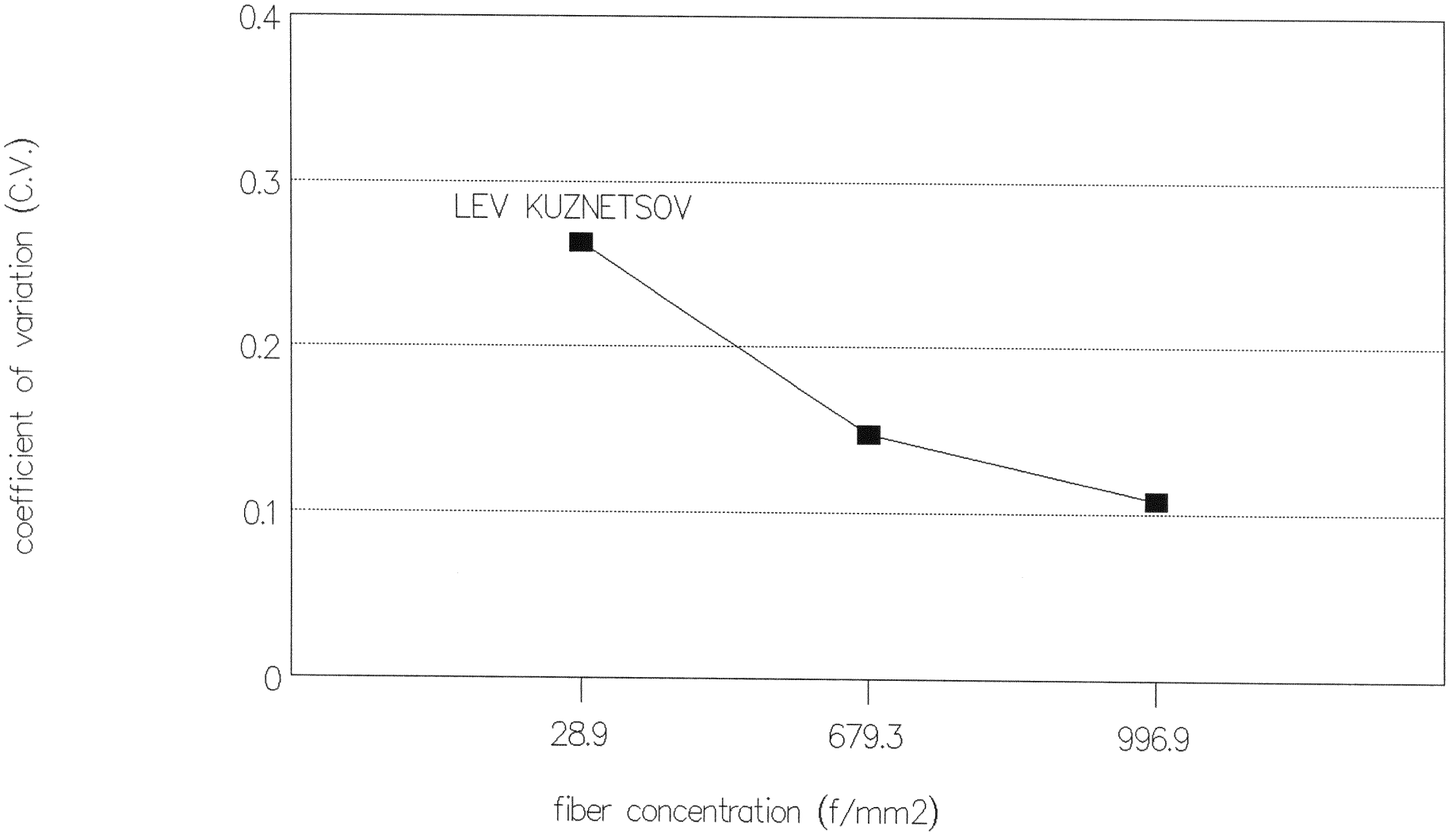
ANALYTICAL ENVIRONMENTAL SERVICES, INC.

PRECISION COUNT FOR VARIOUS FIBER LOADS



ANALYTICAL ENVIRONMENTAL SERVICES, INC.

PRECISION COUNT FOR VARIOUS FIBER LOADS



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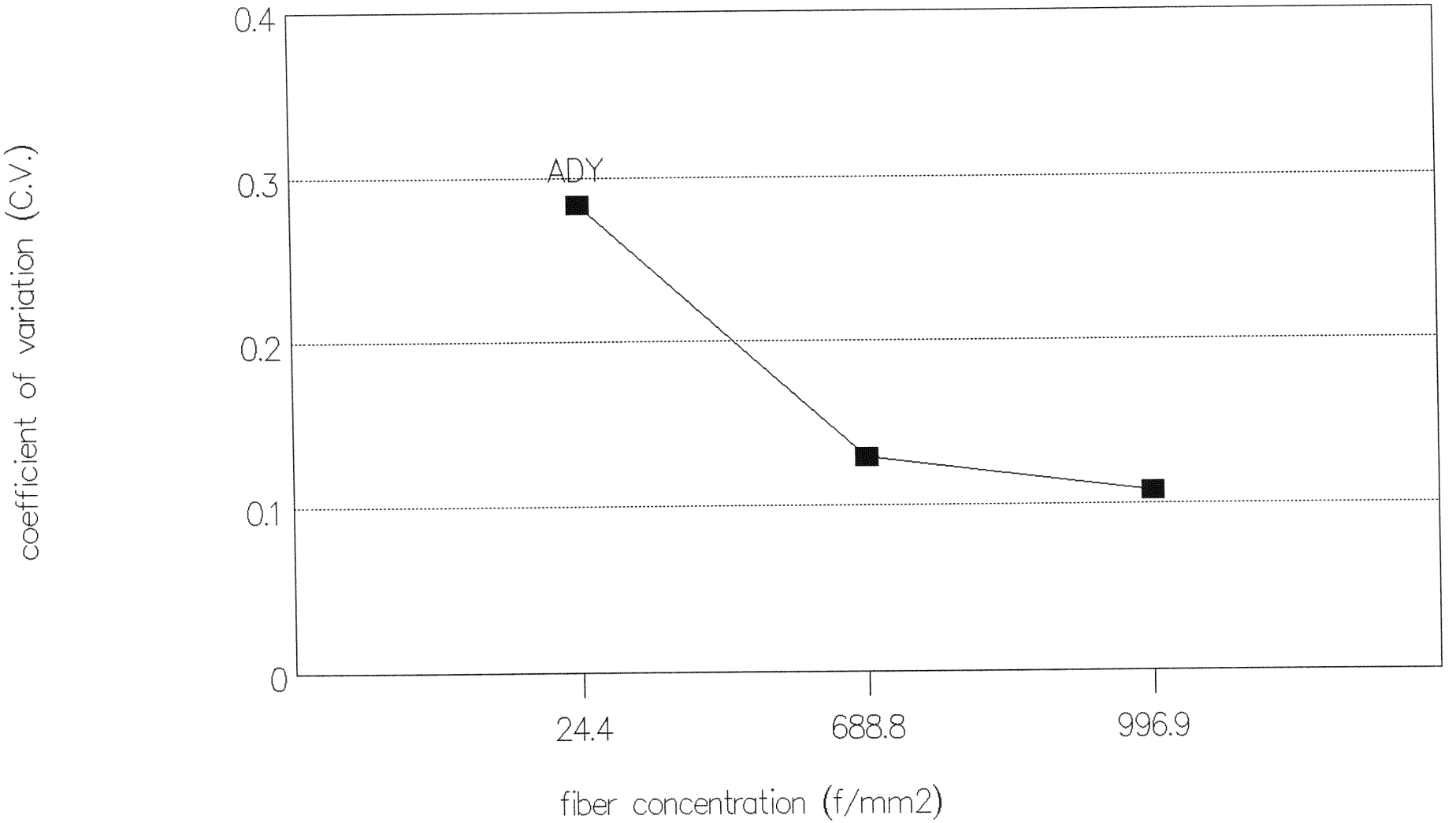
PRECISION COUNT FOR VARIOUS FIBER LOADS



APPENDIX A-2

Standard Operating Procedures
(in laboratory Quality Assurance Manual CD)

SOP #	Title
IA-13002	Standard Operating Procedure for the Determination of Metals in Water, Soils and Waste by ICP by EPA SW-846 Method 6010C and Prep Methods 3010B/3050/SM3030C
IA-13041	Standard Operating Procedure for Determination of Metals in Water, Soils, Wastes, and Surface Wipes by ICP/MS by SW-846 Method 6020A/3005A/3050B/NIOSH7300
IA-13037	Standard Operating Procedure for Determination of Mercury in Water by Manual Cold Vapor Method by EPA SW-846 Method 7470A
IA-13033	Standard Operating Procedure for Mercury in Solid or Semi-Solid Waste (Manual Cold Vapor Technique)(by Method 7471B)
OA-11007	Standard Operating Procedure for 1,2-EDB and 1,2-DCBP by EPA SW-846 Method 8011
OA-11002	Standard Operating Procedure for Diesel Range Organics by EPA SW-846 Method 8015C
OA-11004	Standard Operating Procedure for Chlorinated Herbicides by GC by EPA SW-846 Method 8151A
OA-11010	Standard Operating Procedure for Volatile Organic Compounds by EPA SW-846 Method 8260B/5030/5035
OA-11011	Standard Operating Procedure for Semivolatile Organics by SW-846 GC/MS Method 8270D Prep Methods 3510C/3535A/3540C/3550C/3580A

APPROVAL OF ATTACHED DOCUMENT FOR IMPLEMENTATION

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ORIGINAL SIGNED DOCUMENT RESIDES IN AES QA OFFICE

DOCUMENT TITLE: DETERMINATION OF METALS IN WATER, SOILS AND WASTES BY ICPBY EPA SW-846 METHOD 6010C AND PREP METHODS 3010A/3050B/SM3030C

DOCUMENT CONTROL NUMBER: Rev. 9

DOCUMENT DISTRIBUTION NUMBER: IA-13002

ELECTRONIC DOCUMENT LOCATION
AES Portal Server: http://Procedures/Standard Operating Procedures

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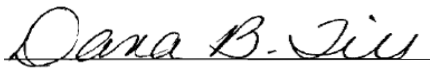
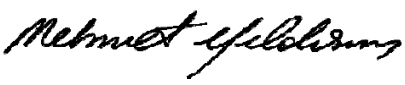
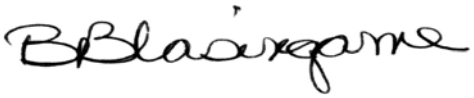
Technical Director:  Date: 9/29/09

Laboratory Manager:  Date: 10/9/09

Quality Assurance Manager:  Date: 10/9/09

Department Supervisor:  Date: 10/7/09

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF METALS IN WATER, SOILS AND WASTES BY ICP BY EPA SW-846 METHOD 6010C AND PREP METHODS 3010B/3050/SM3030C

TABLE OF CONTENTS

Page

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-atomic emission spectrometry determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 2-1. All matrices, excluding filtered groundwater samples but including groundwater aqueous samples, TCLP and EP extracts, and industrial and organic wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion. Results are analytically reported as "dissolved metals". Samples which are not digested must either use an internal standard or be matrix matched with the standards. Refer to the procedure section for appropriate digestion procedures.

1.2 Table 2-1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 7-1 lists the recommended analytical wavelengths and estimated instrumental detection limits for the elements in clean aqueous matrices. Laboratory determined detection limits are listed in the QA Manual, Section 5. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements and matrices other than those listed in Table 7-1 may be analyzed by this method if performance at the concentration levels of interest is demonstrated.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be acidified, solubilized, or digested using appropriate sample preparation procedures as mandated by the EPA.

2.2 The analysis described in this procedure involves multi-elemental determinations by ICP-OES using simultaneous and/or sequential instrumentation. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element specific spectra are produced by radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the line spectra are monitored at specific wavelengths by a photosensitive device. A background correction technique is required to compensate for the variable background contribution to the determination of the analytes.

2.3 The following analytes can be analyzed by this method:

Table 2-1
Metallic Analytes Analyzed by ICP

Aluminum	Antimony	Arsenic
Barium	Beryllium	Boron
Cadmium	Calcium	Chromium
Cobalt	Copper	Iron
Lead	Lithium	Magnesium
Manganese	Phosphorus	Molybdenum
Nickel	Silica	Potassium
Selenium	Strontium	Silver
Sodium	Sulfur	Titanium
Thallium	Tin	Vanadium
Zinc		

3.0 INTERFERENCES

3.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

3.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by the measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectra adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

3.1.2 To determine the appropriate location for an off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for the background correction must be either free of off-line interelement spectral interference or a computer routine must be used for the automatic correction of all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements,

and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentrations, a more appropriate test would be to use a concentration near the upper analytical range limit.

3.1.3 Spectral overlaps may be avoided by using an alternate wavelength, or can be compensated for by using equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interfering effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 3-1. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

3.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 3-1, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 3-1. The interference effects must be evaluated for each individual instrument since the intensities will vary.

3.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending on the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples might contain uncommon elements that could contribute spectral interferences.

3.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each

instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height, and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences (Table 3-1) as well as any other suspected interference that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for the automatic correction of all analyses.

3.1.7 If the correction routine is operating properly, the determined apparent analyte concentration(s) from the analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and dividing by 10. If, after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

3.1.8 When interelement corrections are applied, their accuracy should be verified daily by analyzing spectral interference check solutions. The laboratory performs interelement corrections at least every 6 months and whenever Interferent Check Standards (ICS) do not meet recovery requirements.

3.1.9 The laboratory analyzes an Interference Check Solution(s), which contains similar concentrations of the major components of the samples (> 10 mg/L), on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is > 20% of the analyte concentration, the analyte must be determined using either (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) an alternative wavelength, or (3) another documented test procedure.

3.2 Physical interferences are effects associated with the sample nebulization and transport processes.

3.2.1 Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump, by using an internal standard, or by using a high solids nebulizer.

3.2.2 Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer, or diluting the sample. In addition, it

has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance; this may be accomplished with the use of mass flow controllers. The test described in Section 8 will help determine if a physical interference is present.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, etc), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

3.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample.

3.4.1 Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples.

3.4.2 The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range.

3.4.3 The aspiration time for this sample should be the same as the aspiration time for a normal sample followed by the analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two times the method detection limit should be recorded.

3.4.4 Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

3.5 Table 3-1 indicates analyte concentration equivalents that arise from interferents at the 100 mg/L level. The spaces in Table 3-1 indicate that no measurable interferences were observed even at higher interferent concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

Table 3-1
ANALYTE CONCENTRATION EQUIVALENTS (Mg/L) ARISING FROM INTERFERENTS AT THE 100 (Mg/L) LEVEL

Analyte	**Wavelength nm**	**Interferent –**									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.214	-	-	-	-	-	-	0.21	-	0.25	1.4
Antimony	206.833	0.47	-	2.9	-	0.08	-	-	-	-	0.45
Arsenic	193.696	1.3	-	0.44	-	-	-	-	-	-	1.1
Barium	455.403	-	-	-	-	-	-	-	-	-	-
Beryllium	313.042	-	-	-	-	-	-	-	-	0.04	0.05
Boron	249.773	.04	-	-	-	0.32	-	-	-	-	-
Cadmium	226.502	-	-	-	-	0.03	-	-	0.02	-	-
Calcium	317.933	-	-	0.08	-	0.01	0.01	0.04	-	0.03	0.03
Chromium	267.716	-	-	-	-	0.003	-	0.04	-	-	0.04
Cobalt	228.616	-	-	0.03	-	0.005	-	-	0.03	0.15	-
Copper	324.754	-	-	-	-	0.003	-	-	-	0.05	0.02
Iron	259.940	-	-	-	-	-	-	0.12	-	-	-
Lead	220.353	0.17	-	-	-	-	-	-	-	-	-
Magnesium	279.079	-	0.02	0.11	-	0.13	-	0.07	-	0.07	0.12
Manganese	257.610	0.005	-	0.01	-	0.002	0.002	-	-	-	-
Molybdenum	202.030	0.05	-	-	-	0.03	-	-	-	-	-
Nickel	231.604	-	-	-	-	-	-	-	-	-	-
Analyte	**Wavelength nm**	**Interferent –**									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Selenium	196.026	0.23	-	-	-	0.09	-	-	-	-	-
Silicon	288.158	-	-	0.07	-	-	-	-	-	-	0.01
Sodium	588.995	-	-	-	-	-	-	-	-	0.08	-
Thallium	190.864	0.30	-	-	-	-	-	-	-	-	-
Vanadium	292.402	-	-	0.05	-	0.005	-	-	-	0.02	-
Zinc	213.856	-	-	-	0.14	-	-	-	0.29	-	-

Blank spaces indicate that no interference was observed even when interferences were introduced at the following levels:

Al - 1000 mg/L Mg - 1000 mg/L Ca - 1000 mg/L Mn - 200 mg/L Cr - 200 mg/L
Ti - 200 mg/L Cu - 200 mg/L V - 200 mg/L Fe - 1000 mg/L

Interferences will be affected by background choice and other interferences that may be present.

Table 3-2
Analyte Elemental Concentrations Used For Interference Measurements in Table 3-1

Analytes	(mg/L)		Analytes	(mg/L)
Aluminum	10		Manganese	1
Arsenic	10		Molybdenum	10
Boron	10		Sodium	10
Barium	1		Nickel	10
Beryllium	1		Lead	10
Calcium	1		Antimony	10
Cadmium	10		Selenium	10
Cobalt	1		Silicon	1
Chromium	1		Thallium	10
Copper	1		Vanadium	1
Iron	1		Zinc	10
Magnesium	1			

3.6 Other sources of interference include sample contamination. Dust in the laboratory environment, impurities in reagents, and impurities on laboratory apparatus that the sample comes in contact with are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption

3.6.1 Aqua regia (HNO3+HCl) may be used to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of acid.

3.6.2 If it can be documented through an active analytical quality control program using spiked samples and reagent blanks that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

4.1 For the determination of trace elements, contamination and loss are of prime concern. In this regard, the collection and treatment of the sample prior to analysis require particular attention. Laboratory glassware, including the sample bottle (whether polyethylene, polypropylene, or FEP-fluorocarbon), should be thoroughly washed with detergent and tap water and rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap water, and finally deionized water.

4.2 Before collection of the sample, a decision must be made regarding the type of metal analysis that is desired; that is dissolved, suspended, or total, so the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc. are to be performed at the time the sample is collected, or as soon as possible thereafter.

4.2.1 For the determination of dissolved elements, the sample must be filtered through 0.45µm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus is recommended to avoid possible contamination). Use the first 50 – 100 ml of sample to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH ≤ 2. Normally, 3 ml of (1+1) acid per liter should be sufficient to preserve the sample.

4.2.2 For the determination of suspended elements, a measured volume of unpreserved sample must be filtered through a 0.45µm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservation is required.

4.2.3 For the determination of total elements, the sample is preserved with (1+1) HNO_3 to a pH ≤ 2. Normally, 3 ml of (1+1) acid per liter should be sufficient to preserve the sample.

4.2.4 North Carolina groundwater samples requiring preparation by SM3030C must be prepared within 72 hours of collection.

4.2.5 All samples other than those described in 4.2.4 must be analyzed within 180 days of sampling.

5.0 REAGENTS AND STANDARDS

5.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Re-distilled acids are acceptable.

5.1.1 Hydrochloric Acid, concentrated (sp.gr. 1.19).

5.1.2 Hydrochloric Acid, (1:1): Add 500mL concentrated HCl to 400mL deionized water and dilute to 1 liter.

5.1.3 Nitric Acid, concentrated (sp.gr. 1.41)

5.1.4 Nitric Acid, (1:1): Add 500mL concentrated HNO_3 to 400mL deionized water and dilute to 1 liter.

5.2 Hydrogen Peroxide, 30%. Purchase commercially prepared solution.

5.3 Deionized water, 18 megaohm or greater.

5.4 Single element standard solutions. Purchased from the vendor as certified solution at concentration of 1000 mg/L or other suitable concentration.

5.5 Primary Multielement Standard 1(AESI-CAL-1). Purchased as certified solution from Inorganic Ventures. Two independently prepared lot numbers are required. Concentrations are as follows:

Table 5-1
Primary Multielement Std 1 Concentrations

Concentration (mg/L)	Metal Ion
10	Ag
100	As,Ba,Be,Cd,Co,Cr,Cu,Mn,Ni,Pb,Se,Tl,V,Zn
1000	Al, Ca, Fe, K, Mg, Na

5.6 Primary Multielement Standard 2(VAR-CAL-1). Purchased as certified solution from Inorganic Ventures. Two independently prepared lot numbers are required. Concentrations are as follows:

Table 5-2
Primary Multielement Std 2 Concentrations

Concentration (mg/L)	Metal Ion
100	Mo, Sb, Sn, Ti

5.7 CRI Standard 1(AESI-CAL-2). Purchased as certified solution from Inorganic Ventures. Concentrations are as follows:

Table 5-3
Primary Multielement Std 1 Concentrations

Concentration (mg/L)	Metal Ion
2	Sb
5	Mo, Sn, Ti

5.8 CRI Standard 2(AESI-CAL-3). Purchased as certified solution from Inorganic Ventures. Concentrations are as follows:

Table 5-4
Primary Multielement Std 1 Concentrations

Concentration (mg/L)	Metal Ion
0.5	Ag, Be, Cd, Mn
1	Ba, Co, Cr, Cu, Pb, V
2	Ni, Se, Tl, Zn
5	As
10	Ca, Fe, Mg
20	Al
50	K
100	Na

5.9 Interferent Check Standard (ICS) (ICL-500-6). Purchased as certified solution from Environmental Express. Concentrations are as follows:

Table 5-5
Primary Multielement Std 1 Concentrations

Concentration (mg/L)	Metal Ion
2000	Fe
5000	Al, Ca, Mg

5.10 Initial calibration standard 1 (ICAL1). Prepare by adding 0.5mL each of the IV primary multi-element standards, 4mL of concentrated nitric acid, and 5mL of concentrated hydrochloric acid to a 100mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-6
ICAL1 Concentrations

Concentration (mg/L)	Metal Ion
5	Al, Ca, Fe, K, Mg, Na
0.5	As, B, Ba, Be,Cd,Co,Cr,Cu,Mn,Mo,Ni, Pb, Se,Sb,Sn,Sr,Ti Tl,V,Zn
0.05	Ag

5.11 Initial calibration standard 2 (ICAL2), continuing calibration verification standard (CCV), and check standard (CKSTD). Prepare by adding 5.0mL of the IV primary source multi-element standards, 20mL of concentrated nitric acid, and 25mL of concentrated hydrochloric acid to a 500mL volumetric flask, and diluting to volume with deionized water. The concentrations of the metals are as follows:

Table 5-7
ICAL2, CKSTD, and CCV Standards Concentrations

Concentration (mg/L)	Metal Ion
10	Al, Ca, Fe, K, Mg, Na
1	As, B, Ba, Be,Cd,Co,Cr,Cu,Mn,Mo,Ni, Pb, Se,Sb,Sn, Sr,Tl,Ti,V,Zn
0.1	Ag

5.12 Initial calibration verification standard (ICV). Prepare by adding 5.0mL each of the IV multi-element metals standards, 40mL of concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1000mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-8
ICV Concentrations

Concentration (mg/L)	Metal Ion
5	Al, Ca, Fe, K, Mg, Na
0.5	As, B, Ba, Be,Cd,Co, Cr, Cu,Mn,Mo,Ni, Pb,Se,Sb,Sn,Sr,Ti Tl, V,Zn
0.05	Ag

5.13 Working interference check standard (ICSA). Prepare by adding 50mL of the ICS standard, 40mL of concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1L volumetric flask and diluting to volume with deionized water. The

concentrations of metals in this standard are 250 mg/L for aluminum, calcium, and magnesium, and 100 mg/L for iron.

5.14 Secondary working interference check standard (ICSAB). Prepare by adding 50mL of the ICS standard, 5mL each of the IV multi-element standards, 40mLof concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1000mL volumetric flask and diluting to volume with deionized water. The concentrations of metals in the standard are as follows:

Table 5-9
ICSAB Standard Concentrations

Concentration (mg/l)	Metal Ion
105	Fe
255	Al, Ca, Mg
5.0	K, Na
0.5	As, B, Ba, Be, Cd,Co,Cr,Cu, Mn,Mo, Ni, Pb,Se,Sb,Sn,Sr,Ti Tl,V,Zn
0.05	Ag

5.15 CRI Standards. The CRI is prepared by adding 1mL each of the IV CRI standards, 4mL of concentrated nitric acid and 5mL of concentrated hydrochloric acid to a 100mL volumetric flask and diluting to volume with deionized water. The resulting metal concentrations are as follows:

Table 5-10
CRI Standard Concentrations

CRI Concentration (mg/L)	Metal Ion
0.005	Ag, Be, Cd, Mn
0.01	Ba, Co, Cr, Cu, Pb, V
0.02	Li, Ni, Sb, Se, Tl, Zn
0.05	As, B, Mo, P, S, Sn, Sr, Ti
0.1	Ca, Fe, Mg, Si
0.2	Al
0.5	K
1.0	Na

5.16 Standards for elements not present in the Multielement mixes are prepared by diluting single element stock standards to the desired concentrations per project requirements.

5.17 Two types of blanks are required for the analysis. The calibration blank is used to establish the analytical curve, while the reagent blank (method blank) is used to correct for possible contamination resulting from varying amounts of the acids used in sample processing.

5.17.1 The calibration blank is prepared by diluting 40 ml concentrated HNO_3 and 50 ml concentrated HCl to 1000 ml using deionized water.

5.17.2 The reagent blank (method blank) must contain all the regents, in the same volumes, used in processing the samples. The reagent blank (method blank)

must be carried through the complete procedure and contain the same acid concentration as the sample solution used for analysis.

5.18 Vendor List. The standards used for this method are purchased using the catalog numbers and vendors indicated below.

Table 5-11
ICP Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
Primary Multielement Std 1	Inorganic Ventures	Various	AESI-CAL-1
Primary Multielement Std 2	Inorganic Ventures	Various	VAR-CAL-1
Secondary Multielement Std 1	Inorganic Ventures	Various	AESI-CAL-1 (Separate Lot # from primary)
Secondary Multielement Std 2	Inorganic Ventures	Various	VAR-CAL-1 (Separate Lot # from primary)
CRI Std 1	Inorganic Ventures	Various	AESI-CAL-2
CRI Std 2	Inorganic Ventures	Various	AESI-CAL-3
Interferent Check Std (ICS)	Environmental Express	Varied	ICL 500-6
Primary Boron Std	Environmental Express*	1000 µg/mL	HP10007-4
Secondary Boron Std	VHG Labs*	1000 µg/mL	PBW-100
Primary Lithium Std	VHG	1000ug/ml	PLIN-100
Secondary Lithium Std	Environmental Express*	1000 µg/mL	HP100029-1100
Primary Phosphorus Std	Ultra Scientific*	1000 µg/mL	ICP-015
Secondary Phosphorus Std	Environmental Express*	1000 µg/mL	HP100039-1
Primary Silicon(Silica-Si) Std	Assurance Spex*	1000 µg/mL	PLS19-2Y
Secondary Silicon(Silica-Si) Std	Fisher Scientific*	1000 µg/mL	SS465-100
Primary Strontium Std	Ultra Scientific*	1000 µg/mL	ICP-038
Secondary Strontium Std	Ricca*	1000 µg/mL	8100-4
Primary Sulfur Std	Environmental Express*	1000 µg/mL	HP100054-5
Secondary Sulfur Std	EMD*	1000 µg/mL	1.70355.0100
Hydrochloric Acid	EM Science*	Concentrated	HX0603-3
Nitric Acid	VWR Scientific*	Concentrated	VW4815-6
Hydrogen Peroxide	VWR Scientific*	30% Solution	VW3690-5

***Single element standards and acids may be purchased from alternate sources as needed.**

6.0 APPARATUS AND MATERIALS

6.1 Inductively Coupled Plasma-Atomic Emission Spectrometer. Perkin Elmer Optima 4300 DV and Varian Vista Pro instrument systems.

6.2 Argon gas supply. Analytical grade or better.

6.3 Block digester

6.4 Plastic digestion vessels - 70-ml disposable with graduations.

6.5 General Glassware. Pipettes, volumetric flasks, funnels, etc.

7.0 PROCEDURE

7.1 Preparation of digestion log form and digestion log in LIMS.

7.1.1 Each day the section supervisor prepares a work log. The log lists samples included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN DIGESTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received with a “rush” status will have a chain of custody delivered to the section supervisor by the project manager.

7.1.1.2 Prepare a written digestion log using the metals digestion logbook that is kept in the digestion area of the laboratory. The following entries must be made in the log:

7.1.1.2.1 Date and time the batch is opened or the date and time the sample(s) is placed on the hot plate.

7.1.1.2.2 All samples included in the digestion batch.

7.1.1.2.3 Volume or weight of samples digested.

7.1.1.2.4 Date and time the digestion is completed.

7.1.1.2.5 Digestion procedure employed.

7.1.1.2.6 The initials of the digestion analyst(s).

7.1.1.2.7 Laboratory number of all reagents used, including spiking standard and acids.

7.1.1.2.8 Volume of spiking standard and acids used.

7.1.1.2.9 Final volume of all digestates.

7.1.1.2.10 Date and time the batch is closed.

7.1.1.2.11 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.1.1.3.1 Open a Prep Batch in LIMS by double clicking the "Prep" box.

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS.

7.1.1.3.3 Select the Prep Code "3010A" or other appropriate digestion code from the pull down list. The LIMS will automatically assign a MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCS and enter the information.

7.1.1.3.4 Enter the technician's name from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this method. Select the samples to be included in the batch for desired prep method.

7.1.1.3.6 The samples can be individually selected by highlighting the sample and clicking the "$\rightarrow$" arrow or all samples can be selected by clicking the "$\Rightarrow$" arrow. Then select "OK".

7.1.1.3.7 Samples that have been selected for re-analysis can be manually added to the sample list following the procedure outlined in section 7.1.1.3.3.

7.1.1.3.8 "Save" the batch by clicking on a previous batch number on the list and then return to the newly created batch.

7.1.1.3.9 Close the batch by entering the date and time.

7.1.2 Table 7-1 indicates the type of samples that comprise an analytical batch. Note: NELAC requirements specify that the maximum number of client samples in a prep batch can not exceed 20. Further, a prep batch can not be left "open", i.e. samples added for a period that exceeds 24 hours.

Table 7-1
Samples in a NELAC Batch
Method Blank (MB)
LCS (and LCSD if no MSD)
MS and MSD (If supplied by client)
Dilution Test Sample
Post Digestion Spike Sample
Client Samples (up to 20 per batch)

This method requires the analysis of additional samples not required by NELAC. See the information in Section 7.3, Run Sequence.

7.2 Sample preparation

7.2.1 Sample preparation for the Determination of Dissolved Metals is described in detail in Table 7-9.

7.2.2 Sample preparation for the Determination of Total Recoverable Metals in Water Samples by Method 3010A is describes in detail in Table 7-5.

7.2.3 Sample preparation for the Determination of Total Metals in Soil Samples by Method 3050B is described in detail Table 7-6.

7.2.4 Sample preparation for the Determination of Total Metals in Waste Samples by Method 3050B is described in detail Table 7-7.

7.2.5 Sample preparation for the Determination of Acid Extractable Metals in Water Samples by Method SM3030C is described in detail Table 7-8.

7.2.6 Sample preparation for the determination of metals on wipes is described in Table 7-10.

7.3 Instrument Operation for the Perkin Elmer Optima 4300.

7.3.1 Software Start Up

7.3.1.1 Click on Win Lab32 from the desktop

7.3.1.2 Click on Workspace from the menu list on top to open workspace window

7.3.1.3 Click on [CURRENT METHOD] and click on open

7.3.1.4 Click on [CURRENT METHOD] and open to open current method

7.3.1.5 Click on analysis and scroll down to auto sampler and click on A/S probe Up/Down to move the probe down.

7.3.1.6 Put sample capillary tubing in the rinse bottle filled with DI water

7.3.1.7 Click on Plasma and On to ignite plasma.

7.3.1.8 Wait 30 minutes for instrument to warm up

7.3.1.9 Make calibration standards every morning before analysis
7.3.1.10 Dump standards from previous day and rinse out sample vessels with DI water or get new ones
7.3.1.11 Pour calibration standards into clean dry sample vessels
7.3.1.12 Load standards according to the following sequence
1. Blank
2. ICAl 1
3. ICAL 2 (Check standard)
4. ICV
5. LLICV (CRI)
6. ICSA
7. ICSAB

7.3.1.13 Go to file – new and click on sample info file
7.3.1.14 Type in file name yymmdd followed by A, B, C…..
7.3.1.15 Type in samples under sample ID and sample locations under A/S location starting with location 9
7.3.1.16 Go to file- Save file as and then to Sample Info file and enter file name yymmdd followed by A, B, C….
7.3.1.17 Click on rebuild list on workspace

7.3.2 SAVING RAW DATA
7.3.2.1 Click on Set Up at the bottom left corner of the workspace window
7.3.2.2 Click on open under result data set name
7.3.2.3 Type in date yymmdd followed by A, B, C…. Make sure result date and sample info file date are the same
7.3.2.4 Make sure Save Data, Use Sample Info and Print Log During Analysis, are checked.
7.3.2.5 Click on analyze to go back to workspace
7.3.2.6 Click on file from the menu list and scroll down to print setup and click on it
7.3.2.7 Make sure it is set to pdf Factory Pro. Raw data will save directly to pdf file which will open up once analysis is started
7.3.2.8 Click on pdf file and then click on save
7.3.2.9 Enter file name yymmdd followed by A, B, C… _Raw_Data and save on desktop

7.3.3 Na Bullet Test
7.3.3.1 Run Na bullet test each time the torch is changed
7.3.3.2 Place probe in 1000mg/L solution of Na
7.3.3.3 Examine plasma through viewing window in the sample compartment door. A yellow-orange bullet should be visible in the center of discharge, and should extend from the base of the discharge to about 2-3 mm past the RF coil.
7.3.3.4 If bullet height is too low, increase nebulizer flow in the method editor or plasma control window and if it is too high decrease nebulizer flow.
7.3.3.5 If no bullet, check that sample is being pumped to the nebulizer. Make sure that pump lever is engaged and that pump tubes are connected appropriately.

7.3.4 TORCH ALIGNMENT

7.3.4.1 Torch alignment is done each time the torch is removed.

7.3.4.2 In the tools menu, click on spectrometer control. The spectrometer control window will appear.

7.3.4.3 Select Axial view in the Spectrometer control window.

7.3.4.4 Put probe in 1ppm Manganese solution located next to autosampler.

7.3.4.5 Click on align view in the spectrometer control window to open align view dialogue.

7.3.4.6 Select Axial view. The Y viewing position has to be set to 15.0 mm and the X viewing position set to 0.0 mm.

7.3.4.7 Select Radial view and put probe in 10ppm Mn solution located next to autosampler.

7.3.4.8 Click on align view. The X viewing position should read close to zero but no greater than +/- 1.

7.3.4.9 Close spectrometer control window when done. If alignment is not within specified limits, repeat process.

7.3.5 MERCURY REALIGN

7.3.5.1 Hg realign is completed when needed.

7.3.5.2 Click on spectrometer control in the tools menu.

7.3.5.3 Select Axial view in the Spectrometer control window.

7.3.5.4 Click on Hg realign.

7.3.5.5 Close spectrometer control window when done.

7.3.5.6 Probe stays in rinse during Hg realign.

7.3.5.7 The peak offset on the mercury realign usually reads about -0.002 and the drift reads -0.001.

7.3.6 STARTING ANALYSIS

7.3.6.1 Put sample capillary tubing in the rinse bottle filled with 5%HCL/4%HNO3.

7.3.6.2 Click on analyze all to analyze standards, QC, and samples.

7.3.6.3 Make sure calibration is passing; if not recalibrate.

7.3.7 PRINTING LOADING LIST

7.3.7.1 Click on print list option from workspace. Print window will appear.

7.3.7.2 Change name on print window to HP LaserJet 2100 and click on OK.

7.3.7.3 Click on print set up from the file menu and change name back to pdf and save changes.

7.3.8 ADDING SAMPLES DURING ANALYSIS

7.3.8.1 Click on sample info file icon from the main menu.

7.3.8.2 Type in samples under sample ID and location under A/S location.

7.3.8.3 Save sample info file as File –save –sample info file.

7.3.8.4 Click on append to Analysis list.

7.3.8.5 Enter sample numbers of the first sample and last samples to be added and click on OK. Example – If sample number of the first sample typed in is 15 and the last sample number is 25, enter 15-25.

7.3.9 WinLab32 Offline

7.3.9.1 WinLab32 Offline allows software to be run without controlling the optima.
7.3.9.2 Use to edit method and reprocess data.
7.3.9.3 Click on WinLab32-off-line.
7.3.9.4 Click on examine from the file menu on top.
7.3.9.5 Click on date then select data set
7.3.9.6 Highlight date and select CRI and ICB.
7.3.9.7 Look at peaks for both CRI and ICB and make sure the CRI peaks are higher than the ICB peaks. If not, recalibrate.

7.3.10 RETRIEVING LOST DATA

7.3.10.1 Click on data manager from the desk top.
7.3.10.2 Select data set needed under result name.
7.3.10.3 Click on report.
7.3.10.4 Select Use existing design.
7.3.10.5 Click on browse.
7.3.10.6 Select AES report format.rep. Click on open.
7.3.10.7 Click on preview.
7.3.10.8 Click on printer. Select all and click on OK.
7.3.10.9 Raw data will print to pdf.
7.3.10.10 Save – type File name and click on save.
7.3.10.11 Close out of data manager.

7.3.11 IN CASE OF POWER OUTAGE

7.3.11.1 Make sure spectrometer and chiller switches are turned on.
7.3.11.2 Click on Win Lab32 from the desktop.
7.3.11.3 Wait for spectrometer to warm up for the time indicated in the spectrometer control box.
7.3.11.4 When spectrometer is ready, ignite plasma and follow directions under software start up.

7.3.12 CHANGING FROM ONE METHOD TO ANOTHER

7.3.12.1 Click on the current applicable method from the menu list on the top left corner.
7.3.12.2 Select method needed and click on ok.
7.3.12.2.1 [CURRENT METHOD] for 6010B/ 200.7
7.3.12.2.2 INHOUSE MMDDYY for Inhouse
7.3.12.2.3 SI Test MMDDYY for SI

7.3.13 WHAT TO DO IF WINLAB SOFTWARE CLOSES OUT DURING ANALYSIS

7.3.13.1 Click on WinLab from the desktop
7.3.13.2 Select [CURRENT METHOD] and click on open
7.3.13.3 Select [CURRENT METHOD].icp
7.3.13.4 Click on workspace
7.3.13.5 Click on set up
7.3.13.6 Click on open under Sample Information File
7.3.13.7 Scroll through to find data in use

7.3.13.7.1 Select data and click on open
7.3.13.8 Click on open under result data set
7.3.13.9 Current data will be highlighted. Click on Ok
7.3.13.10 If note pops up saying result data is currently in use, go to data management
7.3.13.10.1 Click on Data Management from the desktop
7.3.13.10.2 Select result set
7.3.13.10.3 Click on file from the main menu
7.3.13.10.4 Click on remove locks
7.3.13.10.5 Select result name and click on remove locks
7.3.13.10.6 A note will pop up. Click on ok
7.3.13.10.7 Close out of data management
7.3.13.11 In the set up menu in the WinLab software
7.3.13.11.1 Click on open under result data set and click on ok to open results in use
7.3.13.12 Click on method and select method in use before software closed out
7.3.13.13 Click on analysis from the main menu
7.3.13.13.1 Scroll down and select recall calibration
7.3.13.13.2 Click on analyze samples. Probe will go into check standard
7.3.13.13.3 Click on analyze samples again to stop analysis
7.3.13.13.4 Click on analyze samples and enter sequence number of sample you wish to continue with.

7.3.14 <u>SETTING UP INSTRUMENT TO SHUT DOWN AT THE END OF ANALYSIS</u>
7.3.14.1 Click on set up from workspace
7.3.14.2 Click on set beside auto shutdown
7.3.14.3 Click in the box beside ‘shutdown’
7.3.14.4 Click in the circle beside ‘At the end of analysis’. Click on ok

7.3.15 <u>SETTING UP INTER-ELEMENT CORRECTIONS (IEC)</u>
7.3.15.1 Open method that need IEC
7.3.15.2 Type in pure solutions for each interfering analyte (Ca, Mg, Fe, Al)
7.3.15.3 Calibrate, run analysis and save data as IEC test
7.3.15.4 Once analysis is complete, select IEC model builder from the tools menu
7.3.15.5 The IEC model builder window set up page appears
7.3.15.6 Select new from the file menu then select IEC model
7.3.15.7 Click open on the set up page to select results from analysis with pure solutions for the interfering analytes
7.3.15.8 Skip the set limits page
7.3.15.9 On the calculate factor page select samples (the pure solutions of the interferents), then match the various analytes requiring interference correction with the samples that contain pure interfering analytes.
7.3.15.10 The factors are automatically calculated after you choose the interfering analyte
7.3.15.11 Click on print on the summarize factor page
7.3.15.12 This page can also be used to edit factors if needed.

7.3.15.13 Select save from file menu and select IEC model to save model as YYMMDD_IEC_CURRENT.iec
7.3.15.14 Click on update method to update analytes in method which require IEC correction
7.3.15.15 Close out of IEC model builder
7.3.15.16 Click on method editor from the work space and select process from the menu list at the bottom of the page
7.3.15.17 Select spectral correction from the side menu
7.3.15.18 Click in the box under overlap correction and select IEC for the analytes that need correction (As, Pb, Se, Co, Mo, Ag, Sn, Tl, Sb and V) and none for all other analytes.
7.3.15.19 Make sure the right model is specified in the box beside IEC model.
7.3.15.20 Go to file save method to save the changes.
7.3.15.21 Calibrate with the new IEC set up and run ICSA standard straight and at 2x and 5x the concentration to check the correction factors.

7.3.16 To edit IEC factors
7.3.16.1 In the Tools menu, click on IEC Model Builder. The IEC window, Set Up page appears.
7.3.16.2 In the File menu, click on Open IEC Model... and select the IEC file name
7.3.16.3 Enter new factors on the Calculate Factors Page.
7.3.16.4 Go to the File menu, select Save IEC Model to save changes
7.3.16.5 If you modified the IEC factors and want to save the changes but also retain the original file, from the File menu select Save As IEC Model.

7.4 Instrument Operation for the Varian Vista Pro.

7.4.1 <u>Software</u>

7.4.1.1 Choose ICPExpert ICON on the desktop. Select '*Worksheet*'. Choose '*New*'. Then '*Sequence'*. Select worksheet type for the following analysis:
7.4.1.1.1 Regular Metals- 200.7_6010.vwst
7.4.1.1.2 Silicon- SILICON.vwst
7.4.1.1.3 Inhouse (Boron, Sulfur, Strontium, Phosphorous) - INHOUSE.vwst.
7.4.1.1.4 Lithium- Lithium.vwst
7.4.1.2 Name file with this format: MMDDYY (LETTER of analysis) (i.e July 7, 2007; 1st worksheet = 070707A). Press '*Save*'.
7.4.1.3 Press 12th ICON from the left or (shift F4) to turn plasma on.
7.4.1.4 Check flow of sample & waste tubing. If not seen, speed the flow rate up by pressing the gas pump button with a solid blue line.
7.4.1.5 Let the instrument warm up for 30 mins. Make calibration standards if not already made.

7.4.2 TORCH ALIGNMENT

7.4.2.1 After 30mins; perform a torch alignment if the torch was replaced. If not then proceed to "Starting Calibration & Analysis".

7.4.2.2 Select Autosampler from the tool bar

7.4.2.3 Under move probe, change rack to 2 and tube to 12

7.4.2.4 Click the Go To button (make sure tuning solution is in position 12 and uncapped).

7.4.2.5 After a few minutes (2-4), Go to the '***Window***' tab of the worksheet, then '***Instrument***' tab then ***'Torch Align'*** tab.

7.4.2.6 Press the ***'Torch Scan'*** button. Once the curve has started on a downward slop, press ***'Stop'*** and change to the other orientation (i.e. Horizontal, Vertical) and repeat procedure.

7.4.2.7 Remove sample probe from the tuning solution and return to the rinse solution by selecting rinse from the autosampler page. The desired range for each orientation is as follows: Horizontal (close to 1), Vertical (close to 0).

7.4.3 STARTING CALIBRATION & ANALYSIS

7.4.3.1 Type in the loading list if samples are present. If not proceed to calibrating.

7.4.3.2 Switch to the ***'Sequence'*** tab of the worksheet.

7.4.3.3 Begin typing from 1:1. (Note: to make the loading list longer, Go to ***'Sequence Editor'*** and change sample count to ~120 or whatever desired

7.4.4 SETTING UP INSTRUMENT TO SHUT-DOWN AT THE END OF THE ANALYSIS

7.4.4.1 If running samples overnight, go to sequence, sequence parameters, and select plasma off/pump off. This will turn plasma off after analysis.

7.4.4.2 Also if loading more than 60 samples, go to ***'Autosampler Setup'*** click on Tray #3 and indicate the 'Rack Type' as 60 x 25ml. Press ***OK*** to update changes.) Switch to the Analysis tab when finished typing in the loading list & highlight all samples to be analyzed by scrolling down left column.

7.4.4.3 Before starting the calibration, click on the ***'Method'*** tab of the worksheet, then ***'Edit Method'*** and ***'Standard'*** tab. Change the curve type for all K & Na from Linear to Quadratic, close out of the window and save changes.)

7.4.4.4 Press the Green arrow button to start the calibration. Make sure all calibration standards are passing; if not, re-calibrate.

Table 7-2
Wavelengths for ICP-PE and ICP Varian Spectrometers

Element	PE 4300Wavelength	Varian Vista Pro Wavelength
Aluminum	308.211	308.215
Arsenic	193.694	193.696
Antimony	206.832	217.582
Barium	413.073	413.064
Beryllium	313.102	313.107
Boron	208.889	249.678
Cadmium	228.800	228.802
Calcium	317.932	373.690
Chromium	267.711	267.716
Cobalt	230.786	230.786
Copper	327.395	327.395
Iron	238.204	238.204
Lead	220.353	220.353
Lithium	--	670.783
Magnesium	279.075	279.800
Manganese	257.610	257.610
Molybdenum	204.596	204.598
Nickel	231.604	221.648
Phosphorus	214.914	213.618
Potassium	766.488	766.491
Selenium	196.026	196.026
Silica	288.158	288.158
Silver	328.069	328.068
Sodium	589.599	589.592
Strontium	232.235	216.596
Sulfur	181.972	181.972
Tin	189.929	189.927
Titanium	337.274	337.280
Thallium	190.801	351.923
Vanadium	292.401	311.837
Zinc	206.198	206.200

Table 7-3
RUN SEQUENCE

1. Check Standard
2. ICV
3. ICB
4. LLICV (CRI)
5. ICSA
6. ICSAB
7. LCS
8. MB
9. Up to 8 samples (6 samples + MS/MSD or 7 samples +LCS)
10. CCV
11. CCB
12. Up to ten samples
13. CCV
14. LLCCV (CRI) – required at end of each analysis batch
15. CCB
16. ICSA
17. ICSAB

Table 7-4
Initial Quality Control Requirements for ICP Analysis

Quality Control Sample	**Acceptable Result**
CRI/LLICV/LLCCV	Various (70-130%)
Check Standard	90-110% recovery
ICV	90-110% recovery
CCV	90-110% recovery
ICB	Result < RL
MB	Result < RL
ICSA	See Section 8
ICSAB	See Section 8

Table 7-5
3010A Prep Checklist
3010A

____ **:** PRINT (check) BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(3010A: 1311_BOTTLE, 1311_M, 1312_B, 6010B_W_T, 6010B_B_W_T, 6010B_PO4_W_T 6010B_SI_W, 6010B_Sr_W_T, 6010B_TAL_W_T, INHOUSE_M_W and SM2340 (if sample is on this report, it may need to go on 200.7)

____ : PICK A SAMPLE FOR QC (homogeneous is ideal), (1311_M for a batch with only 1311_M)

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO (Use the blank that has been tumbled with TCLP samples) (QC, then order of backlog) Write samples in the log book from the back log.

____ : ADD SAMPLE TO DIGESTION VESSEL AND RECORD VOLUME.
If the does not look like drinking water, then evaluate the sample matrix.
-Pour 1ml into test tube and add 1ml DI H2O to check water solubility. If insoluble, see supervisor before proceeding.
-Add 1ml HNO3 to check reaction. If strong or violent reaction occurs, see supervisor before proceeding.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add DUP & MS, check volumes), PRINT A COPY OF THE BATCH

____ : ADD 50 ML of DI WATER to LCS & MB VESSELS from DI wash bottle. USE 10 ML SAMPLE FOR TCLP

____ : SPIKE LCS & MS & MSD with 0.50 ml EACH OF THE 2 MULTI-ELEMENT STANDARDS. (WITNESS NEEDED)
FOR 6010B_SI_W SAMPLES, SPIKE WITH 0.50 ml of the 500 mg/L Si STANDARD ONLY.

____ : ADD 2.5 ml conc. HCL & 2.0 ml conc. HNO_3, cover the vessels with watch glasses and heat for 40-60min.

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50-ML (20-ml for 4X)

____ : ***SHAKE WELL***

____ : LINE UP SAMPLES IN ORDER THAT THEY APPEAR IN LOG

____ : DELIVER SAMPLES TO THE METALS LAB

NOTE: Method SW3010A states that the final acid concentration in the digestate should be no more than 10%. 2.5 mL of conc. HCl equals 5% and 2.0 mL of conc. HNO_3 equals 4% of the 50 mL final volume.

Table 7-6
3050B_S Prep Checklist
3050B_S

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(3050B_S, 6010B_S, 6010_A9_S, 6010B_PO4_S, 6010B_S_CLP, 6010B_TAL_S, INHOUSE_M)

____ : PICK A SAMPLE FOR QC (org., dup, and ms) HOMOGENIZE SAMPLE

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO (QC, then order of backlog)

____ : ADD SAMPLE TO DIGESTION VESSEL AND RECORD WEIGHT (1.0-2.0g)

For the sample chosen for MS/MSD or DUP, the initial weights used for the unspiked sample, matrix spike and matrix spike duplicate, or unspiked duplicate if applicable, must be within 5% of each other.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add DUP & MS, check weight. All 3050B_S batches will need DL & PDS) PRINT A COPY OF THE BATCH

____ : SPIKE LCS & MS with 0.5ml of multi-element std. (WITNESS NEEDED)

____ : ADD 2.0 ML conc. HNO_3, COVER THE VESSELS WITH WATCH GLASSES, THEN HEAT FOR 10 MIN

____ : ADD 4.0 ML conc. HNO_3, THEN HEAT FOR 30 MIN

____ : REMOVE from heat, add 2-3ml DI H_2O, let cool, ADD 1 DROPPER OF 30% H_2O_2, and HEAT FOR 10 MIN

____ : ADD 5 ML HCL, HEAT UNTIL VOLUME drops to 5 ml or 1 HOUR

____ : ***SHAKE WELL***

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50 ML

Batch folders

____ **:** Does it have BACKLOG REPORT FOR THE PREP AND EACH TEST CODE

____ : Is the SAMPLE FOR QC (dup, ms) adequate

____ : ARE the SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : Check SAMPLE final volume in DIGESTION VESSEL, does it match Recorded VOLUME

____ : CHECK prep log against BATCH IN THE LIMS (sample prep, test code, date time, analysts, check sample ID's, OK, add DUP & MS, check volumes), or Printed A COPY OF THE BATCH

Table 7-7
3050B_X Prep Checklist
3050B_X

1) For Samples that are a soil/oil waste mixture or appear to be 90 to 100% Oily matrix:

___ : Print the backlog reports for **3050B_X** and **6010B_X**
-check for comments from Project manager

___ : Pick a sample for QC (org., MS/MSD) and homogenize sample

___ : Put samples in order and check prep and test info (QC then order of backlog). Use crucible for ashing sample. Record sample ID on the crucible bottom using pencil, not Sharpie. Using a Sharpie marker can burn off during ashing. Sometimes pencil will also burn off. Draw diagram of samples to help with identification of samples.

___ : Add 1g to the crucible and record the measurement

For the sample chosen for MS/MSD, the initial weights used for the unspiked sample, matrix spike and matrix spike duplicate must be within 5% of each other.

___ : Ash the sample by placing into a cold muffle furnace and set temp. to 300 to 350°C and let bake for 3-4 hrs., or until sample is fully turned to ash. * **WARNING**- Do NOT open oven if smoke is coming out!

___ : Remove with tongs, let cool

___ : Add **2.0ml of conc. HNO3 and 1.5 ml conc. HCl**, cover the crucible with a watch glass, then heat for 30min.

___ : Remove from heat and let cool. Transfer to a plastic digestion vessel, rinse crucible 2-3 times with DI Water.

___ : Add **2-4 ml of conc. HNO3 and 3-5 ml conc. HCl**, cover the crucible with a watch glass, and heat again until ash is dissolved. NOTE: The process may have to be repeated until ash is dissolved. Record the total amount of conc. HNO3 and HCl used to dissolve sample ash.

___ : Prepare clean, snap cap vials for MB and LCS.

___ : Spike LCS, MS, MSD with 0.5 mL of multielement Standard (need witness).

___ :Add the same amount of conc. HNO3 and conc. HCl to MB and LCS that was required to dissolve sample ash.

___ :Digest in hotblock ~ 30 to 45 minutes.

___ : Remove from heat and let cool. Bring samples to a final volume of 50 mL with DI Water.

2) For Samples that are a liquid waste matrix containing an oily layer:

___ : Print the backlog reports for **3050B_X** and **6010B_X**
-check for comments from Project manager

___ : Pick a sample for QC (org., MS/MSD) and homogenize sample

___ : Put samples in order and check prep and test info (QC then order of backlog)

___ : Add 1g to a digestion vessel and record the weight in the logbook.

For the sample chosen for MS/MSD, the initial weights used for the unspiked sample, matrix spike and matrix spike duplicate must be within 5% of each other.

____ : SPIKE LCS, MS, & MSD with 0.5ml of multi-element std. (WITNESS NEEDED)

____ : ADD 2.0 ML conc. HNO_3, COVER THE VESSELS WITH WATCH GLASSES, THEN HEAT FOR 10 MIN

____ : ADD 4.0 ML conc. HNO_3, THEN HEAT FOR 30-35 MIN

____ : REMOVE from heat, let cool, ADD 1 DROPPER OF 30% H_2O_2 to remove organic material. Digest and check for reaction. Add another dropper of 30% H_2O_2. Digest again and check for reaction. If reaction is strong, then add another dropper of 30% H_2O_2. Digest until reaction stops.

____ : REMOVE FROM HEAT AND LET COOL.

NOTE: Sometimes the an oily residue will be noticed in the vial. If this happens, sample will be transferred to a clean, snap cap vial prior to digestion. Filter if necessary with Whatman 40 filters.

____ : ADD 2-3 ML OF DI WATER AND 5.0 ML OF HCl.

____ :DIGEST FOR 40 MIN.

____ : REMOVE FROM HEAT AND LET COOL.

____ :BRING TO A FINAL VOLUME OF 50 ML.

Table 7-8
SM3030C

____ **:** PRINT (check) BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (SM3030C)

SAMPLES FOR SM3030C REQUIRE FILTRATION WITHIN 72 HOURS OF COLLECTION. CHECK TO SEE IF SAMPLES WERE FIELD FILTERED; CHECK COMMENTS IN LIMS. IF NOT, PROCEED WITH FILTRATION USING 0.45 MICRON FILTER.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add DUP & MS, check volumes), PRINT A COPY OF THE BATCH

____ : PICK A SAMPLE FOR QC (homogeneous is ideal)

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO (QC, then order of backlog) Write samples in the log book from the back log.

____ : ADD 50 ML SAMPLE TO DIGESTION VESSEL

____ : ADD 50 ML of DI WATER to LCS & MB VESSELS from DI wash bottle

____ : SPIKE LCS & MS with 0.5ml multi-element std. (WITNESS NEEDED)

____ : ADD 0.25 ML **CONC.** HNO3 TO 50 ML MB AND LCS. DO NOT ADD HNO3 TO SAMPLES

____ : ADD 1.25 ML **CONC**. HCl TO ALL SAMPLES AND MB AND LCS

____ : HEAT FOR 15 MINUTES IN HOT BLOCK AT APPROX. 95°C. DO NOT ALLOW SAMPLE TO BOIL

____ : REMOVE FROM HEAT AND LET COOL.

____ : FILTER THE DIGESTATE USING DISPOSABLE 0.45 µm SYRINGE FILTER. RINSE THE VESSEL WITH 1-2 ml DI WATER AND FILTER THE RINSE WATER

____ : BRING THE FINAL VOLUME OF FILTRATE TO 50 ML

____ : ***** SHAKE WELL *****

____ : LINE UP SAMPLES IN ORDER THAT THEY APPEAR IN LOG

____ : CHECK THE ORDER OF THE SAMPLES IN THE BATCH

____ : CHECK THE ELEMENTS TO BE ANALYZED

____ : TYPE SAMPLES INTO COMPUTER AND POUR TRAY

____ : CHECK THE ORDER, LAST POSITION ON TRAY

See Reference 16.6 of SOP

Table 7-9
SAMP_FILT (For Dissolved Metals)

____ **:** PRINT (check) BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (SAMP_FILT, 6010B_W_D, 200.7_W_D, 200.8_D, 6020_A1(A2,A9)_W_D and 6020_W_D)

____ : PICK A SAMPLE FOR QC(MS&MSD) (homogeneous is ideal)

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add MS/MSD, check volumes), PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO. Write samples in the log book from the back log.

____ : FILTER 50 ml SAMPLE THROUGH 0.45 µm SYRINGE FILTERS TO DIGESTION VESSEL. (IF THE SAMPLE HAS A BAD MATRIX WHICH MAKES IT DIFFICULT TO FILTER, YOU CAN REDUCE THE VOLUME FILTERED to 25ml)

____ : FILTER 50 ml SAMPLE TO MS AND MSD VESSELS FOR THE QC SAMPLE.

____ : FILTER 50 ml of DI WATER THROUGH SYRINGE FILTERS to LCS & MB VESSELS.

FOR ALL 200.8 AND 6020 TEST CODE PREPS:

____ : ADD 1.0 ml OF **OMNITRACE HNO_3/ 50ml sample** TO EACH SAMPLE AND QC AND SHAKE WELL.

____ : SPIKE LCS, MS & MSD with 0.5ml of **10** ppm multi-element std. (WITNESS NEEDED). **SHAKE WELL**

FOR ALL 200.7 AND 6010 TEST CODE PREPS:

____ : ADD 0.5 ml OF HNO_3 **AND** 0.5 ml HCL/ **50ml sample** TO EACH SAMPLE AND QC AND SHAKE WELL.

____ : SPIKE LCS, MS & MSD with 0.5ml of **100** ppm multi-element std. (WITNESS NEEDED). **SHAKE WELL**

IF THE SAMPLES ARE FIELD FILTERED:

____ : ADD 50 ml SAMPLE TO DIGESTION VESSEL.

____ : ADD 50 ml SAMPLE TO MS AND MSD VESSELS FOR THE QC SAMPLE.

____ : ADD 50 ml DI WATER TO LCS & MB VESSELS.

____ : ACIDIFY FOR EITHER 200.7/6010 TEST CODES OR 200.8/6020 TEST CODES AS ABOVE

____ : SPIKE LCS, MS & MSD FOR EITHER 200.7/6010 TEST CODES OR 200.8/6020 TEST CODES AS ABOVE

____ : ******SHAKE WELL******

____ : LINE UP SAMPLES IN ORDER THAT THEY APPEAR IN LOG

Table 7-10
PREP CHECKLIST

WIPE_MET_ICP_P

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (6010_Wipe).

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO.

____ : BATCH SAMPLES IN THE LIMS, PRINT BATCH.

____ :PREPARE MB BY USING GHOST WIPE.

____ : PREPARE LCS & LCSD (WITNESS NEEDED) BY USING A BLANK GHOST WIPE AND ADDING **0.50 g of soil reference material. Record AES number in logbook of soil reference material.**

____ : PREPARE DIGESTED CRA (**REQUIRED FOR LEAD ONLY**) (WITNESS NEEDED) BY ADDING **2.0 ml** OF **10 mg/l** LEAD CRA STANDARD TO A BLANK WIPE IN A DIGESTION TUBE.

____ : ADD 5 ML CONC. HNO_3. WIPE MUST BE COMPLETELY COVERED WITH DIGESTION SOLUTION; ADD DI WATER IF NECESSARY.

____ : HEAT FOR AT LEAST 10 MIN FOR GHOST WIPES, OR UNTIL SAMPLE HAS THE CONSISTENCY OF MASHED POTATOES WHICHEVER IS LONGER.

____ : REMOVE FROM HEAT AND COOL FOR APPROX. 5 MINUTES.

____ : ADD 6 ML 30% H2O2.

____ : HEAT FOR AT LEAST 10 MIN

____ : ADD 5 mL HCl.

____ : HEAT FOR AT LEAST 10 MIN.

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF **100 ML.**

____ : ***SHAKE WELL***

Table 7-11
Data Review Checklist

DATA REVIEW CHECKLIST- ICP/OES

Batch ID: _____ Data File: _____

QA Analyst

INITIAL RAW DATA REVIEW

___ ___: Have raw data, and run log(s) been printed to pdf and posted to Portal Server
___ ___: Are all instruments standards used listed on raw data
___ ___: Do calibration curves for all target analytes meet requirements (≥0.998 Corr.Coef)
___ ___: Are all replicate RSDs ≤ 15% for all values over PQL? **No requires reanalysis and CAR if still out at reanalysis**
___ ___: Are there any "saturated" readings for targets or interferants for any samples? **Yes require dilution**

GO TO LIMS "MAIN" RUN SCREEN

___ ___: Are all Sample IDs properly assigned per Backlog Report (Double click each SampleID to verify)
___ ___: Are all Test Codes properly assigned per Backlog Report
___ ___: Is instrument QC run at the required frequencies
___ ___: Are all Sample Types properly assigned
___ ___: Are all samples linked to the Prep Batch properly with correct PFac, SpkFac and OFac
___ ___: Are dilution factors entered correctly per the raw data Run Log
___ ___: Are all Blkref, SPKref, RPDref and CCVref assigned correctly
___ ___: Are there any Comments present that require attention? **May require CAR**

GO TO DATA SCREEN

___ ___: Calculate Sequence (Cal SEQ tab) to insure LIMS calculations are completed.
___ ___: Are there any S or B flags for target analytes on the Instrument QC (ICV, CRI,CCV,etc. **Yes Requires CAR**
___ ___: Are all values for target analytes between <PQL and > -PQL on ICB, CCB and MBs? **No Requires CAR**
___ ___: Are all values for targets analytes (except interferents) between<PQL and >-PQL on ICSA? **No requires CAR**
___ ___: Are there any S flags for element of interest on any samples and/or batch QC? **Yes Requires CAR**
___ ___: Are there any B flags for elements of interest on any samples and/or Batch QC? **Yes Requires CAR**
___ ___: Are there any S and/or R flags for elements of interest on Batch QC (LCS/D, MS/D, DL, PDS)? **Yes Requires CAR except R or S flags on DL or PDS cause by value at or near PQL or insignificant spike.**
___ ___: Are there any H flags present for any samples? **Yes Requires CAR**
___ ___: Are there any E flags for elements of interest on any samples and/or Batch QC selected for reporting? **Yes Requires CAR**
___ ___: Are there any J flags on any target analytes selected to report on diluted sample runs? **Yes Requires reanalysis at lower dilution or CAR to narrate elevated reporting limits**
___ ___: Have readings been entered properly (check at least 2 entries for downloaded data, **ALL** entries for manual data)
___ ___: Have at least 2 sample's final results in LIMS been verified by hand calculation
___ NA: Have all CARs been closed and narratives written prior to QA?
___ NA: Have PM, Lab Manager and PM Director been notified for any CAR resulting in reextract and/or due date exceedance?

CAR#: _____ _____ _____ _____

Analyst Signature: _____ Date: _____ Time: _____

Reviewer Signature: _____ Date: _____ Time: _____

Table 7-11 (cont)
Data Review Checklist

DILUTIONS

Sample ID	Metal(s) Needed	Done
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes

REANALYSIS

Sample ID	Metals Needed	Done
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes

Comments:

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP# QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Calibration of the ICP. This is accomplished through a 3-point calibration curve (blank and 2 standards). The correlation coefficients (r) for all target analyte curves must be $\geq$0.998. Calibration curves on the ICP-Varian for Sodium and Potassium are constructed using quadratic curve fit with a correlation coefficient (r) that must be $\geq$0.998.

8.1.3 Initial Calibration Verification (ICV). An ICV standard must be analyzed after establishment of each calibration curve. Values must be within ±10% of true value.

8.1.4 Continuing Calibration Verification (CCV). A CCV standard must be analyzed after every 10 injections and to close each run/sequence. Values must be within ±10% of true value.

8.1.5 Continuing Calibration Blank (CCB). A CCB must be analyzed immediately after each CCV sample. Value must be less than the PQL for all target analytes.

8.1.6 Low-Level Initial/Continuing Calibration Verification standard (LLICV/LLCCV or CRI). The LLICV/LLCCV or CRI standard should be made at the established lower limit of quantitation. The standard must be analyzed at a concentration expected to be the lower limit of quantitation, and at the beginning and at the end of each analysis batch with closing CCB/CCV. A more frequent analysis may be necessary to avoid re-analysis should the LLCCV fail if only analyzed at the end of the analysis batch. The acceptance criteria for the LLICV/LLCCV/CRI standard should be $\pm$30%.

8.1.7 Method Detection Limit Study (MDL). The method detection limit is calculated by analyzing at least seven replicates prepared and analyzed at concentrations at or near the lowest reporting limit to be used for samples in the appropriate matrix. All MDLs must be less than the PQL for each target metal. MDL's

must be updated at least annually and whenever instrument conditions have changed such that the established detection limits may have been affected. Current MDLs and PQL can be found in LIMS.

8.1.8 Lower Limit of Quantitation Check Sample (LLQC). The Lower Limit of Quantitation Check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard (LLCCV) should be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ±30% of their true value. This check should be used to both establish and confirm the lower quantitation limit.

8.1.9 Instrument Detection Limits (IDL). IDLs are verified quarterly by analyzing reagent blank samples for seven consecutive measurements each day for three non-consecutive days. Standard deviations for each day are calculated with final IDLs equal to 3X the average standard deviation from the three days for each target metal.

8.1.10 The upper limit of the linear dynamic range (UQL) must be established for each wavelength utilized. The upper limit is determined by the Linear Dynamic Range study. The results of at least 3 consecutive standards up to this concentration must fall within 90-110% of true value. Once established, any raw sample values over the established UQL must be diluted and reanalyzed. Linear ranges must be verified and/or re-established at least every six months and whenever instrument conditions have changed such that the established UQL values may have been affected.

8.1.11 Interelement Correction (IEC). The laboratory must verify the absence of spectral interferences or establish Interelement Correction Factors (IEC) to be used during all sample analysis. The procedure for generating IECs is specific to each instrument and described in Section 7. The criteria for verifying the absence of spectral interference with or without the use of IECs is as follows:

8.1.11.1 Analysis of the ICSA solution with each unspiked target analyte result of zero ± one PQL. ICSA must be analyzed immediately following calibration and at the end of each analytical sequence with closing CCV/CCB.

8.1.11.2 Analysis of the ICSAB solution with each spiked target analyte (excluding interferents) recovery ± 20% of the expected value. ICSAB must be analyzed immediately following calibration and at the end of each analytical sequence with closing CCV/CCB.

8.1.11.3 Once established, IECs must be updated at least every six months and whenever instrument conditions have changed such that the

established IECs may have been affected. All data used for the generation of IECs must be kept on file.

8.1.12 Matrix spike. Matrix spikes are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples of similar matrix. The recovery of the analytes should meet established laboratory guidelines.

8.1.13 Method blank. Reagent blank analysis must be performed at the following frequency: Every twenty (20) samples or once per Batch of samples of similar matrix or whenever samples are extracted and analyzed by the same procedure at same time, whichever is more frequent. The concentration of the method blank of any analyte of interest must not exceed the laboratory established practical quantitation limit (PQL).

8.1.14 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD). The LCS is used to monitor, assess, and document laboratory method performance and is performed on every batch or twenty samples, whichever occurs more frequently. The LCSD is only analyzed in the absence of an MSD. The recovery of the analytes must be within $\pm$20%. RPD value when compared to the LCS must be $\leq$20%. Recovery or RPD outside control limits must be handled in accordance with Section 8.2.

8.1.15 Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD). Matrix spikes are used to determine the effect of the sample matrix on the recovery and reproducibility of analytes, and are prepared and analyzed with each analytical batch up to 20 samples. The recovery of the analytes should be within $\pm$25%.

8.1.16 Post Digestion Spike (PDS). If the MS/MSD recoveries are unacceptable , the same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. Otherwise another sample from the sample preparation should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. If this spike fails, then the Dilution Test (8.1.17) should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed, and the data should be appropriately narrated.

8.1.17 Dilution Test (DL). If the analyte concentration is sufficiently high (minimally, a factor of 10 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within $\pm$10% of the original determination. If not, then a chemical or physical interference effect should be suspected, and the data should be appropriately narrated.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP# QA-01000, “Quality Assurance Manual,” in Section 5. Included are tables that detail corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance, and any procedure deviations in appropriate logbooks.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 Health and Safety: Safety glasses and latex type gloves must be worn at all times when dealing with any chemicals, samples, or reagents. A lab coat is also highly recommended. Close-toed shoes and clothing that covers the legs (no shorts or dresses) must be worn any time an analyst is working in the laboratory.

9.2 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a health hazard and exposure should be kept as low as reasonably possible. All health and safety concerns for any chemicals are listed in the Material Safety Data Sheets (MSDS) provided by the supplier or manufacturer of these chemicals. A copy of any MSDS is available for review at any time.

9.3 The exhaust hoods should be maintained in a clean condition. Avoid getting Kimwipes or other paper inside the hood stack. This is not only a fire hazard but can also decrease the flow within the hood.

9.4 Acids should be handled with care. Always add acids and caustic solutions to water.

10.0 DATA REPORTING

10.1 The LIMS system automatically calculates the data based upon factors that are set up for each test category.

10.2 Current laboratory MDLs and Reporting Limits (PQLs) can be found in tables in Section 5 of SOP# QA-01000, “Quality Assurance Manual” and LIMS test codes.

10.3 Calculations are performed by LIMS as follows:

10.3.1 Aqueous Samples:

$$\textbf{mg/L} = \frac{\textbf{Inst. Result (ug/ml) x Final Vol. (ml) x DF}}{\textbf{Volume Digested (ml)}}$$

10.3.2 Soil Samples:

$$\textbf{mg/Kg} = \frac{\textbf{Inst. Result (ug/ml) x Final Vol. (ml) x DF}}{\textbf{Initial Sample wt (g) (x decimal \%Solids if dry wt basis)}}$$

10.3.3 Surface Wipe Samples:

$$\textbf{ug, Total} = \textbf{Inst. Result (ug/ml) x Final Vol. (ml) x DF}$$

1(entire wipe digested)

10.3.4 Data import to LIMS for Varian Vista Pro:

10.3.4.1 Data import to excel spreadsheet from software.

10.3.4.1.1.1 Go to ***'File'***. Click on Export. Say Yes to Overwrite. Double click on ***'Shortcut to Export Icon'*** on desktop. Double click on ***'Data_Prep.xls'***, then follow along ***'Tools-Macro-Macros-Macro1-Run'***, and enter test code (ie. 6010B_W_T).

10.3.4.1.1.2 When the next screen appears, check the desired data to import. Always delete rows 2, 3, 4 and the other rows from other data that will not be imported. Verify that the sample name, sample type, and test code are correct.Change ***'Samp ID'*** column to Abbreviations (ie. Cont Calib Verif= CCV) & ***'Samp Type ID'*** for each sample. Verify that all samples and QC samples such as CCV, CCB, ICAL, ICSA, are imported according to correct test code (this varies).

10.3.4.1.1.3 Click on the following tabs to end procedure. ***'Tools-Macro-Macros2-Run-OK'***, say ***'Yes'*** to command. Close Excel & open LIMS.

10.3.4.2 **Data Export to LIMS from Excels spreadsheet.**

10.3.4.2.1 Click on '*Data Entry*'.

10.3.4.2.2 Fill out the following lines: 1) Instrument ID, 2) Run Start Date, 3) Analyst

10.3.4.2.3 Click on the ***"Data Import"*** box (on the right side of the screen).A box titled ***"File Import Specifications"*** will appear. In the pull-down menu for specifications, select **"ICP_Varian"**. Then click on ***"Run Import".***

10.3.4.2.4 A box will appear that contains a directory of data files. Select the directory ***'LIMSdata.xls'*** and press ***'Open'***.

10.3.4.2.5 Press ***"OK".***

10.3.4.3 **SAVE DATA**

10.3.4.3.1 **Save Data.** Save both raw data & run log on desktop.

10.3.4.3.2 Go to the ***'File'*** tab- ***'Report Settings'***- *Style- Rack loading guide-* ***Print*** (to Adobe PDF) - ***OK***. Save as MMDDYY A, B, C, etc._Run Log.pdf. (Note: Include in the naming before run log Lithium, Silicon/ INHOUSE if it is that type of analysis).

10.3.4.3.3 Go to Report Settings window- click on **'All Data'** from *'Style'* section- Select '**QC Solutions**' from '*Content*' section. Print (to Adobe PDF) - OK. Save as MMDDYY A, B, C, etc_Raw Data.pdf (Note: Include Lithium, Silicon, INHOUSE in the naming before raw data if it is that type of analysis).

10.3.4.4 **Copy and Paste Raw Data & Run Log to Portal Folder.**

10.3.4.4.1 Click on the following: 'Metals on Lab' Icon on desktop- 'ICP_VARIAN'-Current Year- Month of Analysis.

10.3.4.4.2 Include MET #s which can be found in a previous run of the appropriate analysis, on the raw data; copy and paste MET #s in folder created. Include the name of the person who checked the loading list on the run log.

10.3.4.4.3 Create a new folder. Rename as ICP_VARIAN_YYMMDD A,B,C,etc. Cut and paste both raw data and run log into the folder.

10.3.5 Data import to LIMS for PE 4300:

10.3.5.1 EXPORTING DATA

10.3.5.1.1 Click on Data Manager from the desktop

10.3.5.1.2 Click on the data set desired under result name and then export

10.3.5.1.3 Select Use existing design and click on browse

10.3.5.1.4 Select AES Export Test.xpt, then open, click next several times, then export data, then finish. Close out of data manager

10.3.5.1.5 Click on Shortcut to Export from the desktop

10.3.5.1.6 Data_Prep-Enable Macros-Running man-Enter test code- ok

10.3.5.1.7 Delete calibration and samples not needed

10.3.5.1.8 Type in LCS, MBLK, MS, MSD, DL, PDS under sample type.

10.3.5.1.9 Click on the red diamond, select yes to overwrite data

10.3.5.1.10 Close out of Data_Prep

10.3.5.2 EXPORTING INHOUSE AND SI DATA

10.3.5.2.1 Click on data manager from desk top

10.3.5.2.2 Click on data set desired under result name and then export

10.3.5.2.3 Select Use Existing design and click on browse

10.3.5.2.4 Select AES Export Test.xpt and click on open

10.3.5.2.4.1 Click on next

10.3.5.2.4.2 Click on add all to add analytes to the list of selected analytes on the third screen- Select analytes to export.

10.3.5.2.4.3 Next-next-next---next

10.3.5.2.4.4 Export data

10.3.5.2.4.5 Finish

10.3.5.2.4.6 Continue with step 10.3.5.1.5 from above.

10.3.5.3 IMPORTING DATA IN LIMS

10.3.5.3.1 Click on Data Entry

10.3.5.3.2 Click on add from the menu list

10.3.5.3.3 Scroll down and select ICP_PE in Instrument ID

10.3.5.3.4 Fill in date and analyst

10.3.5.3.5 Click on data import

10.3.5.3.6 Scroll down and select ICP_Varian under specification

10.3.5.3.7 Make sure Use Alternate Ids is checked

10.3.5.3.8 Click on run import
10.3.5.3.9 Select Limsdata and click on open. Click on ok

11.0 FILE MAINTENANCE

11.1 All data are printed or scanned to pdf files and electronically stored on the Portal Server.

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto CD-ROM disks. Two copies are made. One copy of the disks is stored on the laboratory premises, while the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks. Instrument logbooks must be completed any time maintenance is performed on the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of the GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as the replacement of motors in tower autosamplers.

Each instrument logbook must have a cover page that includes the following information.
Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.2 Routine Maintenance: Typical routine maintenance consists of keeping the system clean and insuring that all generated QC remains acceptable.

12.2.1 Routine Maintenance of the ICP-Varian.

12.2.1.1 **VISUAL INSPECTION**
Spray Chamber, Nebulizer, Torch, Cone, Pump Tubing (sample & waste), and Exhaust. Check the ICP-VARIAN maintenance logbook to verify if the tubing & torch need to be replaced. Rule of Thumb- tubing/ torch are always replaced every other day; however, they can be changed daily if the torch is dirty or tubing has been clamped overnight.

12.2.1.2 **To change the torch**:
12.2.1.3 Open plasma door.
12.2.1.4 Remove all connections from torch.
12.2.1.5 Press down & twist latch to the left.
12.2.1.6 Remove the torch towards the left. Place the torch in concentrated HCL for cleaning for a period not to exceed 24 hours. Remove from acid, rinse with deionized water, dry with paper towel, and store.
12.2.1.7 Remove the cone by unscrewing counterclockwise.
12.2.1.8 Clean cone in hot/warm tap water with scouring pad.

12.2.1.9 Dry cone with paper towel.
12.2.1.10 Put cone back first. (Note: Can only fit one way, rotate until screws fit.)
12.2.1.11 Place torch back in plasma compartment and the connections to it. The tube closest to the gas outlet is placed in the nearest hole.
12.2.1.12 Close plasma compartment.
12.2.1.13 Change sample & waste tubing. Remove one end first and replace with new tubing. Follow along the path and replace the other ends.
12.2.1.14 Clamp tubing down.
12.2.1.15 A torch alignment is performed any time the torch is replaced. Refer to section 7.4.2 for instructions on how to perform a torch alignment.
12.2.1.16 Clean nebulizer as needed by squeezing HNO3 through it.
12.2.1.17 Spray chamber is cleaned as needed by sonicating with Triton X-100

Note: The software has a help menu that can be referred to for additional assistance.

12.2.2 Routine Maintenance of the ICP-PE

12.2.2.1 Change pump tubing daily. Black tubing goes with the BB marking and the red tubing goes with the RR marking on the pump. Follow the To and From directions marked on the pump. Clamp tubes in place.
12.2.2.2 Inspect torch, injector, and bonnet. Make sure glassware is clean with no traces of deposits or melting. Change torch and injector on Mondays and Wednesdays.
12.2.2.3 Change radial and axial purge windows every month or sooner if needed.
12.2.2.4 Change/clean all three filters on Mondays
12.2.2.5 Make sure there is enough coolant fluid in the chiller. If low top off reservoir with coolant fluid.
12.2.2.6 Check and make sure that the nebulizer is not clogged.
12.2.2.7 Fill out maintenance logbook daily

12.2.3 <u>CHANGING THE TORCH AND INJECTOR</u>

12.2.3.1 Pull open clamp that holds spray chamber in place, this will push the spray chamber out.
12.2.3.2 Hold on to spray chamber and lift it out and disconnect it from the injector adaptor.
12.2.3.3 Open door to sample compartment
12.2.3.4 Grasp torch coupler and turn it counter clockwise until it lines up with the XX mark. Carefully slide torch out.
12.2.3.5 Loosen nut on torch holder and remove torch and O-rings.
12.2.3.6 Copper strip, torch and injector are in the drawer opposite the instrument.
12.2.3.7 Place copper strip over ignitor hole on a clean torch.
12.2.3.8 Push clean torch down slowly into torch mount with the glass inlets facing in front. Rotate torch so that the strip on the torch lines up with the index mark on the torch mount and the guide pin on the torch coupler. Replace O-rings and tighten nut.

12.2.3.9 Push and hold the injector lock with thumb and pull injector out of the torch coupler

12.2.3.10 Loosen nut and remove injector and O-rings from injector mount. Place clean injector and O-rings back on mount and tighten nut.

12.2.3.11 Push and hold injector lock while pushing injector into the coupler until it locks into place. Injector should be 1.5mm below the inner glass of the torch.

12.2.3.12 With the copper strip facing down, slide torch coupler clockwise into the torch mount so the two locking lugs and guide pin in the torch coupler engage the channels in the torch mount. Rotate the torch clockwise until it reaches the -2 position.

12.2.3.13 Make sure that the Ground Pointer is in close proximity to the end of the torch glass but not inside the torch glass

12.2.3.14 Make sure the ignitor contact finger is on the copper strip

12.2.3.15 Slide the bonnet into position around the coil and the torch so that it is not touching the radial window.

12.2.3.16 Orient the spray chamber so that the drain connection is facing down

12.2.3.17 Push the spray chamber into the spray chamber mounting block. Close the clamp around the spray chamber until it is locked into place.

12.2.3.18 Make sure drain tubing is connected to the spray chamber.

12.2.3.19 Remove copper strip from torch being replaced and clean both torch and injector by soaking them in 10% nitric acid for several hours. Rinse torch and injector with DI water and allow to air dry.

12.2.3.20 Clean O-rings with soap and water, rinse thoroughly.

12.2.3.21 Clean spray chamber and nebulizer with deionized water as needed

12.2.4 CHANGING THE WINDOWS

12.2.4.1 Open door to sample compartment.

12.2.4.2 Remove torch.

12.2.4.3 Radial and Axial windows are in the drawer opposite the instrument.

12.2.5 Axial Purge Window

12.2.5.1 Turn the axial purge window holder counter clockwise so that it unlocks from the shoulder screws which are part of the axial window mount.

12.2.5.2 Pull out and remove axial purge window assembly and place it on the table.

12.2.5.3 Use a 2 mm hex key to unscrew one flat head screw and loosen the other so that the two halves of the assembly that secure the axial window cap to the axial window holder can be twisted apart

12.2.5.4 Remove axial purge window from the window holder and replace with clean one.

12.2.5.5 Place the axial window cap in front of the window holder with the clean window and replace the flat head screws.

12.2.5.6 Orient the axial purge window assembly so that the notch on the axial window holder lines up with the purge tube fitting.

12.2.5.7 Push the assembly into the mount and twist the axial window assembly clockwise so that it locks with the shoulder screws which are part of the axial window mount

12.2.6 Radial Purge Window

12.2.6.1 Loosen the knurled nut that holds the radial purge window in place and remove radial window and place on table.

12.2.6.2 Put clean O-ring on clean window and adjust height of the O-ring so that it is about the same as the height of the O-ring on the window being replaced.

12.2.6.3 Place the knurled nut on top of the O ring.

12.2.6.4 Insert the radial window tube into the knurled nut and gently push it down through the O ring.

12.2.6.5 Radial purge window should be about the same height as the Shear gas nozzle.

12.2.6.6 Shear gas nozzle should be at a 90^{o} angle to the torch

12.2.7 CHANGING THE AIR FILTERS

There are three air filters, one on the chiller and two on the spectrometer.

12.2.7.1 Changing the chiller air filter

12.2.7.1.1 Slide air filter out from the front.

12.2.7.1.2 Rinse filter with Di H2O and allow to air dry.

12.2.7.1.3 Replace with clean filter.

12.2.7.2 Changing spectrometer air filters

12.2.7.2.1 Filter in front the fan

12.2.7.2.1.1 Pull off the snap-on plastic grid that holds filter in place.

12.2.7.2.1.2 Remove dirty filter and replace it with a clean and dry filter.

12.2.7.2.1.3 Push plastic grid back in place.

12.2.7.2.1.4 Wash dirty filter with DI water and allow to air dry.

12.2.7.2.2 Filter in the back of the instrument

12.2.7.2.2.1 Slide filter out.

12.2.7.2.2.2 Replace with clean dry filter. Make sure the fine screen side is towards the instrument.

12.2.7.2.2.3 Wash filter with DI water and allow to air dry

12.3 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as an autosampler that will not function. If this type of problem occurs, contact the department manager for assistance.

13.0 METHOD PERFORMANCE

13.1 Method performance data can be found in the referenced methods.

13.2 Laboratory specific method performance data is referenced in Section 17 of this SOP.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 SOP# HS-03005 Waste Disposal and SOP# SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade –A dry chemical that has been dried at 250°C for 4 hours cooled and stored in a desiccator.

15.2 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to distilled water or clean soil and the concentration measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.3 DI water- Deionized water

15.4 RSD – Relative Standard Deviation

15.5 MS- Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the

measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.6 MSD- Matrix Spike Duplicate.

15.7 CCV - Continuing calibration verification standard.

15.8 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot that the calibration standard.

15.9 LCSD - Laboratory Control Sample Duplicate

15.10 Dissolved - Those elements which will pass thorough a 0.45 µm membrane filter.

15.11 Total Recoverable or Total Metals – The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

15.12 Interference Check Samples (ICSA/ICSAB) **–** A solution containing both interfering and analyte elements of known concentration that can be used to verify background for the inter-element correction factors.

15.13 Linear Dynamic Range – The concentration range over which the analytical curve remains linear.

15.14 Interelement Correction Factor (IEC) – The mathematical formula for the correction of a positive or negative bias on a target analyte due to spectral overlap.

15.15 Reagent blank - A volume of deionized water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.

15.16 Calibration blank - A volume of deionized water acidified with HNO3 and HCl.

15.17 Quality control sample - A solution obtained from an outside source having known concentration values to be used to verify the calibration standards.

15.18 Calibration standards - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

16.0 REFERENCES

16.1 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, February 2007, Methods 6010C.

16.2 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, April 1998, Methods 3010A and 3050B.

16.3 NIOSH Manual of Analytical Methods, Fourth Edition, Method 7105, Issue 2.

16.4 Standard Methods for the Examination of Water and Wastewater, 20th Edition, Method 3030C.

16.5 *Guidelines for Sampling*, Tables 3 and 8, UST Section NCDENR Division of Waste Management, July 15, 2008 Version, Change 1, Effective December 1, 2008.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://home/aes/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://home/aes/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

AES, Inc.
3785 Presidential Pkwy.
Atlanta, GA 30340

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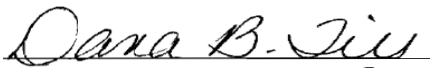
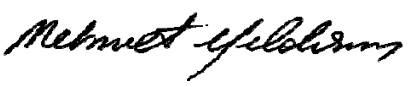
Technical Director:  Date: 5/13/10

Laboratory Manager:  Date: 5/13/10

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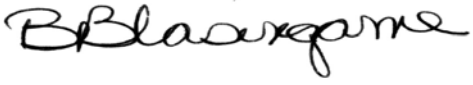
Department Supervisor:  Date: 5/10/10

STANDARD OPERATING PROCEDURE FOR
DETERMINATION OF METALS IN WATER, SOILS, WASTES, AND SURFACES WIPES BY ICP/MS BY SW-846 METHOD 6020A/3005A/3050B/NIOSH7300

TABLE OF CONTENTS

Page

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-mass spectrometry determines trace metals in solution. The method is applicable to all of the elements listed in Table 2-1. All matrices, excluding filtered groundwater samples but including groundwater, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, and surface wipes require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion when results are reported as "dissolved metals". Internal standards are used for all analyses. Refer to the procedure section for appropriate digestion procedures.

1.2 Table 2-1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the mass, spectrometer, matrix and operating conditions. Table 7-1 lists the routine analytical masses used for quantitation. Laboratory determined detection limits are listed in the QA Manual, Section 5. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements and matrices other than those listed in Table 7-1 may be analyzed by this method if performance at the concentration levels of interest is demonstrated.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be acidified, solubilized, or digested using appropriate sample preparation procedures as mandated by the EPA and/or NIOSH as appropriate.

2.2 For the determination of dissolved analytes in a filtered aqueous sample, or for the “direct analysis” total determination of analytes in drinking waters, the sample is made ready for analysis by the appropriate addition of nitric acid, and then diluted to a predetermined volume and mixed before analysis.

2.3 Determination of the analyte concentrations is accomplished by introduction of the sample solution by pneumatic nebulization into a radio frequency plasma where the energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer with minimum capability of 1amu peak width at 5% peak height. The ions are detected by an electron multiplier or Faraday detector and the ion information is processed by a data handling system. Interferences relating to the technique are corrected for. All quantitation is accomplished using internal standard calibration with internal standard solution added in line by the sample introduction system.

2.4 The following analytes may be analyzed using this method:

Table 2-1
Metallic Analytes Analyzed by ICP/MS

Aluminum	Antimony	Arsenic
Barium	Beryllium	Calcium
Chromium	Cadmium	Copper
Cobalt	Iron	Lead
Magnesium	Manganese	Molybdenum
Nickel	Potassium	Selenium
Sodium	Silver	Thallium
Tin	Titanium	Vanadium
Zinc		

3.0 INTERFERENCES

3.1 Several interference sources may cause inaccuracies in the determination of trace elements by ICP-MS. These are:

3.1.1 Isobaric elemental interferences - Are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method (Table 3-1), only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, one or more isobaric interferences may occur. All data obtained under such conditions must be corrected for these interferences by measuring the signal from other isotopes of potential interfering elements and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

3.1.2 Abundance sensitivity - Is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and mass analyzer operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

3.1.3 Isobaric polyatomic ion interferences - Are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope

of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified, and these are listed in Table 3-2 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions. In particular, the common 82 Kr interference that affects the determination of both arsenic and selenium, can be greatly reduced with the use of high purity krypton free argon.

3.1.4 Physical interferences - Are associated with the physical processes which govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Internal standards ideally should have similar analytical behavior to the elements being determined.

3.1.5 Memory interferences - Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to 10 times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of 10 of the method detection limit, should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If a memory

interference is suspected, the sample should be reanalyzed after a long rinse period. In the determination of mercury, which suffers from severe memory effects, the addition of 100 µg/L gold has been shown to effectively rinse 5µg/L mercury in approximately two minutes. Higher concentrations may require a longer rinse time.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

4.1 For the determination of trace elements, contamination and loss are of prime concern. In this regard, the collection and treatment of the sample prior to analysis require particular attention. Laboratory glassware, including the sample bottle (whether polyethylene, polypropylene, or FEP-fluorocarbon), should be thoroughly washed with detergent and tap water and rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap water, and finally deionized water or certified clean by supplier or laboratory.

4.2 Before collection of an aqueous sample, a decision must be made regarding the type of metal analysis that is desired; that is dissolved, suspended, or total, so the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc. are to be performed at the time the sample is collected, or as soon as possible thereafter.

4.2.1 For the determination of dissolved elements, the sample must be filtered through 0.45µm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus is recommended to avoid possible contamination). Use the first 50 – 100 ml of sample to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH ≤ 2. Normally, 3 ml of (1+1) acid per liter should be sufficient to preserve the sample.

4.2.2 For the determination of suspended elements, a measured volume of unpreserved sample must be filtered through a 0.45µm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservation is required.

4.2.3 For the determination of total elements, the sample is preserved with (1+1) HNO_3 to a pH ≤ 2. Normally, 3 ml of (1+1) acid per liter should be sufficient to preserve the sample.

4.3 Soil samples should be placed in clean glass or plastic containers, normally 2-4oz jars.

4.4 Surface wipe samples should be placed in clean zip lock bags.

5.0 REAGENTS AND STANDARDS

5.1 All acids used in the preparation of samples and standards must be ultra high purity grade or equivalent.

5.2 Hydrogen Peroxide, 30%. Purchase commercially prepared solution.

5.3 Nitric Acid, concentrated.

5.4 Nitric Acid, (1+1): Add 500-ml concentrated HNO_3 to 500 ml deionized water.

5.5 Deionized water, 10 megaohm or greater.

5.6 Primary Multielement Stock Standard 1 (AESI-CAL-1). Purchased as certified solution from IV (Inorganic Ventures). Concentrations are as follows:

Table 5-1
Primary Multielement Stock Standard 1 Concentrations

Concentration (ug/mL)	**Metal Ion**
1000	Al, Ca, Fe, K, Mg, Na
100	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl, V, Zn
10	Ag

5.7 Primary Multielement Stock Standard 2 (VAR-CAL-1). Purchased as certified solution from IV. Concentrations are as follows:

Table 5-2
Primary Multielement Stock Standard 2 Concentrations

Concentration (ug/mL)	**Metal Ion**
100	Mo, Sb, Sn, Ti

5.8 Secondary Multielement Stock Standard. Purchased as certified solution from IV. Concentrations are as follows:

Table 5-3
Secondary Multielement Stock Standard 1 Concentrations

Concentration (ug/mL)	Metal Ion
1000	Al, Ca, Fe, K, Mg, Na
100	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Sb, Tl, V, Zn
10	Ag

Table 5-4
Secondary Multielement Stock Standard 2 Concentrations

Concentration (ug/mL)	Metal Ion
100	Mo, Sn, Ti

5.9 Stock Lithium Standard, 1000 mg/l. Purchased commercially.

5.10 Stock Cerium Standard, 1000 mg/l. Purchased commercially.

5.11 Stock Uranium Standard, 1000 mg/l. Purchased commercially.

5.12 Internal Standard (IS) Stock Solution. 100 ug/mL Li6, Sc, Y, In, Tb, Bi, Ga in 2% HNO3. Purchased certified solution from VHG.

5.13 Internal Standard Working Solution, 50 ug/mL. Prepare by diluting 0.5 mL of the IS stock solution and 20mL HNO3 to 1L with DI water.

5.14 Interferent Check Standard A (ICSA) Stock Solution. Purchased as certified solution from IV. Concentrations are as follows:

Table 5-5
ICSA Stock Standard Concentrations

Concentration (ug/mL)	Metal Ion
10,000	Cl
2,000	C
1,000	Al, Ca, Fe, K, Mg, Na, P, S
20	Mo, Ti

5.15 Interferent Check Standard B (ICSAB) Stock Solution. Purchased as certified solution from IV. Concentrations are as follows:

Table 5-6
ICSAB Stock Standard Concentrations

Concentration (ug/mL)	Metal Ion
2	Ag, As, Cd, Co, Cr, Cu, Mn, Ni, Zn

5.16 Initial calibration standards (ICAL).

First prepare the Metals Intermediate standard by using 1 mL of AESI-CAL-1, 1 mL of VAR-CAL-1, 0.2mL of HNO3, and dilute with DI water to a final volume of 10 mL.

Use the following to make the different levels of the ICAL:

200 ug/L: 2mL of AESI-CAL-1, 2 mL of VAR-CAL-1, 20 mL of HNO3 to a final volume of 1000mL with DI water.
100 ug/L: 1mL of AESI-CAL-1, 1 mL of VAR-CAL-1, 20 mL of HNO3 to a final volume of 1000mL with DI water.
10 ug/L: 1mL of Metals Intermediate, 20 mL of HNO3 to a final volume of 1000mL with DI water.
5 ug/L: 0.5mL of Metals Intermediate, 20 mL of HNO3 to a final volume of 1000mL with DI water.
1 ug/L: 0.1mL of Metals Intermediate, 20 mL of HNO3 to a final volume of 1000mL with DI water.

The resulting concentrations are as follows:

Table 5-7
Initial Calibration Concentrations

Concentration (µg/l)	Metal Ion
10, 100, 1000, 2000	Al, Ca, Fe, K, Mg, Na
1.0, 10, 100, 200	As, Ba, Be, Cd, Co, Cr, Cu, Mo, Mn, Ni, Pb, Se, Sb, Sn, Tl, Ti, V, Zn
0.1, 1.0, 10, 20	Ag

5.17 Initial calibration verification check standard (ICV). Prepare by diluting 1.0mL of the IV multi-element metals second source standard(s) and 20mL of Nitric Acid to 1000mL in a volumetric flask using deionized water. The resulting concentrations are as follows:

Table 5-8
ICV Concentrations

Concentration (µg/l)	Metal Ion
1000	Al, Ca, Fe, K, Mg, Na
100	As, Ba, Be, Cd, Co, Cr, Cu, Mo, Mn, Ni, Pb, Se, Sb, Sn, Ti, V, Tl, Zn
10	Ag

5.18 CCV Standard. Prepare by adding 1.0mL of each of the IV multi-element standards and 20mL of concentrated Nitric Acid to a 1000mL volumetric flask and diluting to volume with deionized water. The same standard (ICAL-100 ppb) is used for the CCV. The concentrations of the metals are as follows:

Table 5-9
CCV Concentrations

Concentration (ug/l)	Metal Ion
1000	Al, Ca, Fe, K, Mg, Na
100	As, Ba, Be, Cd, Co, Cr, Cu, Mo, Mn, Ni, Pb, Se, Sb, Sn, Ti, Tl, V, Zn
10	Ag

5.19 CRI Standard. The CRI standards are prepared from the same stock materials as the ICAL standards levels listed below. Other levels may be prepared if necessary to meet project specific PQL requirements if the prepared CRI Standards do not meet requested PQLs of specific analytes.

Table 5-10
CRI Concentrations

Concentration (µg/l)	Metal Ion
10, 50,100	Al, Ca, Mg, Fe, K, Na
1, 5,10	As, Ba, Be, Cd, Cr, Co, Cu, Ni, Mn, Mo, Pb, Se, Sb, Sn, Ti, Tl, Zn, V
0.1, 0.5, 1.0	Ag

5.20 Three types of blanks are required for the analysis. The calibration blank is used to establish the analytical curve, while the reagent blank (a.k.a. method blank) is used to assess possible contamination resulting from sample processing and to assess spectral background and the rinse blank is used to flush the instrument between samples when necessary to reduce memory interferences.

5.20.1 The calibration blank is prepared by diluting 20mL concentrated HNO_3 to 1000mL using deionized water.

5.20.2 The reagent blank (Method Blank) must contain all the regents, in the same volumes, used in processing the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration as the sample solution used for analysis.

5.20.3 The rinse blank is prepared by diluting 20mL concentrated HNO_3 to 1000mL (or 400 mL to 2000mL) using deionized water.

5.21 Tuning Intermediate Solution, 100,000 ug/L Ce, Li, U. Prepare by adding 1.0ml each of the Li, Ce and U stock standards and 0.2 ml of high purity concentrated HNO3 to a 10 ml round bottom graduated tube. Dilute to 10.0ml with DI water. A 2% blank rinse may be used to bring the solution to volume instead of the 0.2mL HNO_3/DI water.

5.22 Interferent Check Standards (ICSA/ICSAB). Two solutions are used for verification of interference correction equations as follows:

5.22.1 ICSA Working Standard. Prepare by adding 50.0ml of ICSA Stock Solution and 10ml high purity HNO3 to a 500ml volumetric flask and diluting to volume with DI water. The resulting concentration are as follows:

Table 5-11
ICSA Working Standard Concentrations

Concentration (µg/L)	Metal Ion
1,000,000	Cl
200,000	C
100,000	Al, Ca, Fe, K, Mg, Na, P, S
2000	Mo, Ti

5.22.2 ICSAB Working Standard. Prepare by adding 50.0 mL of 6020ICS-0A standard, 5.0 mL of 6020ICS-0B standard and 10 mL of high purity concentrated nitric acid to a 500 mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-12
ICSAB Working Standard Concentrations

Concentration (µg/L)	Metal Ion
1,000,000	Cl
200,000	C
100,000	Al, Ca, Fe, K, Mg, Na, P, S
2000	Mo, Ti
20	Ag, As, Cd, Co, Cr, Cu, Mn, Ni, Zn

5.23 Cross Calibration Solution. Prepare by diluting 3.0 mL of VAR-CAL-1 standard, 3.0 mL of the AESI-CAL-1 standard, 3.0 mL of the internal standard solution, 3.0 mL of the Tune intermediate standard and 20mL of concentrated high purity nitric acid to 1000mL in a volumetric flask using deionized water. The resulting concentrations are as follows:

Table 5-13
Cross Calibration Solution Concentrations

Concentration (µg/L)	Metal Ion
3000	Al, Ca, Fe, K, Mg, Na
300	Ce, U, ^{7}Li, Sb, Sn, Ti, Mo, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Tl, V, Zn, ^{6}Li, Sc, Tb, Y, Bi, In, Ga
30	Ag

5.24 Performance Report/Mass Calibration and Tuning Solution.
Prepare using 0.1 mL of Tuning Intermediate Solution (5.21), 0.1mL of AESI-CAL-1 (5.6), VAR-CAL-1 (5.7), Internal Standard (IS) Stock Solution (5.12), 20 mL of high purity HNO_3, and dilute to a final volume of 1000mL with DI water.

The resulting concentrations are as follows:

Concentration (µg/L)	Metal Ion
100	Al, Ca, Fe, K, Mg, Na
10	Ce, U, ^{7}Li, Sb, Sn, Ti, Mo, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Tl, V, Zn, ^{6}Li, Sc, Tb, Y, Bi, In, Ga
1	Ag

5.25 Vendor List. The standards used in this test are purchased using the catalog numbers and vendors indicated below.

Table 5-14
ICPMS Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
Hydrochloric Acid	Fisher	Concentrated	A144C -212
Nitric Acid	EMD	Concentrated	NX040712
Primary Multielement Standard 1	IV	Varied	AESI-CAL-1
Primary Multielement Standard 2	IV	Varied	VAR-CAL-1
Ce Stock Standard	RICCA	1000 mg/L	PCE1KN-100
Li Stock Standard	VHG	1000 mg/L	PK1N-100

U Stock Standard	RICCA	1000 mg/L	PU1KN-100
Internal Standard	VHG	100 mg/L	LIS1-100
ICSA	Inorganic Ventures	Varied	6020ICS-0A
ICSAB	Inorganic Ventures	Varied	6020ICS-0B
Secondary Multi. Standard 1	IV	Varied	AESI-CAL-1QC
Secondary Multi. Standard 2	IV	100 ug/mL	VAR-QC-1

6.0 APPARATUS AND MATERIALS

6.1 Inductively Coupled Plasma- Mass Spectrometer system: Thermo Electron Model X7 with autosampler, data system and Plasmalab software or equivalent.

6.1.1 Note: Instrument capable of scanning the mass range 5-250 amu with a minimum resolution capability of 1 amu peak width at 5% peak height. Since an electron multiplier is being used, precautions should be taken to prevent exposure to high ion flux. Otherwise changes in instrument response or damage to the multiplier may result.

6.2 Argon gas supply - welding grade or better.

6.3 Block digestor

6.4 Plastic digestion vessels - 70-ml disposable with graduations.

6.5 Pipettes – various volumes

6.6 Disposable syringe filtration apparatus including 0.45um disposable filters.

7.0 PROCEDURE

7.1 Preparation of digestion log form and digestion log in LIMS.

7.1.1 Each day the section supervisor prepares a work log. The log lists samples included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN DIGESTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received with a “rush” status will have a chain of custody delivered to the prep department by sample receiving.

7.1.1.2 Prepare a written digestion log using the metals digestion logbook that is kept in the digestion area of the laboratory. The following entries must be made in the log.

7.1.1.2.1 Date the batch is opened or the date and time the sample(s) is placed on the hot plate.

7.1.1.2.2 All samples included in the digestion batch.

7.1.1.2.3 Volume or weight of samples digested.

7.1.1.2.4 Date the digestion is completed.

7.1.1.2.5 Digestion procedure employed.

7.1.1.2.6 The initials of the digestion analyst(s).

7.1.1.2.7 Laboratory number of all reagents used, including spiking standard and acids.

7.1.1.2.8 Volume of all reagents used, including spiking standard and acids.

7.1.1.2.9 Final volume of all digestates.

7.1.1.2.10 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.1.1.3.1 Open a Prep Batch in LIMS by double clicking the "Prep" box.

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS.

7.1.1.3.3 Select the Prep Code "3005A" or other appropriate digestion code from the pull down list. The LIMS will automatically assign a MB and LCS sample to the prep list. If an LCSD is desired, click on the blank space below and enter the information.

7.1.1.3.4 Select the technician's name from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this method. Select the samples to be included in the batch for desired prep method.

7.1.1.3.6 The samples can be individually selected by highlighting the sample and clicking the "→" arrow or all samples can be selected by clicking the "⇒" arrow.

7.1.1.3.7 Samples that have been selected for re-analysis can be manually added to the sample list following the procedure outlined in section 7.1.1.3.3.

7.1.1.3.8 "Save" the batch by clicking on a previous batch number on the list and then return to the newly created batch.

7.1.2 Table 7-1 indicates the type of samples that comprise an analytical batch. Note: NELAC requirements specify that the maximum number of client samples in a prep batch can not exceed 20. Further, a prep batch can not be left "open" for a period that exceeds 24 hours.

Table 7-1

Samples in a NELAC Batch
Method Blank (MB)
LCS (and LCSD if no MSD)
MS and MSD (If supplied by client)
Dilution Test Sample
Post Digestion Spike Sample
Client Samples(up to 20 per batch)

This method requires the analysis of additional samples not required by NELAC. See the information in Section 7.3, Run Sequence.

7.2 Sample preparation

7.2.1 For the determination of dissolved elements see Table 7-4 SAMP_FILT prep checklist.

7.2.2 For determination of total elements in water samples see Table 7-5 Method 3005A Sample Prep Checklist.

7.2.3 For determination of total elements in soil samples see Table 7-6 Method 3050B Sample Prep Checklist.

7.2.4 For the determination of Total Vanadium in soil samples see Table 7-7 Method 3050_B_S_V_6020 Sample Prep Checklist.

7.2.5 For determination of total elements on wipe samples see Table 7-8 Method Wipe_MET_MS_P(N7300) Sample Prep Checklist.

7.3 Instrument Start-up

7.3.1 Turn on the hood above the ICP.

7.3.2 Check to insure that the waste bottles are empty and the rinse and internal standard bottle is full.

7.3.3 Make sure the gas pressure on the regulator is 90 psi. Make sure that there is sufficient gas to complete the analytical run.

7.3.4 Check to see if the peristaltic pump tubing is in good condition and tubing is clamped. Pump tubing is replaced at least every other day of operation.

7.3.5 Ensure Faraday cage is in its forward position and that the Faraday cage door is closed.

7.3.6 Open the Plasmalab program by double clicking on the Plasmslab icon.

7.3.7 Check the instrument status window, it should read "**Vacuum Ready**".

7.3.8 Click on the **ON** button at the top of the window.

7.3.9 After start up is complete, the status window should read "**Operate**".

7.3.10 Click on the **INSTRUMENT** icon to the left of the Plasmalab window.

7.3.11 Under Configuration Editor Tab, select "**Accessories**".

7.3.12 Select "**Autosampler**" tab.

7.3.13 Place both sample probe and internal tubing in DI water or 2% HNO_3.

7.3.14 Allow at least 30 minutes warm up time.

7.4 Instrument Tuning and Mass Calibration

7.4.1 Initially and whenever performance criteria in Section 7.6 fails to meet specifications, the instrument must be retuned. Retuning is accomplished by using the Autotune function and, when necessary, the mass calibration routine.

7.4.2 Autotuning is performed as follows:

7.4.2.1 Select the **Instrument** icon on the far left panel.

7.4.2.2 Select the **Tune** tab.

7.4.2.3 Select the **Start the Autotune Wizard** icon (musical notes).

7.4.2.4 When the dialogue comes up select **Next**.

7.4.2.5 Select **IQ Xi Interface** from the **Available Sequences** box and click **Next**.

7.4.2.6 Under **Stage Ordering**, highlight **Nebulizer: iteration 1** and **Sweeps 60**.

7.4.2.7 Under **Sample Input** option, select **I want to introduce the sample manually**, then press **Next**.

7.4.2.8 Place the sample probe in the Tune solution and select **Next**.

7.4.2.9 Wait for uptake delay to complete (45 seconds). (Optional)

7.4.2.10 Save new instrument settings as "**TUNE_YYMMDD[A,B,D,etc.]**".

7.4.2.11 Run another Performance Report as follows:

7.4.2.11.1 Select the **Instrument** icon on the far left panel.

7.4.2.11.2 Select the **Tune** tab.

7.4.2.11.3 Select the **Start the Performance Report Wizard** icon (musical notes on a piece of paper).

7.4.2.11.4 When the Performance Report Acquisition Report dialogue box opens select **Next**.

7.4.2.11.5 The next heading is **Select a Performance Report**. Under **Available Reports** select **Performance070413** then click **Next**.

7.4.2.11.6 Under **Sample Input Option** select the first option of "**I want to introduce the sample manually**" then press **Next**.

7.4.2.11.7 Place sample probe in the Tune solution and select **Next**.

7.4.2.11.8 Wait for uptake delay to complete (45 seconds). (Optional)

7.4.2.11.9 Save as **Performance Report_YYMMDD[A,B,C,etc.].**

7.4.2.12 **If after two attempts to Autotune the instrument, criteria are still failing, do not continue until after instrument maintenance and/or service is completed.**

7.4.3 Mass Calibration is performed as follows:

7.4.3.1 Select the **Instrument** icon on the far left panel.

7.4.3.2 Select the **Tune** tab.

7.4.3.3 Select the drop down menu arrow next to the **Launch The Instrument Wizard** icon (clipboard with a check mark).

7.4.3.4 Select **Mass Calibrate the Quadrupole**.

7.4.3.5 When the dialogue box opens select **Next**.

7.4.3.6 Under **Samples Input Options** select **I want to introduce the sample manually** then press **Next**.

7.4.3.7 Place the sample probe in the Tune solution and select **Next.**

7.4.3.8 Wait for uptake delay to complete (45 seconds). (Optional)

7.4.3.9 If Mass Calibration fails as indicated by error message on the screen, the **Standard Resolution** must be adjusted as follows:

7.4.3.9.1 Click on the **Instrument** icon and then the **Tune** tab.

7.4.3.9.2 Select the **Global** tab.

7.4.3.9.3 Using the **Standard Resolution slider**, **increase the Standard Resolution to decrease the peak width** or **decrease the Standard Resolution to increase the peak width**.

7.4.3.10 After resolution adjustments have been made, repeat steps 7.4.3.1 through 7.4.3.8.

7.4.3.11 **If after two attempts to mass calibrate the instrument, criteria are still failing, do not continue until after instrument maintenance and/or service is completed.**

7.5 Detector Cross Calibration

7.5.1 The analogue and pulse counting detectors must be cross calibrated such that both are providing similar results for all masses being reported.

7.5.2 When criteria described in Section 7.6.10 indicate the need for recalibration of the detectors the following procedure is used:

7.5.2.1 Select the **Launch the instrument wizard** icon (clipboard with a check mark).

7.5.2.2 Select **Detector Cross Calibration** then click **Next**.

7.5.2.3 Under **Samples Input Options** select "**I want to introduce the sample manually**" the press **Next**.

7.5.2.4 Place the sample probe in the Cross Cal Standard solution.

7.5.2.5 Select Next and wait for uptake delay to complete (45 seconds).

7.5.2.6 Save new Cross Cal settings as **"Cross Cal YYMMDD[A,B,C,etc.]".**

7.5.2.7 If detector cross cal fails as indicated by error message on the screen, detector voltages must be adjusted as follows:

7.5.2.7.1 Select the **Launch the Instrument Wizard** icon (clipboard with check mark).

7.5.2.7.2 Select **Voltage Setup** and **Detector Cross Calibration** then click **Next**.

7.5.2.7.3 Under Samples Input Options select "**I want to introduce the sample manually**" then click **Next**.

7.5.2.7.4 Place sample probe in the Cross Calibration Solution.

7.5.2.7.5 Select **Next** and wait for uptake delay to complete (45 seconds).

7.5.2.7.6 Save the new detector voltage settings as **"Detector Voltage Setup YYMMDD[A,B,C,etc.]".**

7.5.2.8 Rerun the Short Term Stability and Performance Verification procedure per Section 7.6 to verify acceptability of new settings.

7.5.2.9 If after two attempts to Cross Calibrate the detectors, criteria are still failing, do not continue until after instrument maintenance and/or service is completed.

7.6 Short Term Stability and Performance Verification

7.6.1 Place the autosampler probe and the internal standard tubing into the Tune solution vial.

7.6.2 Under **Instrument** go to the **Configuration** tab and **de-select Autosampler**.

7.6.3 Go to the Desktop and select the STS 200.8 template.

7.6.4 Select the **Sample List** tab and click on **Instrument Setup**.

7.6.5 Select **Show Advanced,** then click on the **Instrument Performance Tests** tab.

7.6.6 Under **Set Up** tab, select **Xi Interface**.

7.6.7 Click on **Q** to start analysis and save as **STS YYMMDD[A,B,C,etc.]** then click **Append**, then **OK**.

7.6.8 After analysis is complete, go to the **Results** tab, then the **Spectra** tab.

7.6.9 Double click on **Tune** in the Spectra window.

7.6.10 Click on the **Magnifying Glass** icon and go over the spectra at masses 6 for Li, 24 for Mg, 115 for In and 206 for Pb.

7.6.10.1 The spectra from the analogue (dotted line) and pulse counting (solid line) detectors should completely, or very nearly, overlap. If they do not, the detectors will require recalibration by performing a new Detector Cross Calibration per Section 7.5.

7.6.11 Once the run has completed, select the boxes for **Results, Numerical Result** and **Mass Uncorrected ICPS**.

7.6.12 Go to the **Reports** tab and select **Performance Reports**. Performance criteria must be met as follows:

7.6.12.1 Mass resolution is evaluated using peak width at 5% of maximum peak height. Peak width must be 0.75 $\pm$0.1 amu for Be9, Mg24, Mg25, Mg26, In115, Pb206, Pb207 and Pb208.

7.6.12.2 Instrument Stability is evaluated using %RSD of the absolute responses for specific Tune Solution components. %RSD between the five measurements must be ≤5% for Be9, Mg24, Mg25, Mg26, Co59, In115, Pb206, Pb207 and Pb208.

7.6.12.3 Instrument Sensitivity is evaluated using minimum Counts/second (CPS) measured for specific Tune Solution components. Mean CPS values must be >1000 for Be9, Mg24, Mg25, Mg26, Co59, Pb206, Pb207 and Pb208, and >70000 for In115.

7.6.12.4 Oxide and polyatomic ion formations are evaluated using mass ratios for Ce156/Ce140 and Ce++140/Ce140. Ce156/Ce140 ratio must be <0.02 and the Ce++140/Ce140 ratio must be <0.03.

7.6.13 If all specified Tune Evaluation criteria are met, the **Performance Report** will list the statement **"Result: the performance report has passed"** and calibration and analysis may begin.

7.6.14 If any of the specified Tune Evaluation criteria fail specifications, instrument settings must be adjusted before beginning calibration and sample analysis as follows:

7.6.14.1 If failure is due to peak resolutions outside specified limits, Mass Calibration must be performed per Section 7.4.3.

7.6.14.2 If failure is due to stability, sensitivity or ratio criteria outside specified limits, Autotune must be performed per Section 7.4.2.

7.6.15 Once all criteria are met, click the **Refresh** icon and print the Performance Report as an ADOBE document named **"Performance Report_[YYMMDD[A,B,C,etc]]**". Save the Performance Report with the Raw Data in the Portal Server.

7.7 Initial Calibration Description and Criteria

7.7.1 As the initial part of the Normal Run Sequence described in Section 7.8 and Table 7-3 and after all tuning criteria have been met, Initial Calibration (ICAL) curves must be constructed for each target metal. Initial calibration is performed daily or whenever initial (ICV) or continuing (CCV) calibration verification standards and/or blanks fail to meet method specified criteria.

7.7.2 Initial Calibration is performed using internal standard techniques, calibration blanks and calibration standards at 4 levels for each target metal. Internal standards are introduced via in-line injection using a second channel of the sample introduction peristaltic pump. Specific calibration criteria are as follows:

7.7.2.1 All curve fits are Linear Regression through the blank with no weighting and interference correction on for all target metals. Correlation Coefficient must be ≥0.998 before sample analysis may begin. See Section 8 for corrective action requirements.

7.7.3 Initial Calibration Verification (ICV). All initial calibration curves must be verified by analyzing a second source standard for each target metal. All recoveries must be within ±10% before sample analysis may begin. See Section 8 for corrective action requirements.

7.7.4 Initial Calibration Blank (ICB). Absence of carryover after analysis of the calibration and ICV standards must be verified by analyzing the Calibration Blank solution . Values for all target metals must be <PQL before sample analysis may begin. See Section 8 for corrective action requirements.

7.7.5 Low-Level Initial/Continuing Calibration Verification standard (LLICV/LLCCV or CRI). The LLICV/LLCCV or CRI standard should be made at the established lower limit of quantitation and analyzed at the end of each analysis batch with closing CCB/CCV. A more frequent analysis may be necessary to avoid re-analysis should the LLCCV fail if only analyzed at the end of the analysis batch. The acceptance criteria for the LLCCV standard must be ±30%.

7.7.6 Interference Correction Verification (ICSA/ICSAB). ICSA and ICSAB must be analyzed at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Verification of the interference correction equations is accomplished by the analysis of the two interference check standards as follows:

7.7.6.1 ICSA solution is analyzed to verify the absence of positive or negative interference at the PQL level. This solution contains only the potentially interfering elements. The absolute value of the results for all target metals not included as interferents must be <PQL. See Section 8 for corrective action requirements.

7.7.6.2 ICSAB solution is analyzed to verify the absence of positive or negative interference with target analyte present. This solution contains potentially interfering elements plus specific target analytes. Percent recovery for all spiked target metals not included as interferents must be ±20% before sample analysis may begin. See Section 8 for corrective action requirements.

7.7.7 Continuing Calibration Verification (CCV). Calibration curves must be verified throughout the run sequence by analyzing CCV standards at least every 10 samples and at the end of the sequence. All target metals must recover within 90-110% of the expected values or the sequence must be stopped, new calibration curves obtained and all samples back to the last passing CCV

reanalyzed.

7.7.8 Table 7-2 below lists the masses used for quantitation and estimated detection limits for the masses used.

Table 7-2
Element Masses and Estimated Method Detection Limits

Element	**Mass**	**Estimated Detection Limit µg/L**
Aluminum	27	4.75
Arsenic	75	0.93
Antimony	123	0.14
Barium	137	0.10
Beryllium	9	0.14
Cadmium	111	0.11
Calcium	43	94
Chromium	52	0.14
Cobalt	59	0.04
Copper	65	0.18
Iron	56	3.35
Lead	208	0.03
Magnesium	24	0.50
Manganese	55	0.28
Molybdenum	98	0.11
Nickel	60	0.28
Potassium	39	56
Selenium	82	0.63
Silver	107	0.01
Sodium	23	28
Thallium	205	0.02
Vanadium	50	0.29
Tin	118	0.53
Zinc	66	1.81

7.8 Starting the Analytical Sequence/Run

7.8.1 Once all tuning criteria have been met, the instrument sequence/run is then set up.

7.8.2 Place the sample probe into the autosampler arm and the internal standard probe into the internal standard solution.

7.8.3 Go to the **Instrument** page and select the **Configuration** tab.

7.8.4 Under **Available Accessories and Devices** select the box for **Autosampler** to turn it back on.

7.8.5 On the same page under **ACL Script** select **Fast Uptake Wash** from the drop down menu.

7.8.6 Click on the Template on the Desktop **6020new**.

7.8.7 The experiment should open to the **Configuration Editor** page under the **Setup Tab.**

7.8.8 At the bottom of the page under **Instrument Settings** select the most recent **Autotune-IQ Xi Interface** file.

7.8.9 Go to the Sample List tab and type in the samples to be analyzed in order. The standard sequence order will be as follows:

Note: Order numbers in Table 7-3 do not necessarily represent Sequence Table positions, they only represent the order of analysis. Rinse blanks may be inserted as deemed necessary by analyst. Also, items 1-11 will already be present in the sequence template and will not need to be typed in.

ICAL (steps 1-11) may be run first, then evaluated and, if passing, then samples added and analyzed if desired.

Table 7-3

EXAMPLE RUN SEQUENCE

1. ICAL Blank
2. ICAL Std 1
3. ICAL Std 2
4. ICAL Std 3
5. ICAL Std 4
6. ICV(s)
7. ICB
8. LLICV/CRI 1.0ppb (additional CRIs may be added if needed)
9. LLICV/CRI 5.0ppb
10. ICSA(Repeat at least every 12 hours)
11. ICSAB (Repeat at least every 12 hours)**(DO NOT PROCEDE TO STEP 12 UNLESS ALL ICAL CRITERIA PER 7.8 HAVE BEEN MET)**
12. Up to10 Batch QC and/or client samples
13. CCV
14. LLCCV (CRI 1.0ppb/5 ppb; see #8&9)

15. CCB
16. Up to10 Batch QC and/or client samples
17. CCV
18. LLCCV (CRI 1.0ppb/5 ppb; see #8&9)
19. CCB
20. Repeat 14-16 as needed
21. Last set of instrument QC must include CCV/CCB/LLCCV (CRI 1.0ppb/5 ppb; see #8&9)

7.8.10 CCVs, CRI, and CCBs must be inserted at the proper intervals by clicking on the grey box in front of the 11th sample (and every 11th sample thereafter) then right click and select **Insert Before** 3 times to insert 3 lines.

7.8.11 Rinse included before CCV/CCB, RINSE-Rack-0, Row-1, Column-6, height-150.

7.8.12 Type in the table information for CCV as Label-**CCV**, Rack-**0,** Row-**1,** Column-**4,** Height-**150.**

7.8.13 Type in the table information for CCB as Label-**CCB**, Rack-**0,** Row-**1,** Column-**1,** Height-**150.**

7.8.14 Double check all Labels, positions, heights and number of main rows.

7.8.15 Go to the **Results** page then **Numerical Results** then select **Refresh** to save the sample list changes.

7.8.16 Print a hard copy of the sample list by going to **Reports**, then check the box to select **Sample List**, then click the **Refresh** icon. The sample list will be displayed in a printable format, click the **Print** icon to print.

7.8.17 **ALL SAMPLE LISTS MUST BE CHECKED AGAINST THE SAMPLES AS LOADED IN THE AUTOSAMPLER BY A SECOND ANALYST TO VERIFY NO SAMPLE SWITCHING AT LOADING BEFORE SAMPLES ARE ANALYZED.**

7.8.18 Once the sample list is verified correct, the run is started by clicking on the **Q** icon, then enter the requested save file name as **"YYMMDD[A,B,C,etc]",** click **OK**, then **Append**, then **OK** again.

7.8.19 Analyst should monitor the QC samples as they run and stop the run at any point where CCV/CCB fails to meet the required criteria.

Table 7-4
SAMP_FILT (For Dissolved Metals)

____ **:** PRINT (check) BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(SAMP_FILT, 6010B_W_D, 6010B_B_W_D, 6010B_PO4_W_D, 6010B_Sr_W_D, 6010B_TAL_W_D, 200.7_W_D, 200.7_B_W_D, 200.7_PO4_W_D, 200.7_Si_W_D, 200.7_Sr_W_D, 200.8_D, 6020_W_D, 6020_A1_W_D, 6020_A2_W_D, 6020_A9_W_D, 245.1_W_D)

____ : PICK A SAMPLE FOR QC(MS&MSD) (homogeneous is ideal)

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add MS/MSD, check volumes), PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO. Write samples in the log book from the back log.

____ : FILTER 50 ml SAMPLE THROUGH 0.45 µm SYRINGE FILTERS TO DIGESTION VESSEL. (IF THE SAMPLE HAS A BAD MATRIX WHICH MAKES IT DIFFICULT TO FILTER, YOU CAN REDUCE THE VOLUME FILTERED to 25ml)

____ **IF SAMPLE IS CLOUDY OR SEDIMENT IS PRESENT AFTER FILTRATION, OR IF SAMPLES CANNOT BE SYRINGE FILTERED DUE TO HIGH SOLIDS CONTENT, SEE SUPERVISOR BEFORE CONTINUING. DOCUMENT ALL SAMPLING DIFFICULTIES IN THE LOG BOOK COMMENTS.**

____ : FILTER 50 ml SAMPLE TO MS AND MSD VESSELS FOR THE QC SAMPLE.

____ : FILTER 50 ml of DI WATER THROUGH SYRINGE FILTERS to LCS & MB VESSELS.

FOR ALL 200.8 AND 6020 TEST CODE PREPS:

____ : ADD 1.0 ml OF **OMNITRACE HNO_3/ 50ml sample** TO EACH SAMPLE AND QC AND SHAKE WELL.

____ : SPIKE LCS, MS & MSD with 0.5ml of **10** ppm multi-element std and/or single element std as required by select list (WITNESS NEEDED). **SHAKE WELL**

FOR ALL 200.7 AND 6010 TEST CODE PREPS:

____ : ADD 0.5 ml OF HNO_3 **AND** 0.5 ml HCL/ **50ml sample** TO EACH SAMPLE AND QC AND SHAKE WELL.

____ : SPIKE LCS, MS & MSD with 0.5ml of **100** ppm multi-element std and/or single element std as required by select list (WITNESS NEEDED). **SHAKE WELL**

IF THE SAMPLES ARE FIELD FILTERED:

____ : ADD 50 ml SAMPLE TO DIGESTION VESSEL.

____ : ADD 50 ml SAMPLE TO MS AND MSD VESSELS FOR THE QC SAMPLE.

____ : ADD 50 ml Filtered DI WATER TO LCS & MB VESSELS EACH. MB AND LCS MUST BE FILTERED PRIOR TO SPIKING.

____ : ACIDIFY FOR EITHER 200.7/6010 TEST CODES OR 200.8/6020 TEST CODES AS ABOVE

____ : SPIKE LCS, MS & MSD FOR EITHER 200.7/6010 TEST CODES OR 200.8/6020 TEST CODES AS ABOVE

____ : ******SHAKE WELL******

____ : LINE UP SAMPLES IN ORDER THAT THEY APPEAR IN LOG

Table 7-5
3005A

____ **:** PRINT (check) BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(200.8, 200.8_PP, 6020B_W, 6020B_A1_W, 6020B_A2_W, 6020B_A9_W)

____ : PICK A SAMPLE FOR QC (homogeneous is ideal)

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO
(QC, then order of backlog) Write samples in the log book from the back log.

____ : **MIX SAMPLE THOROUGHLY**, ADD SAMPLE TO DIGESTION VESSEL AND RECORD VOLUME.
If the does not look like drinking water, then evaluate the sample matrix.
-Pour 1ml into test tube and add 1ml DI H2O to check water solubility.
-Add 1ml HNO3 to check reaction. Some samples may need reduced volume.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add MS & MSD, check weight. All 6020_S batches will need DL & PDS. RECORD THE WEIGHT OF THE ORIGINAL SAMPLE FROM LIMS) PRINT A COPY OF THE BATCH

____ : ADD 50 ML of DI WATER to LCS & MB VESSELS from DI wash bottle.

____ : SPIKE LCS/MS/MSD with 0.5ml of 100ppm multi-element standard. (WITNESS NEEDED)

____ : ADD 1.0 ml HNO_3, cover the vessels with watch glasses and heat at 95 $\pm 2^{o}C$ till volume reduces to ~30 ml.
(Use Omnitrace Nitric Acid from metals lab)

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50-ML

____ : ***SHAKE WELL***

____ : LINE UP SAMPLES IN ORDER THAT THEY APPEAR IN LOG

____ : CHECK THE ORDER OF THE SAMPLES IN THE BATCH

____ : CHECK THE ELEMENTS TO BE ANALYZED

____ : TYPE SAMPLES INTO COMPUTER AND POUR TRAY

____ : CHECK THE ORDER, LAST POSITION ON TRAY

Table 7-6
3050B_S_6020

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3050B_S_6020, 6020_S)

____ : PICK A SAMPLE FOR QC (ORG, MS, AND MSD) HOMOGENIZE SAMPLE

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO (QC, then order of backlog)

____ : ADD SAMPLE TO DIGESTION VESSEL AND RECORD WEIGHT (1.0-2.0g)

For the sample chosen for MS/MSD, the initial weights used for the unspiked sample, matrix spike, and matrix spike duplicate must be within 5% of each other.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add MS & MSD, check weight. All 3050B_S batches will need DL & PDS. RECORD THE WEIGHT OF THE ORIGINAL SAMPLE FROM LIMS) PRINT A COPY OF THE BATCH

____ : SPIKE LCS/MS/MSD with 0.5ml of **low level multi-element std** (same as 3005). (WITNESS NEEDED)

____ : ADD 2.0 ML HNO_3, COVER THE VESSELS WITH WATCH GLASSES, THEN HEAT AT 95 $\pm 2^{o}C$ FOR 10 MIN

____ : ADD 4.0 ML HNO_3, THEN HEAT FOR 30 MIN

____ : REMOVE from heat add 2-3ml DI H_2O, let cool, ADD 1 DROPPER OF H_2O_2, and HEAT FOR 10 MIN

____ : ADD 5 ML HCL, HEAT UNTIL VOLUME drops to 5 ml or 1 HOUR

____ : ***SHAKE WELL***

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50 ML

____ **:** Does it have BACKLOG REPORT FOR THE PREP AND EACH TEST CODE

____ : Is the SAMPLE FOR QC (dup, ms) adequate

____ : ARE the SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : Check SAMPLE final volume in DIGESTION VESSEL, does it match Recorded VOLUME

____ : CHECK prep log against BATCH IN THE LIMS (sample prep, test code, date time, analysts, check sample ID's, OK, add DUP & MS, check volumes), or Printed A COPY OF THE BATCH

Table 7-7
3050B_S_V_6020

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3050B_S_V_6020, 6020_S_V)

____ : PICK A SAMPLE FOR QC (org., ms, and msd) HOMOGENIZE SAMPLE

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO (QC, then order of backlog)

____ : ADD SAMPLE TO DIGESTION VESSEL AND RECORD WEIGHT (1.0-2.0g)

For the sample chosen for MS/MSD, the initial weights used for the unspiked sample, matrix spike, and matrix spike duplicate must be within 5% of each other.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, test code, date time, analysts "you", user select, single arrow samples into selected column, check sample ID's, OK, add MS & MSD, check weight. All 3050B_S batches will need DL & PDS. RECORD THE WEIGHT OF THE ORIGINAL SAMPLE FROM LIMS) PRINT A COPY OF THE BATCH

____ : SPIKE LCS & MS/MSD with 0.5ml of **low level multi-element std** (same as 3005). (WITNESS NEEDED)

____ : ADD 2.0 ML HNO_3, COVER THE VESSELS WITH WATCH GLASSES, THEN HEAT AT 95 $\pm 2^{o}C$ FOR 10 MIN

____ : ADD 4.0 ML HNO_3, THEN HEAT FOR 30 MIN

____ : ***SHAKE WELL***

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50 ML

____ **:** Does it have BACKLOG REPORT FOR THE PREP AND EACH TEST CODE

____ : Is the SAMPLE FOR QC (ms, msd) adequate

____ : ARE the SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : Check SAMPLE final volume in DIGESTION VESSEL, does it match Recorded VOLUME

____ : CHECK prep log against BATCH IN THE LIMS (sample prep, test code, date time, analysts, check sample ID's, OK, add MS & MSD, check volumes), or a Printed COPY OF THE BATCH

Table 7-8
WIPE_MET_MS_P (N7300)

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (6020_Wipe)

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : BATCH SAMPLES IN THE LIMS, PRINT BATCH

____ : PREPARE LCS & LCSD (WITNESS NEEDED) BY USING A BLANK GHOST WIPE AND ADDING 0.50ml OF 6020 MULTIELEMENT SPIKE SOLUTION.

____ : ADD 2.0 ML OF CONC. HNO_3. HEAT IN HOT BLOCK AT 95 $\pm 2^oC$ FOR 3-5 MINUTES.

____ : ADD 5.0-10.0 ML CONC. HNO_3. HEAT FOR 5 MINUTES. (IT DEPENDS ON THE SIZE AND TYPE OF THE WIPE AS HOW MUCH CONC. HNO_3 IS ADDED).

____ :REMOVE FROM HEAT AND ALLOW TO COOL.

____ : ADD ~ 1 DROPPPER OF 30% H2O2. WIPE MUST BE COMPLETELY COVERED WITH DIGESTION SOLUTION; ADD DI WATER IF NECESSARY.

____ : HEAT FOR 5 MIN FOR GHOST WIPES, OR UNTIL SAMPLE HAS THE CONSISTENCY OF MASHED POTATOES.

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50 ML.

____ : ***SHAKE WELL***

Table 7-9

DATA REVIEW CHEKLIST- ICP/ MS

Batch ID: _____ Data File: _____

QA Analyst

INITIAL RAW DATA REVIEW

____ ____: Have raw data, tune reports, performance reports and run log(s) been printed to pdf and posted to Portal Server
____ ____: Are all instruments standards used listed on raw data
____ ____: Are all instrument performance criteria met? **No requires retuning or other action and reanalysis**
____ ____: Do calibration curves for all target analytes meet requirements (≥0.998 Corr.Coef)
NA ____: Are all internal standards within method specified limits? **No requires action as per each circumstance**
____ ____: Are all replicate RSDs ≤ 15% for all values over PQL? **No requires reanalysis and CAR if still out at reanalysis**

GO TO LIMS "MAIN" RUN SCREEN

____ ____: Are any CARs listed in comments box **(Yes requires determining possible actions before continuing)**
____ ____: Are all Sample IDs properly assigned per Backlog Report (Double click each SampleID to verify)
____ ____: Are all Test Codes properly assigned per Backlog Report
____ ____: Is instrument QC run at the required frequencies
____ ____: Are all Sample Types properly assigned
____ ____: Are all samples linked to the Prep Batch properly with correct PFac, SpkFac and OFac
____ ____: Have sample ID's been recorded into the logbook and do they match exactly the IDs in the LIMS
____ ____: Are all initial and final volumes recorded into the logbook and do they match the initial and final volumes in LIMS
____ ____: Did the analyst add a narrative as to why initial and / or final volumes were modified ?
Narrative for elevated RLs due to limited sample volume or reduced volume prepped due to matrix
____ ____: Are dilution factors entered correctly per the raw data Run Log
____ ____: Are all Blkref, SPKref, RPDref and CCVref assigned correctly
____ ____: Are there any Comments present that require attention? **May require CAR**

GO TO DATA SCREEN

____ ____: Calculate Sequence (Cal SEQ tab) to insure LIMS calculations are completed.
____ ____: Are there any S or B flags for target analytes on the Instrument QC (ICV, CRI,CCV,etc. **Yes Requires CAR**
____ ____: Are all values for target analytes between <PQL and > -PQL on ICB, CCB and MBs? **No Requires CAR**
____ ____: Are all values for targets analytes (except interferents) between<PQL and >-PQL on ICSA? **No requires CAR**
____ ____: Are there any S flags for element of interest on any samples and/or batch QC? **Yes Requires CAR**
____ ____: Are there any B flags for elements of interest on any samples and/or Batch QC? **Yes Requires CAR**
____ ____: Are there any S and/or R flags for elements of interest on Batch QC (LCS/D, MS/D, DL, PDS)? **Yes Requires CAR except R or S flags on DL or PDS cause by value at or near PQL or insignificant spike.**
____ ____: Are there any H flags present for any samples? **Yes Requires CAR**
____ ____: Are there any E flags for elements of interest on any samples and/or Batch QC selected for reporting? **Yes Requires CAR**
____ ____: Are there any J flags on any target analytes selected to report on diluted sample runs? **Yes Requires reanalysis at lower dilution or CAR to narrate elevated reporting limits**
____ ____: Are there "*" qualifiers for J flagged target analytes? **Yes requires removal of "*" qualifiers**
____ ____ Have readings been entered properly (check at least 2 entries for downloaded data, **ALL** entries for manual data)
____ ____: Have at least 2 sample's final results in LIMS been verified by hand calculation
____ NA: Have **ALL** CARs been closed and narratives written prior to QA?
____ NA: Have PM, Lab Manager and PM Director been notified for any CAR resulting in reextract and/or due date exceedance?

CAR#: _____ _____ _____ _____

Analyst Signature: _____ Date: _____ Time: _____

Reviewer Signature: _____ Date: _____ Time: _____

Table 7-9 (cont)

DILUTIONS

Sample ID	Metal(s) Needed	Done
		YES
		YES
		YES
		YES
		YES
		YES
		YES
		YES
		YES
		YES

REANALYSIS

Sample ID	Metals Needed	Done
		YES
		YES
		YES
		YES
		YES
		YES
		YES
		YES
		YES

Comments:

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP# QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Method Detection Limit Study (MDL). The method detection limit is calculated by analyzing at least seven replicates prepared and analyzed at concentrations at or near the lowest reporting limit to be used for samples. Quantitation limits are laboratory derived from the MDL study data set and project specific requirements. All PQLs must be >MDLs. MDLs are to be performed annually or whenever instrument conditions have changes that will affect the established detection limits. Current MDLs and PQLs can be found in LIMS

8.1.3 Lower Limit of Quantitation Check Sample (LLQC). The Lower Limit of Quantitation Check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard (LLCCV) should be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within $\pm$30% of their true value. This check should be used to both establish and confirm the lower quantitation limit

8.1.4 Instrument Detection Limits (IDL). IDLs are verified quarterly by analyzing reagent blank samples for seven consecutive measurements each day for three non-consecutive days. Standard deviations for each day are calculated with final IDLs equal to 3X the average standard deviation from the three days for each target metal. Eight replicates are analyzed.

8.1.5 The upper limit of the linear dynamic range must be established for each mass utilized by running a Linear Range Study. The upper limit (upper quantitation

limit or UQL in LIMS) is derived by analyzing consecutively higher standards until recoveries within 90-110% can no longer be obtained. At least 3 consecutive standards must meet the 90-110% recovery criteria. The UQL is set at the highest concentration in the study for each target metal. Any digestate value determined at levels above the UQL must be diluted and reanalyzed or qualified as an Estimated Value. Linear range studies are repeated at least every six months to update or verify UQLs.

8.1.6 The interference correction equations used for environmental sample matrices are provided by the manufacturer and are entered into the software prior to the analysis of samples. Examples are given in table 8-1 below.

Table 8-1
Example Correction Equations

Analyte	Mass	Interference Species	Equation Used
V	51	ClO	V=51cps-(3.127x^{53}ClO)
	53	ClO	^{53}ClO=^{53}cps-(0.113C52Cr)
As	75	ArCl	As=75cps-(3.127x^{77}ArCl)
	77	ArCl	ArCl=77cps-(0.815x^{82}Se)
Se	82	Kr	Se=82cps-(1.001x^{83}Kr)
Se	78	Ar2	Se=78cps-(0.1869x^{76}Ar2)
Cd	111	MoO	Cd=111cps-(1.073x^{108}MoO)
	108	MoO	MoO=108cps-(0.712x^{106}Pd)
Pb	208	Abundance	Pb=(^{206}Pb+^{207}Pb+^{208}Pb)

8.1.7 Prior to beginning Initial Calibration the instrument hardware and acquisition settings (Tune) must be verified. All specifications per Section 7.6 must be met prior to beginning analysis.

8.1.8 Initial Calibration is performed using internal standard techniques, calibration blanks and calibration standards at 4 levels for each target metal. Internal standards are introduced via in-line injection using a second channel of the sample introduction peristaltic pump. Specific calibration criteria are as follows:

8.1.8.1 All curve fits are Linear Regression through the blank with no weighting and interference correction on for all target metals. Correlation Coefficient must be $\geq$0.998 before sample analysis may begin.

8.1.8.2 The lowest calibration level must be at or below the LIMS specified PQL for each target metal.

8.1.9 Initial Calibration Verification (ICV). All initial calibration curves must be verified by analyzing a second source standard for each target metal. All recoveries must be within ±10% before sample analysis may begin.

8.1.10 Low-Level Initial/Continuing Calibration Verification standard (LLICV/LLCCV or CRI). The LLICV/LLCCV or CRI standard should be made at the established lower limit of quantitation. The standard must be analyzed at a concentration expected to be the lower limit of quantitation and at the beginning and at the end of each analysis batch with closing CCB/CCV. A more frequent analysis may be necessary to avoid re-analysis should the LLCCV fail if only analyzed at the end of the analysis batch. The acceptance criteria for the LLICV/LLCCV/CRI standard should be ±30%.

8.1.11 Interference Correction Verification (ICSA/ICSAB). ICSA and ICSAB must be analyzed at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Verification of the interference correction equations is accomplished by the analysis of the two interference check standards as follows:

8.1.11.1 ICSA solution is analyzed to verify the absence of positive or negative interference at the PQL level. This solution contains only the potentially interfering elements. The absolute value of the results for all target metals not included as interferents must be <PQL. See Section 8 for corrective action requirements.

8.1.11.2 ICSAB solution is analyzed to verify the absence of positive or negative interference with target analyte present. This solution contains potentially interfering elements plus specific target analytes. Percent recovery for all spiked target metals not included as interferents must be ±20% before sample analysis may begin. See Section 8 for corrective action requirements.

8.1.12 Continuing Calibration Verification (CCV). Calibration curves must be verified throughout the run sequence by analyzed CCV standards at least every 10 samples and at the end of the sequence. All target metals must recover within 90-110% of the expected values or the sequence must be stopped, new calibration curves obtained and all samples back to the last passing CCV reanalyzed.

8.1.13 Internal standards. The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard falls below 70% percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. First, make sure the instrument has not drifted by observing the internal standard intensities in the nearest clean

matrix (calibration blank). If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilutions of the affected samples. The sample must be diluted 9usually starting with 1:2 dilution) and reanalyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise to the minimum 70% limit. Reported results must be corrected for all dilutions.

8.1.14 Method blank. Method blank analysis must be performed with every batch of 20 or fewer samples. The MB is DI water containing all reagents used for analytical samples and must be carried through all preparation and analysis steps used for analytical samples. Target analytes must not be detected at levels above the PQL. Target analytes detected in Method blanks at levels >PQL must be handled in accordance with Section 8.2.

8.1.15 Laboratory Control Sample (LCS). The LCS is used to monitor, assess, and document laboratory method performance and is performed with every batch or twenty samples, whichever occurs more frequently. The LCS is spiked DI water and must be carried through all preparation and analysis steps used for analytical samples. The recovery of the analytes must meet established guidelines of 85-115% recovery (minerals 80-120%). Recovery outside control limits must be handled in accordance with Section 8.2.

8.1.16 Laboratory Control Duplicate Sample (LCSD). The LCSD is only analyzed in the absence of a batch MSD and is used to monitor, assess, and document laboratory method performance with every batch or twenty samples, whichever occurs more frequently. The LCSD is spiked DI water and must be carried through all preparation and analysis steps used for analytical samples. The recovery of the analytes must meet established guidelines of 85-115% recovery (minerals 80-120%). RPD value when compared to the LCS must be $\leq$20%. Recovery or RPD outside control limits must be handled in accordance with Section 8.2.

8.1.17 Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD). Matrix spikes are used to determine the effect of the sample matrix on the recovery and reproducibility of analytes, and are prepared and analyzed with each analytical batch up to 20 samples. The recovery of the analytes must meet established guidelines of 75-125% recovery. RPD value for the MSD as compared to the MS should be $\leq$20%. Recovery or RPD outside control limits must be handled in accordance with Section 8.2.

8.1.18 Post Digestion Spike (PDS). If the MS/MSD recoveries are unacceptable , the same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. Otherwise, another sample from the batch

should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. If this spike fails, then the Dilution Test (8.1.18) should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed, and the data should be appropriately narrated.

8.1.19 Dilution Test (DL). If the analyte concentration is sufficiently high (minimally, a factor of 10 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within ±10% of the original determination. If not, then a chemical or physical interference effect should be suspected, and the data should be appropriately narrated.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP# QA-01000, "Quality Assurance Manual," in Section 5. Included are tables that detail corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance, and any procedure deviations in appropriate logbooks.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 Health and Safety: Safety glasses and latex type gloves must be worn at all times when dealing with any chemicals, samples, or reagents. A lab coat is also highly recommended. Close-toed shoes and clothing that covers the legs (no shorts or dresses) must be worn any time an analyst is working in the laboratory.

9.2 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a health hazard and exposure should be kept as low as reasonably possible. All health and safety concerns for any chemicals are listed in the Material Safety Data Sheets (MSDS) provided by the supplier or manufacturer of these chemicals. A copy of any MSDS is available for review at any time.

9.3 The exhaust hoods should be maintained in a clean condition. Avoid getting Kimwipes or other paper inside the hood stack. This is not only a fire hazard but can also decrease the flow within the hood.

9.4 Acids should be handled with care. Always add acids and caustic solutions to water.

10.0 DATA REPORTING

10.1 The LIMS system automatically rounds the data based on factors set up for each test category. Typically, the LIMS reports to three significant figures.

10.2 The reporting limits can be changed in LIMS. Various laboratory personnel including project managers have the right to change the limits per the requirements of the clients. Reporting limits are based upon the MDLs and CRI results developed for each test.

10.3 Current laboratory MDLs and Reporting Limits (PQLs) can be found in tables in Section 5 of SOP# QA-01000, "Quality Assurance Manual" and LIMS test codes.

10.4 Calculations are performed by LIMS as follows:

10.4.1 Aqueous Samples:

µg/L = Inst. Result (µg/L) x DF

10.4.2 Soil Samples:

$$\text{mg/Kg} = \frac{\text{Inst. Result (µg/L)} \times \text{Final Vol. (L)} \times \text{DF}}{\text{Initial Sample wt (g)}}$$

(Correct for dry wt if required)

10.4.3 Surface Wipe Samples:

$$\text{µg, Total} = \frac{\text{Inst. Result (µg/L)} \times \text{Final Vol. (L)} \times \text{DF}}{\text{1(entire wipe digested)}}$$

10.4.4 Data import to LIMS.

10.4.4.1 Left click on the upper left hand corner of the Numerical Results Page in Plasma Lab

10.4.4.2 Right click to reveal the pull down menu and select "Copy All".

10.4.4.3 Go to "Local Disc C" on the Network Neighborhood and open the file labeled "Omega Me" then open Lims Import file.

10.4.4.4 Select "Enable Macros"

10.4.4.5 Left click on the upper left hand cell of the screen.

10.4.4.6 Right click and select “Paste”.

10.4.4.7 Click on the ICON of a “Running Man” on the upper right hand of the screen and enter the test code for the samples to import.

10.4.4.8 Click on the lower left hand tab labeled “Data”.

10.4.4.9 Delete calibration samples and any others you do not wish to import.

10.4.4.10 Check and edit (if necessary) Test Codes and Sample Types. Be sure to delete 2 spaces to the left of the time analyzed to ensure its placement to the far right.

10.4.4.11 Click on the “Red Diamond” ICON at the upper right and select “Replace Existing File”.

10.4.4.12 Close Excel and open LIMS.

10.4.4.13 Click on "Data Entry by Run".

10.4.4.14 Above the "Analytical Run Index, type the word "ICP" in the green empty box, then click on the ICPMS_TJA list, then "↵".

10.4.4.15 Select the desired ICP run from the list by double clicking on the run.

10.4.4.16 Click on the "Data Import" box (on the right side of the screen).

10.4.4.17 A box titled “File Import Specifications” will appear. In the pull-down menu for specifications, select "ICPMS". Then click on "OK".

10.4.4.18 A box will appear that contains a directory of data files. Select the directory C:\Omega_ME. From this directory, select the ICPMS data file.

10.4.4.19 Click “OK” to import data.

10.4.5 If the file does not link to LIMS, the error message "R15864" will appear next to a sample or samples in the "batch ID" field. If the data link was successful, a batch ID will appear in this field.

10.4.6 To check the information in the batch, press "Control B". This will take you to the prep batch section of LIMS. Correct the batch number, test code, or sample type as necessary.

10.4.7 Repeat the import process.

11.0 FILE MAINTENANCE

11.1 All data are electronically stored on the Portal Server.

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto CD-ROM disks. Two copies are made. One copy of the disks is stored on the laboratory premises, while the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks. Instrument logbooks must be completed any time maintenance is performed on the instrument. This includes routine maintenance such as changing or cleaning nebulizers, trimming tubing, and in the case of the ICP/MS, cleaning the cones. It also includes any non-routine maintenance that may be performed such as the replacement of motors in autosamplers.

Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: ICP/MS-TJA
Manufacturers name. Example: TJA
Serial Number. Example: #####
Date Received. Example: ####
Date Placed into Service. Example: mm/dd/yy

12.2 Routine Maintenance: Typical routine maintenance consists of keeping the system clean and insuring that all generated QC remains acceptable.

12.2.1 Cleaning the ICPMS.

12.2.1.1 Disassemble the nebulizer assembly by removing the threaded screws holding the spray chamber to the torch assembly. Remove the o-ring, support rods, and screws from the assembly. Disconnect the argon hose from the assembly.

12.2.1.2 Use acid to clean the injector tip.

12.2.1.3 Use 10% nitric acid in a bottle to soak the torch.

12.2.1.4 Sonicate the spray chamber to clean.

12.2.1.5 Reassemble the torch and spray chamber. The torch has a glass nipple on one side that is used for alignment.

12.2.1.6 Soak cones for 30 minutes in DI water. The use of sonication is permissible.

12.3 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as an autosampler that will not function. Except as indicated below, if this type of problem occurs, contact the department manager for assistance.

12.3.1 Electron multiplier replacement.

12.3.1.1 Select the "Off" ICON on the Plasmalab screen and select "Shutdown".

12.3.1.2 Remove the cover from the instrument.

12.3.1.3 Release the buckles on the face of the detector housing.

12.3.1.4 Disconnect the power supply to the Electron Multiplier and gently lift directly upward to remove.

12.3.1.5 Install new multiplier and reconnect the power supply.

12.3.1.6 Close the detector housing and select the "On" ICON on Plasmalab then select "Vacuum".

13.0 METHOD PERFORMANCE

13.1 General method performance data can be found in the referenced methods.

13.2 Laboratory specific method performance data is referenced in Section 17.0 of this SOP.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 SOP# HS-03005 Waste Disposal and SOP# SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 **ICB/CCB (Calibration Blank)** - A Volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument.

15.2 **Calibration Standard (ICAL)** - A solution prepared from the dilution of stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

15.3 **Dissolved Analyte** - The concentration of analyte in an aqueous sample that will pass through a 0.45 µm membrane filter assembly prior to the sample acidification.

15.4 **Instrument Detection Limit (IDL)** - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the selected analytical mass(es). (Table1).

15.5 **Internal Standard** - Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample or solution. The internal standard must be an analyte that is not a sample component.

15.6 **Laboratory Control Sample (LCS and LCSD)** - An aliquot of Di Water to which known quantities of the method analytes are added in the laboratory. The LCS (LCSD) is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

15.7 **Matrix Spike Sample (MS and MSD)** – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD are analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate

aliquot and the measured values in the MS/MSD corrected for background concentrations.

15.8 **Method Blank (MB)** – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

15.9 **Linear Dynamic range (LDR)** – The concentration range over which the instrument response to an analyte is linear.

15.10 **Method Detection Limit (MDL)** – The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentrations is greater than zero.

15.11 **Initial Calibration Verification Standard (ICV)** – A solution of method analytes of known concentrations which is used to check either laboratory or instrument performance.

15.12 **Stock Standard Solution** – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

15.13 **Total Analyte (a.k.a. Total Recoverable Analyte)** - The concentrations of analyte determined either by "direct analysis" of an unfiltered acid preserved drinking water sample with turbidity of 1< NTU, or by analysis of the solution extract of an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method.

15.14 **Tuning Solution** - A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses.

16.0 REFERENCES

16.1 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, February 2007, Methods 6020A.

16.2 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, April 1998, Methods 3005A and 3050B.

16.3 NIOSH Manual of Analytical Methods, Fourth Edition, Method 7300, Issue 2.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://home/aes/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://home/aes/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

AES, Inc.
3785 Presidential Pkwy.,
Atlanta, GA 30340

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DOCUMENT CONTROL NUMBER: Rev. *7*

DOCUMENT DISTRIBUTION NUMBER: IA-13037

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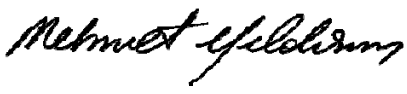
Laboratory Manager:  Date: 03/31/08

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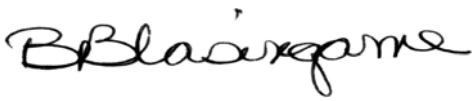
Department Supervisor:  Date: 03/31/08

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF MERCURY IN WATER BY MANUAL COLD VAPOR METHOD BY EPA SW-846 METHOD 7470A

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This procedure is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this document.

2.2 The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this procedure is 0.0002 mg/L.

3.0 INTERFERENCES

3.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents could be used to determine if this type of interference is present.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

4.1 All sample containers must be pre-washed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

4.2 Aqueous samples must be acidified with nitric acid to a pH of 2 or lower immediately after collection. Samples must be analyzed within the suggested holding time of 28 days.

5.0 REAGENTS

5.1 Reagent Water: Reagent water must be mercury free. The laboratory DI water fulfills the requirements of reagent water.

5.2 Sulfuric Acid (H_2SO_4), concentrated.

5.3 Hydrochloric Acid (HCl), 0.5N: Dilute 30 mL of concentrated HCl to 1.0 liter with reagent water.

5.4 Hydrochloric acid, concentrated (HCl).

5.5 Nitric Acid (HNO_3) concentrated. Reagent grade of low mercury content. Noticeable levels of mercury in a reagent blank usually are indicative of contaminated Nitric Acid.

5.6 Stannous Chloride ($SnCl_2$) solution: For FIMS add 22 g of stannous chloride to 1000 mL of 3% HCl. For Varian FAA add 250g of stannous chloride to 1000 ml **20%** HCl.

5.7 Sodium chloride-hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1000 ml.

5.8 Potassium permanganate ($KMnO_4$), 5% solution (w/v): Dissolve 50-g potassium permanganate in water and dilute with DI water to 1 liter.

5.9 Potassium persulfate ($K_2S_2O_8$), 5% solution (w/v): Dissolve 50 g of potassium persulfate in water and dilute with DI water to 1 liter.

5.10 Stock mercury solution, 1000 mg/L, purchased commercially.

5.11 Mercury intermediate standard: In a volumetric flask, dilute 1 ml of the stock mercury solution to 100-ml. The concentration of this solution will be 10 mg/L. This intermediate standard is prepared fresh each week. The acidity of the intermediate standard should be maintained at 2.0% nitric acid. This acid should be added to the flask before addition of the aliquot.

5.12 Mercury working standard, 100 µg/L. This standard is prepared fresh daily by dilution of 1.0-ml of the intermediate standard to 100-ml in a volumetric flask. The acidity of this standard should be maintained at 2.0% nitric acid by addition of 2.0-ml of concentrated acid to the volumetric flask prior to dilution to final volume.

5.13 Mercury calibration curve. Prepare standards in disposable digestion vessels as indicated in Table 5-1 below. Calibration standards are digested in a manner similar to samples. See Section 7 for the digestion procedure.

Table 5-1
Mercury Calibration Curve

Volume of Working Standard (5.12) (ml)	**Final Digestate Volume (ml)**	**Concentration in Sample (µg/L)**
0.0	50	0
0.08	50	0.16
0.25	50	0.5
0.50	50	1.0
1.0	50	2.0
2.5	50	5.0

5.14 The reagent blank must contain all the reagents, in the same volumes, used in processing the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration as the sample solution used for analysis.

5.15 Calibration verification standards.

5.15.1 Initial Calibration Verification Standard (ICV). This standard is prepared from a second source standard at a concentration of 4.0 ug/L.

5.15.2 Continuing Calibration Verification Standard (CCV). These standards are prepared from the same source as the calibration curve and analyzed at a concentration of 2.0 ug/L.

5.16 Vendor List. The standards used in this test are purchased using the catalog numbers and vendors indicated below.

Table 5-2
Mercury Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
Mercury Standard	VHG	1000 mg/L	PHGN-100
Mercuric Standard 2nd Source	Env Express	1000 mg/L	HP 100033-1
Stannous Chloride	EM Science	Pure	SX0885-7
Hydroxylamine Hydrochloride	VWR	Pure	BDH0236-500G
Potassium Permanganate	JT Baker	Pure	3227-05
Sulfuric Acid	Fisher	Concentrated	A300C 212
Hydrochloric Acid	Fisher	Concentrated	A144C 212
Nitric Acid	Fisher	Concentrated	A2005-212
Stannous Chloride	Fisher	Pure	T142-3
Potassium persulfate	Acros	Pure	7727-21-1
Potassium Perrmanganate	Fisher	Pure	P279-212

6.0 APPARATUS AND MATERIALS

6.1 Varian Spectra AA 220 spectrophotometer with SPS-5 autosampler and VGA77 mercury vapor generation system.

6.1.1 Mercury hollow cathode lamp or electrodeless discharge lamp.

6.1.2 Recorder: Any multi-range variable speed recorder that is compatible with the UV detection system is suitable.

6.1.3 Absorption Cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed of Plexiglas tubing, 1 in. O.D X 4.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in x 2-in cards. One inch diameter holes are cut in the middle of each card. The cards are then positioned and adjusted vertically and horizontally to give the maximum transmittance.

6.1.4 Vapor generation assembly complete with flow meter and internal drying tube.

The cold vapor generator is assembled as shown in the Varian owner manual. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system. Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system either to vent the mercury vapor out through an exhaust hood or to pass the vapor through an absorbing medium such as:

-Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4.
-0.25% Iodine in a 3% KI solution.

-Treated charcoal.

6.2 PE FIMS 100 Flow Injection Mercury Analysis System

6.2.1 PC with AA Winlab software

6.2.2 AS 90 Auto sampler.

6.3 Hot Block digestion system with digestion tubes or equivalent.

6.4 Assorted volumetric glassware as needed.

7.0 PROCEDURE

7.1 Preparation of digestion log form and digestion log in LIMS.

7.1.1 Each day the section supervisor prints a backlog report. The log lists samples included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN DIGESTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received with a "rush" status will have a chain of custody delivered by the receiving technician to the section supervisor of the department that performs Prep.

7.1.1.2 Prepare a written digestion log using the metals digestion logbook that is kept in the digestion area of the laboratory. The following entries must be made in the log.

7.1.1.2.1 Date and time the batch is opened or the date and time the sample(s) is placed on the hot plate.

7.1.1.2.2 All samples included in the digestion batch.

7.1.1.2.3 Volume or weight of samples digested.

7.1.1.2.4 Date and time the digestion is completed.

7.1.1.2.5 Digestion procedure employed.

7.1.1.2.6 The initials of the digestion analyst(s).

7.1.1.2.7 Laboratory number of all reagents used, including spiking standard and acids.

7.1.1.2.8 Final volume of all digestates.

7.1.1.2.9 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.1.1.3.1 Open a Prep Batch in LIMS by double clicking the "Prep" box.

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS after the prep code is entered.

7.1.1.3.3 Select the Prep Code "7470A_W_P" from the pull down list. The LIMS will automatically assign a MB and LCS sample to the prep list. If a LCSD is desired, click on the space below the sample name LCS and enter the information.

7.1.1.3.4 Enter the technician's name from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this method. Use "User" to select the samples to be included in the batch for desired prep method.

7.1.1.3.6 The samples can be individually selected by highlighting the sample and clicking the "$\rightarrow$" arrow or all samples can be selected by clicking the "$\Rightarrow$" arrow.

7.1.1.3.7 Samples that have been selected for re-analysis can be manually added to the sample list following the procedure outlined in section 7.1.1.3.3.

7.1.1.3.8 "Save" the batch by clicking on a previous batch number on the list and then return to the newly created batch.

7.1.2 Table 7-1 indicates the type of samples that comprise an analytical batch. Note: NELAC requirements specify that the maximum number of client samples in a prep batch can not exceed 20. Further, a prep batch can not be left "open" for a period that exceeds 24 hours.

Table 7-1
Samples in a NELAC Batch

Method Blank (MB)
LCS and LCSD
Client Samples
MS and MSD (If supplied by client)

7.2 Sample preparation. (See Table 7-2 for Sample Preparation Checklist)

7.2.1 Transfer 40 mL, or smaller volume if necessary, of each sample and blank containing < 1.0 g of mercury, to a disposable digestion vessel. If less than 40 ml is used, add DI water to a volume of 40 ml in the digestion tube.

7.2.2 Prepare Cal Curve standards by adding appropriate amounts of Hg working standard to digestion tubes per Table 5-1 and adding DI water to a volume of 40 ml. **Standard concentrations are based on the final digestate volume of 50 ml.**

7.2.3 Prepare an ICV at a concentration of 4.0 ug/L by adding 2.0 ml of second source Hg standard to a digestion vessel and adding DI water to a volume of 40 ml. ICV standard must be from a source other than that used for the ICAL standards.

7.2.4 Prepare the CRA at a concentration of 0.16 ug/L by adding 0.08 ml of second source Hg standard to a digestion vessel and adding DI water to a volume of 40 ml.

7.2.5 Prepare MB and LCS by adding 40 ml of DI water to digestion tubes.

7.2.6 Spike LCS and MS/MSD tubes by adding 2.0 ml of the 100 ug/L Hg working standard used for the ICV.

7.2.7 Add 2.5 mL of H_2SO_4 and 1.25 mL of concentrated HNO_3, mixing after each addition to each digestion tube. Add 7.5 ml of permanganate solution to each digestion tube. Some samples may require additional permanganate as indicated by loss of purple color. **Purple color must be maintained throughout digestion**.

7.2.8 Shake and add 4 ml of potassium persulfate to each vessel and heat for 2 hours in a digestion block maintained at 95°C.

7.2.9 Cool and add 3 mL of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanganate. Stannous chloride is added by the vapor generator per the instrument instruction manual.

7.3 Instrument Set Up for Varian AA

7.3.1 Turn on the hood over the instrument.

7.3.2 Power up the Varian instrument.

7.3.3 Open the cover and verify that the mercury lamp is on and allow it to warm up for at least 30 minutes. Lamp selection lever must be pointing to the correct lamp.

7.3.4 Attach Hg cell to screws on burner

7.3.5 Attach the VGA 77 sampling system.

7.3.6 Place tubing in appropriate reagent solutions

7.3.7 Verify rinse is correct for analysis

7.3.8 Check the condition of the pump tubes and replace if necessary.

7.3.9 Fill the appropriate tubes in the autosampler trays and place the trays on the sampler. Note that the system will not operate if the trays are not positioned properly in the sampler.

7.3.10 The analytical sequence is run in the following order:

-Cal Curve Standards
-ICV (Second Source)
-ICB
-CRA
-Up to 10 samples
-CCV
-CCB
-Repeat as necessary

7.4 Sample analysis for Varian AA.

7.4.1 Click on the "Spectra AA" icon on the data computer.

7.4.2 Select “Worksheet” then “New From” and highlight old run to be used as the template.

7.4.3 Name the new file as “Hg MM/DD/YYYY” then enter initials and click “OK”.

7.4.4 Click on "Optimize" icon, select the method, and then click "OK".

7.4.5 Make sure lamp levels are close to each other and close to 1.0, if not it may be necessary to adjust the burner height. Light may not be passing directly through the center. Use a 2 in. x 2 in. card to test level of transmittance. Adjust burner height by using the gray knob (horizontal) or black knob (vertical).

7.4.6 If the lamp is not a Varian lamp, go to the Lamp Screen and verify the lamp settings such as current, etc. Light the lamp from the Lamp Screen.

7.4.7 Click on the "Labels" tab. Type in your new sequence making sure that old samples are deleted from the new sequence. When done, click “instrument” tab.

7.4.8 Select the "Select" tool and highlight the sequence information on the screen. Click on "Start" to initiate the run.

7.4.9 The Spectra AA-220 system will automatically generate the calibration curve from the standards that were analyzed and calculate the sample values from the calibration curve.

7.5 FIMS System Operation (See Table 7-4 for FIMS Loading Diagram)

7.5.1 Turn instrument on and double click the “FIMS” icon to start the software.

7.5.2 Use a custom designed workspace icon then double click on “hgfims.fms”. This opens all working windows needed for operation.

7.5.3 Click on “File” then “New” then “Sample Info File”.

7.5.4 Toggle to Batch ID and type in batch ID information as “YYMMDD (A,B,C,etc.)”.

7.5.5 Tab to “Analyst” and type in analyst’s initials.

7.5.6 Tab to “Sample Volume” and enter appropriate value (50ml).

7.5.7 Tab to Autosampler location 9 and type in sample IDs, starting with CRA

7.5.8 After all samples have been entered, click on “File” then “Save As” then “Sample Info file”, enter file name as “YYMMDD” then the appropriate letter (A,B,C,etc) then toggle to OK.

7.5.9 Close the Sample Info File.

7.5.10 Under the automated analysis window, make sure the setup tab is open. Make sure HGFIMS is selected as the method. If not, double click on the method and select the correct one.

7.5.11 Click on Sample Information Browse and type in the Sample Information File name click on OK then click on the "use entire sample info" box.

7.5.12 Click on results data set name and type in the results data name, then click ok.

7.5.13 Click on "Lamp" if you want the lamp to be turned off at the end of the run automatically.

7.5.14 Click on the "Analyze" tab at the button of the Automated Analysis window.

7.5.15 Click on "Analyses" tool bar at the top of program window. Scroll down to "Autosampler Loading List" and click on it. Double check that all samples are present. Click "Print" then exit.

7.5.16 Fill solution reagent bottles.
-Clear Bottle properly labeled: 3% HCl
-Clear Bottle properly labeled: SnCl2 (**MUST HAVE ENOUGH TO COMPLETE THE RUN. CHANGING SnCl2 WILL REQUIRE RECALIBRATION)**
-DI Water Bottle

7.5.17 Replace filter daily or more frequently if needed.

7.5.18 Connect the Tygon tubing on the FIMS 100 and snap clamps into place. Adjust clamp tightness if necessary.

7.5.19 Change tubing every other day.

7.5.20 Click on the "FIAS" toolbar.

7.5.21 Place the tubing lines and the autosampler probe into deionized water and click the "Pump 1" icon to start the pump. Let the system rinse for 15 minutes. Check flow rate for all four tubes. Click the "Pump 1" icon to stop the pump at the end of the rinse period.

7.5.22 Place the red line into the SnCL2 bottle and the blue line into the 3% HCl bottle and close the FIAS window.

7.5.23 Load the Autosample tray with standards, QC and samples.

7.5.24 The analytical sequence is run in the following order:

-Cal Curve Standards
-ICB
-ICV (Second Source)
-CRA
-Up to 10 samples

-CCV
-CCB
-Repeat as necessary

7.5.25 Click “Calibrate” to calibrate. Make sure correlation coefficient is at least 0.995. Monitor the reagent bottles during analysis and refill if necessary. **DO NOT ALLOW THE REAGENT BOTTLES TO GO DRY.**

7.5.26 Verify calibration is passing , then click “ Analyze Sample”

7.5.27 After all samples have been analyzed, place the red and blue lines into the DI water bottle, click on the “Pump 1” icon to flush the system for approx. 5 minutes.

7.5.28 Remove the line and autosampler probe and place in the FIMS tray and allow the pump to operate for 2-3 minutes to air purge the system. Click on the “Pump 1” icon to stop the pump after the air purge is complete.

7.5.29 Close the FIAS window.

7.5.30 Unsnap the tubing clamps, turn off FIMS, click on “File” then “Exit” then turn off computer.

7.5.31 Turn off vent fan.

7.5.32 See Table 7-5 for FIMS Data Exporting Instructions.

Table 7-2
7470A_W_P (245.1)

Batch No._____

____ 1**:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (7470A_W_T, 7470A_W_D, 7470A_W_P, 1311_HG, 245.1_W)

____ 2: PICK A SAMPLE FOR QC (ORIGINAL, MS, AND MSD)

____ 3: PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ 4: ADD 40 ML OF EACH SAMPLE TO DIGESTION VESSEL. SMALLER VOLUMES MAY BE USED FOR SAMPLES KNOWN TO CONTAIN HIGH LEVELS OF HG OR IN THE EVENT OF MATRIX INTERFERENCE. IF LESS THAN 40 ML IS USED, ADD SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 ML. RECORD THE ACTUAL SAMPLE VOLUME PLACED IN THE DIGESTION VESSEL.

____ 5: BATCH SAMPLE IN THE LIMS, PRINT A COPY OF THE BATCH

____ 6: MAKE ICAL STANDARDS FRESH DAILY AT 0.16, 0.5, 1.0, 2.0, 5.0, UG/L BASED ON THE FINAL DIGESTATE VOLUME OF 50 ML BY ADDING 0.08, 0.25, 0.50, 1.0, AND 2.5 OF THE 100 UG/L HG STANDARD AND SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 ML IN THE DIGESTION VESSELS.

____ 7: PREPARE AN ICV AT 4.0 UG/L BY ADDING 2.0 ML OF SECOND SOURCE 100 UG/L HG WORKING STANDARD AND SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 ML IN THE DIGESTION VESSEL. **STANDARD MUST BE FROM A DIFFERENT SOURCE THAN THAT USED FOR THE ICAL STANDARDS.**

____ 8: PREPARE THE CRA AT 0.16 UG/L BY ADDING 0.08 ML OF SECOND SOURCE 100 UG/L HG WORKING STANDARD AND SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 ML IN THE DIGESTION VESSEL.

____ 9: ADD 40ML of DI WATER TO THE LCS & MB DIGESTION VESSELS.

____ 10: SPIKE LCS, MS & MSD WITH 2.0 ML OF THE 100 UG/L HG WORKING STANDARD USED TO PREPARE THE ICV. (WITNESS NEEDED)

____ 11: ADD 2.5 ML H_2SO_4, 7.5 ML $KMNO_3$, 1.25 ML HNO3, 4 ML $K_2S_2O_8$, THEN HEAT FOR 2 HR AT 95°C. **SAMPLES MUST MAINTAIN PURPLE COLOR**, ADD ADDITIONAL KMNO3 CRYSTALS AS NEEDED AND REHEAT IF NECESSARY.

____ 12: REMOVE FROM HEAT AND LET COOL

____ 13: LINE UP SAMPLES IN ORDER AS THEY APPEAR IN LOG

PREPARED BY: _____ DATE: _____

Batch folders

____ **:** Does it have BACKLOG REPORT FOR THE PREP AND EACH TEST CODE

____ : Is the SAMPLE FOR QC (dup, ms) adequate

____ : ARE the SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : Check SAMPLE final volume in DIGESTION VESSEL, does it match Recorded VOLUME

____ : CHECK prep log against BATCH IN THE LIMS (sample prep, test code, date time, analysts, check sample ID's, OK, add DUP & MS, check volumes), or Printed A COPY OF THE BATCH

ANALYST: _____ DATE: _____

Table 7-3
DATA REVIEW CHECKLIST- Hg by FIMS/FAA

Batch ID: _____ Data File:_____

QA Analyst

INITIAL RAW DATA REVIEW

___ ___: Have prep logs, prep checklists, raw data and run log(s) been printed to pdf and posted to Portal Server
___ ___: Are all instrument standards used listed on raw data
___ ___: Do calibration curves for all target analytes meet requirements where (≥0.995 Corr.Coef.)
___ ___: Are all replicated RSDs≤15% for all values over PQL? **No requires reanalysis and CAR if still out at reanalysis**

GO TO LIMS "MAIN" RUN SCREEN

___ ___: Has the LIMS Prep batch been properly entered matching the prep log book and closed
___ ___: Are any CARs listed in comments box **(Yes requires determining possible actions before continuing)**
___ ___: Are all Sample IDs properly assigned per Backlog Report (Double click each SampleID to verify)
___ ___: Are all Test Codes properly assigned per Backlog Report
___ ___: Is instrument QC run at the required frequencies
___ ___: Are all Sample Types properly assigned
___ ___: Are all samples linked to the Prep Batch properly with correct PFac, SpkFac and OFac
___ ___: Are dilution factors entered correctly per the raw data Run Log
___ ___: Are all Blkref, SPKref, RPDref and CCVref assigned correctly
___ ___: Are there any Comments present that require attention? **May require CAR**

GO TO DATA SCREEN

___ ___: Calculate Sequence (Cal SEQ tab) to insure LIMS calculations are completed.
___ ___: Are there any S or B flags for element of interest on the Instrument QC (ICV, CRA, CCV)? **Yes Requires CAR**
___ ___: Are all values for target analytes between <PQL and > -PQL on ICB, CCB and MBs? **No Requires CAR**
___ ___: Are there any B flags for elements of interest on any samples and/or Batch QC? **Yes Requires CAR**
___ ___: Are there any S and/or R flags for elements of interest on Batch QC (LCS/D, MS/D, PDS)? **Yes Requires CAR except R or S flags on PDS caused by value at or near PQL or insignificant spike.**
___ ___: Are there any H flags present for any samples? **Yes Requires CAR**
___ ___: Are there any E flags for elements of interest on any samples and/or Batch QC selected for reporting? **Yes Requires CAR**
___ ___: Are there any J flags on any target analytes selected to report on diluted sample runs? **Yes Requires reanalysis at lower dilution or CAR to narrate elevated reporting limits.**
___ ___: Have readings been entered properly (check at least 2 entries for downloaded data, **ALL** entries for manual data)
___ ___: Have at least 2 sample's final results in LIMS been verified by hand calculation
___ NA: Have **ALL** CARs been closed and narratives written prior to QA?
___ NA: Have PM, Lab Manager and PM Director been notified for any CAR resulting in reextract and/or due date exceedance?

CAR#: _____ _____ _____ _____

Analyst Signature: _____Date: _____ Time: _____

Reviewer Signature: _____ Date: _____ Time: _____

Table 7-4
FIMS Loading Instruction Diagram

I. To open a "work space"

1. Workspace HgFIMS.fms

or

2. File Open- Workspace- HgFIMS.fms

II. Create a New autosampler loading list

1. File Open- Sample info file
   Batch ID: YYMMDD "letter A,B,C,…" Analyst: Initials
   Sample Vol: 50.0

1
2
3
4
5
6
7
8
9 CRA
10 MB-
11 LCS-
12 Sample
13 MS
14 MSD
15 PDS

File Save as- Sample info file- YYMMDD "letter A,B,C,…"

III. Run setup

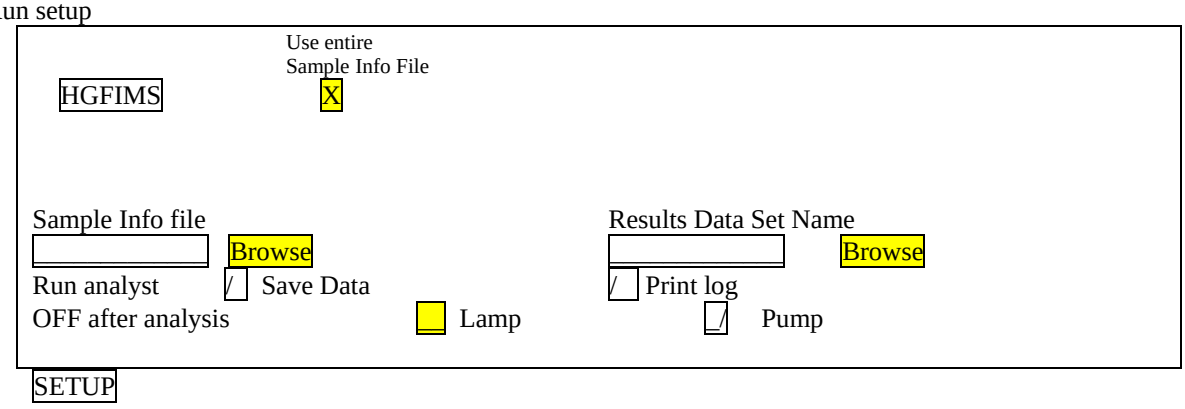
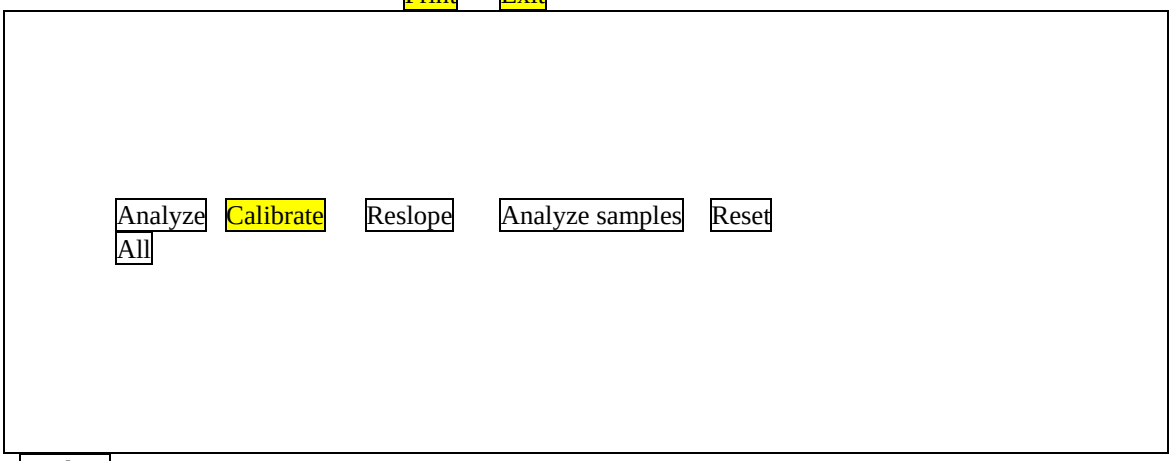


Table 7-4 (Cont)
FIMS Loading Instruction Diagram

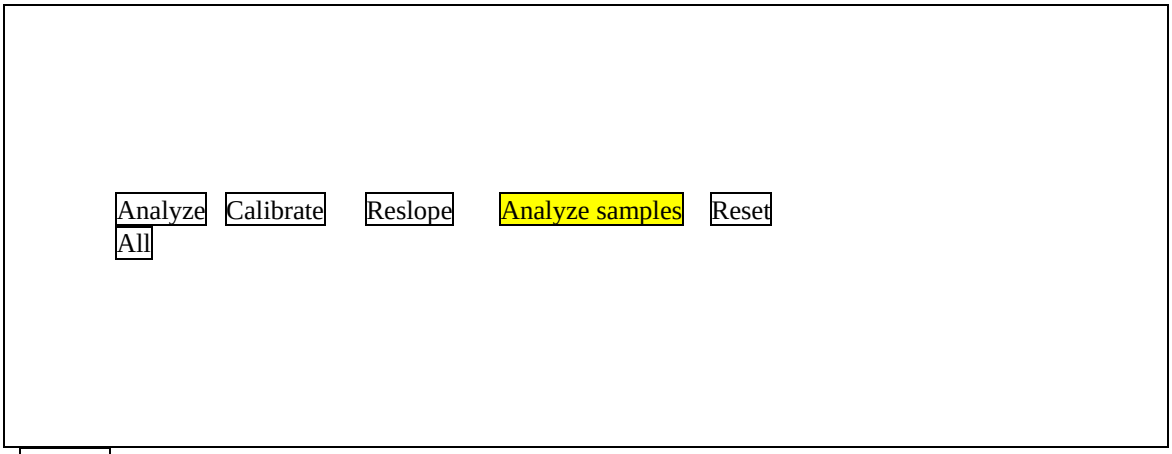
1. Under Sample Info file, use Browse to select the autosampler file that you just saved
2. Under Results Data Set, use Browse and then type in the name of the file that you want the data stored in. File name is usually the same as Sample Info File.
3. Click on the block under use entire sample info file
4. If you are putting a run on to finish after you go home, click on the Lamp block

Switch to the Analyze page- Analyses- Autosampler loading list



Analyze

1. Select Calibrate, the program will stop after calibration is complete



Analyze

1. Select Analyze samples after a passing calibration

IV. Adding to Autosampler loading list

1. File Open- Sample Info File- open file that you want to add to
2. File Save

Table 7-5
FIMS Data Exporting Instruction Diagram

I. [START]-PROGRAMS-FIMS-AA Winlab reformat
[open design]
[export]-Double check
[browse]-data set name, (select file)
[save results]
[OK]
X -close window
[OK]

II. My computer-C:/OmegaME/Fims export
Enable Macros
Run macro (running man)
Enter test code(s)
[OK]
Delete curve and unused samples-change IDs-change test codes if needed
Run macro <> (diamond)
[OK]
[OK]
X Exit excel
[YES]

III. Omega SQL - LIMS (Make sure that you have closed the prep batch)
[Data entry]
[Add]

Instrument ID:	Fims
Run Start Date:	MM/DD/YY
Analyst:	Your name

[Data Import]
Specification
Fims
C:/omegaME/FimsData
[OK]

Table 7-6
FAA Loading Instruction

1. Double click on the SpectraAA icon on the desktop.
2. Select "Worksheet" then "New From" and highlight the most recent run to be used as the template.
3. Name the new file as "Hg MM-DD-YYYY [A, B, C, etc.]" then enter initials and click OK.
4. Click on the "Labels" tab. Type in the new sequence as follows:
    - ICV
    - ICB
    - CRA [0.16 ppb for waters and 0.5 ppb for soil & waste samples]
    - MB
    - LCS
    - Up to 7 samples
    - CCV
    - CCB
    - Up to 10 samples
    - CCV
    - CCB
    - Repeat as necessary

5. Rows can be added by clicking on "Total Rows…" and typing in required number of rows.
6. Save the loading list by clicking on the "Loading Guide" tab to the left of the screen.
7. The loading list will pop-up. Click the "Print" tab at the bottom.
8. Make sure "Adobe PDF" is selected from the drop down menu and click "OK"
9. Save the file under Desktop → FAA Analysis → Mercury → and save in this format "Hg MM-DD-YYYY [A, B, C, etc.] _Run Log"
10. A pdf file of the loading list will pop-up. At this point you can print the loading list by selecting File→ Print → from the drop down menu. Under "Printer", select HP Laserjet 2100 and click OK.
11. Once the loading list is complete, saved, and printed, select the "Analysis" tab. All of the samples should be highlighted in red as an indication to be analyzed.
    A. To not analyze a sample simply click on the "Select" tab to the left of the screen.
    B. The cursor should change into a highlighter icon. Click on the sample you wish to not analyze and the sample should be a white block.
    C. Once done un-selecting samples click on the "Select" tab again.
12. Begin analysis by clicking the "Start" icon to the left of the screen. Then click "OK" on the next pop-up screen.
13. To re-analyze a sample click the "Stop" icon to the left of the screen. Once the probe has finished its rinse cycle, click on the "Select" icon and then highlight the sample(s) needed to be re-analyzed then click the "Select" icon once again to return to the main analysis screen.
14. At the top of the screen select "Instrument" and click "Start At…"
15. From the drop down menu select "Solution" and click Start.
16. Click "OK" on the "Analysis Checklist" pop-up screen.

Table 7-7
Exporting FAA Hg Data into LIMS

1. Click reports (printer icon) at the top of the SpectraAA 220 program.
   - Select the data set to be imported.

2. Select the Settings tab and make sure only the following items are checked under Solution Data and Report Contents:

Concentration	Date	Dilution Factors
%RSD	Time	
	Overrange Dilution Factor	

3. Select the Report tab and click Export to PRN file…
   Save the file name same as the worksheet name and click Open and close out of Reports.

4. Open the Excel program by clicking Start → Programs → Microsoft Office → Microsoft Office Excel 2003 then,
   - Click File → Open → My Computer from the pull down menu→ Local Disk: (C) → Varian → sp100 → Data → Files of type: Select "Text Files (*.prn;*.txt;*.csv) from the drop down box.
   - Select the file just exported as .prn file and click Open.
   - "Text Import Wizard – Step 1 of 3" should pop-up. Click Next and uncheck the Tab icon and check the Comma icon under "Delimiters"
   - Click Finish.
   - Right click on Column C and delete and then File → Save → Yes
   - Close the program
   - Click No to save changes made.

5. Open "FAA_Export.xls" on the desktop of the FAA computer.

6. Click Enable Macros.

7. If the spreadsheet contains sample data, click the "Clear Data" button.

8. Click "Import Data" → Double click My Computer → Local Disk: (C) → Varian → sp100 → Data → Scroll over to select data set.

9. Click "Set Test" and enter the appropriate test code for the samples to be imported.

10. Copy an empty row by right clicking on the number to the left of the cell and click copy. Next right click row 6 and click "Insert Copied Cells." This is to ensure that the ICV properly imports to LIMS.

11. Using the row selector (the column of numbered boxes to the left of the cells), select the data not needed. Right-click the selection and select "Delete".

Table 7-7 (cont)

12. If any samples contain dilution information in the Sample ID, enter that dilution under the DF column and delete it from the Sample ID.

13. Using the row selector, select all rows containing samples and row 6 that contains no information to be imported into LIMS.

14. Click “Export” to transfer all selected data to the “FAA_DATA.xls” spreadsheet. And click **yes** to replace the file.

15. The FAA_Data.xls spreadsheet will remain open, allowing the user to review the data transferred. If all desired samples are listed, close the FAA_DATA.xls spreadsheet and click **no** when asked to save changes.

16. In the LIMS, add an entry under Data Entry. Then click the Data Import button. Select the “FAA” specification and click “Run Import”.

17. Click on My Computer → Local Disk: (C) → OmegaME → Open file “FAA_DATA.xls” → Open → OK.

8.0 QUALITY CONTROL

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP# QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Initial Calibration (ICAL): This is accomplished through a minimum 5-point calibration curve. The correlation coefficients (r) for the curve must be $\geq$0.995.

8.1.3 Initial Calibration Verification (ICV). An ICV standard prepared from a source other than that used for the ICAL standards must be analyzed immediately following calibration. Recovery must be within ±10% of true value.

8.1.4 Reporting Limit Verification Standard (CRI). The CRI must be analyzed after each calibration to verify recovery at the required PQL. The results are acceptable if the measured concentration of each analyte is within limits specified in LIMS.

8.1.5 Continuing Calibration Verification (CCV). A CCV standard must be analyzed after every 10 injections and to close each run/sequence. Recoveries must be within ±10% of true value.

8.1.6 Continuing Calibration Blank (CCB). A CCB must be analyzed immediately after each CCV sample. Value must be less than the PQL for all target analytes.

8.1.7 Method Detection Limit Study. The method detection limit is calculated by analyzing at least seven replicates prepared in blank water at concentrations near, at or equal to the lowest PQL reported. Quantitation limits are laboratory derived from the MDL study data set and project specific requirements. All PQLs must be >MDLs. MDLs are to be performed annually or whenever instrument conditions have changes that will affect the established detection limits.

8.1.8 Method blank. Reagent blank analysis must be performed at the following frequency: Every twenty (20) samples of similar concentration and/or sample

matrix or whenever samples are extracted and analyzed by the same procedure at same time, whichever is more frequent. The concentration of the method blank of any analyte of interest must not exceed the laboratory established practical quantitation limit (PQL).

8.1.9 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD). The LCS is used to monitor, assess, and document laboratory method performance and is performed on every batch or twenty samples, whichever occurs more frequently. The LCSD is only analyzed in the absence of an MSD. The recovery of the analytes must meet established laboratory guidelines.

8.1.10 Matrix spike. Matrix spikes are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples of similar matrix. The recovery of the analytes should meet established laboratory guidelines.

8.1.11 Matrix Spike Duplicate (MSD). Matrix Spike Duplicates are used to determine the reproducibility of the test method on a specific matrix. One MSD per analytical batch is digested and analyzed for all target metals. The results are used to calculate RPD values which should be within the established limits. If MSD analysis is not possible due to limited sample volume, LCSD is used to evaluate precision.

8.1.12 Post Digestion Spike (PDS). PDS is used to monitor for matrix interference effects. At least one sample per batch is diluted 1:2 with a PDS solution that is made from the highest calibration standard and analyzed. Value for the DL test sample must agree with the true value within 15% or interference should be suspected. When interference is indicated, samples must be diluted to a level where interference is no longer present and reanalyzed or data appropriately narrated.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP# QA-01000, “Quality Assurance Manual,” in Section 5. Included are tables that detail corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance, and any procedure deviations in appropriate logbooks.

9.0 SAFETY REQUIREMENTS

9.1 Health and Safety: Safety glasses and latex type gloves must be worn at all times when dealing with any chemicals, samples, or reagents. A lab coat is also highly recommended. Close-toed shoes and clothing that covers the legs (no shorts or dresses) must be worn any time an analyst is working in the laboratory.

9.2 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a health hazard and exposure should be kept as low as reasonably possible. All health and safety concerns for any chemicals are listed in the Material Safety Data Sheets (MSDS) provided by the supplier or manufacturer of these chemicals. A copy of any MSDS is available for review at any time.

9.3 The exhaust hoods should be maintained in a clean condition. Avoid getting Kimwipes or other paper inside the hood stack. This is not only a fire hazard but can also decrease the flow within the hood.

9.4 Acids should be handled with care. Always add acids and caustic solutions to water.

10.0 DATA REPORTING

10.1 The LIMS system automatically calculates the data based upon factors that are set up for each test code.

10.2 Out-Of-Control Data - Contingencies for handling out-of-control or unacceptable data are included in SOP #QA-01000, "Quality Assurance Manual," of the in-house method manual in Section 5 including corrective actions for failing QC and/or acceptance criteria.

10.3 Calculations

10.3.1 The LIMS automatically calculates the data based upon factors that are set up for each test code and applicable preparation factors.

10.3.2 Calculations are performed by LIMS as follows:

10.3.2.1 Aqueous samples:

$$\text{ug/L} = \frac{\text{Inst. Result (ug/L)} \times \text{Final Vol. (L)} \times \text{DF}}{\text{Volume Digested (L)}}$$

10.3.3 Data import to LIMS. (See Tables 7-6 and 7-7).

11.0 FILE MAINTENANCE

11.1 All data are electronically printed as pdf files and stored on the Portal Server.

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto writeable CD ROM disks. Two copies are

made. One copy is stored on the laboratory premises; the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks. Instrument logbooks must be completed each time that any maintenance is performed on the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as replacement of motors in tower autosamplers.

Each instrument logbook must have a cover page that includes the following information.

Equipment name: Example: GC-5
Manufacturers' name: Example: Hewlett Packard 6890 GC
Serial Number: Example: 13226589A
Date Received: Example: 11/01/00
Date Placed into Service: Example: 11/05/00

12.2 Routine Maintenance: Typical routine maintenance consists of keeping the system clean and insuring that all generated QC remains acceptable. Examples would be cleaning of the torch and sample introduction lines.

12.3 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as autosampler that will not function. If this type of problem occurs, contact the department manager for assistance.

Table 12-1
Routine Maintenance Log for PE FIMS

AES, Inc.
3785 Presidential Pkwy
Atlanta, GA 30340

February 2008 **FIMS 1429**

Day	1	2	3	4	5	6	7	8
Date	F	SA	SU	M	T	W	H	F
Routine Maintenance								
Daily Visual inspection:								
Tubing								
Filter								
Exhaust fans on								
gas pressure (at least 100psi)								
Regular Maintenance:								
Replace tubing								
Replace filter								
Remake Stannous Chloride solution								
Replace rinse								
Waste discarded (daily)								
Rinse Instrument w/ 8% concentrated acid								
Inspect cell body, make sure no moisture is present								
Weekly:								
Instrument/autosampler wiped down								
Change charcoa								
Monthly Maintenance:								
Inspect lamp (as needed)								
Comments (Please note with an *)								
Analyst								

Table 12-2
Routine Maintenance Log for Varian FAA

Analytical Environmental Services, Inc.
3785 Presidential Parkway
Atlanta, GA 30340

February 2008 **FAA AES1148**

Day	F	SA	SU	M	T	W	H
Date	1	2	3	4	5	6	7
Routine Maintenance							
Daily Visual inspection:							
Pump tubing							
Burner Head							
Exhaust fans on							
Check Acetylene							
Check lamp (no arcing)							
Regular Maintenance:							
Pump tubing conditioned (30 min. minimum)							
Rinse station filled							
Acetylene replaced (if below 100psi)							
For Hg only - rinse lines with DI H_2O after analysis							
Cover exhaust on instrument with cardboard at end of analysis (daily)							
Weekly:							
Instrument/autosampler wiped down							
Waste discarded							
Standard tubes cleaned							
Tubing replaced							
Monthly Maintenance:							
Burner Head cleaned							
Condition of lead lamp checked							
Nebulizer sonicated							
Glass bead checked							
Spray chamber and liquid trap cleaned							
Nebulizer flow optimized							
O-rings checked							
Comments (Please note with an *)							
Analyst							

13.0 METHOD PERFORMANCE

13.1 Method performance data can be found in the referenced methods.

13.2 Laboratory specific method performance data is referenced in Section 17 of this SOP.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 SOP WM-17001 Waste Disposal and SOP SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information:

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade –A dry chemical that has been dried at 250°C for 4 hours cooled and stored in a desiccator.

15.2 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to deionized water or clean soil and the concentration measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.3 DI water- Deionized water

15.4 RSD – Relative Standard Deviation

15.5 MS- Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.6 MSD- Matrix Spike Duplicate.

15.7 CCV - Continuing calibration verification standard.

15.8 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot than the calibration standard.

15.9 LCSD - Laboratory Control Sample Duplicate

15.10 Dissolved - Those elements which will pass thorough a 0.45 µm membrane filter

15.11 Total Recoverable or Total Metals – The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

15.12 Reagent blank - A volume of deionized water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.

15.13 Calibration blank - A volume of deionized water acidified with HNO3 and HCl.

15.14 Calibration standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Test Procedures for Evaluating Solid Wastes, SW-846, Revised December 1996, Method 7470A.

16.2 Code of Federal Regulations, 40CFR Part 136, Appendix B, July 1993

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://home/aes/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://home/aes/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

AES, Inc.
3785 Presidential Pkwy.
Atlanta, GA 30340

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DOCUMENT CONTROL NUMBER: Rev. 5

DOCUMENT DISTRIBUTION NUMBER: IA-13033

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STANDARD OPERATING PROCEDURES FOR MERCURY IN SOLID OR SEMI-SOLID WASTE (MANUAL COLD-VAPOR TECHNIQUE)(BY METHOD 7471B)

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This procedure is approved for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution step prior to analysis. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this procedure is not applicable for that matrix.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the solid or semi-solid samples must be prepared according to the procedures discussed in this procedure.

2.2 This procedure, a cold-vapor atomic absorption method, is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes though a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical instrument detection limit (IDL) for this procedure is 0.0002 mg/L and 0.0025 mg/kg.

3.0 INTERFERENCES

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

3.3 Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL).

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

4.1 All sample containers must be pre-washed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

4.2 Aqueous samples must be acidified with nitric acid to a pH of 2 or lower immediately after collection. Samples must be analyzed within the suggested holding time of 26 days for CLP reportable samples and 28 days for all others.

4.3 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

5.0 REAGENTS AND STANDARDS

5.1 Reagent Water: Reagent water must be mercury free. The laboratory DI water fulfills the requirements of reagent water.

5.2 Hydrochloric Acid (HCl), 0.5N: Dilute 30 mL of concentrated HCl to 1.0 liter with reagent water.

5.3 Hydrochloric acid, concentrated (HCl).

5.4 Nitric Acid (HNO_3) concentrated. Reagent grade of low mercury content. Noticeable levels of mercury in a reagent blank usually are indicative of contaminated Nitric Acid.

5.5 Stannous Chloride ($SnCl_2$) solution: For FIMS add 22 g of stannous chloride to 1000 mL of 3% HCl. For Varian FAA add 250g of stannous chloride to 1000 ml **20%** HCl.

5.6 Sodium chloride-hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1000 ml.

5.7 Potassium permanganate ($KMnO_4$), 5% solution (w/v): Dissolve 50-g potassium permanganate with DI water to 1 liter in a volumetric flask.

5.8 Stock mercury solution, 1000 mg/L, purchased commercially.

5.9 Mercury intermediate standard: In a volumetric flask, dilute 1 ml of the stock mercury solution to 100-ml. The concentration of this solution will be 10 mg/L. This intermediate standard is prepared fresh each week. The acidity of the intermediate standard should be maintained at 2.0% nitric acid. This acid should be added to the flask, as needed, before addition of the aliquot.

5.10 Mercury working standard, 100 µg/L. This standard is prepared fresh daily by dilution of 1.0-ml of the intermediate standard to 100-ml in a volumetric flask. The acidity of this standard should be maintained at 2.0% nitric acid by addition of 2.0-ml of concentrated acid to the volumetric flask prior to dilution to final volume.

5.11 Mercury calibration curve. Prepare standards in disposable digestion vessels as indicated in Table 5-1 below. Calibration standards are digested in a manner similar to samples. See Section 7 for the digestion procedure.

Table 5-1
Mercury Calibration Curve

Volume of Working Standard (5.12) (ml)	Final Digestate Volume (ml)	Concentration in Sample (µg/L)
0.0	50	0
0.08	50	0.16
0.25	50	0.5
0.50	50	1.0
1.0	50	2.0
2.5	50	5.0
5.0	50	10.0

5.12 The reagent blank must contain all the reagents, in the same volumes, used in processing the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration as the sample solution used for analysis.

5.13 Calibration verification standards.

5.13.1 Initial Calibration Verification Standard (ICV). This standard is prepared from a second source standard at a concentration of 2.0µg/L.

5.13.2 Continuing Calibration Verification Standard (CCV). This standard is prepared from the same source as the calibration curve and analyzed at 2.0 ug/L. The 2.0 µg/L calibration standard is used as the CCV.

5.14 Vendor List. The standards used in this test are purchased using the catalog numbers and vendors indicated below.

Table 5-2
Mercury Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
Mercury Standard	VHG	1000 µg/L	PHGN-100
Mercury Standard 2nd Source	Environmental Express	1000 µg/L	HP100033-1
Stannous Chloride	Fisher	Pure	T142-3
Hydroxylamine Hydrochloride	VWR	Pure	BDH0236-500G
Potassium Permanganate	Fisher	Pure	P279-212
Hydrochloric Acid	Fisher	Concentrated	A144C-212
Nitric Acid	Fisher	Concentrated	A200S-212

6.0 APPARATUS AND MATERIALS

6.1 Varian Spectra AA 220 spectrophotometer with SPS-5 auto-sampler and VGA77 mercury vapor generation system.

6.1.1 Mercury hollow cathode lamp or electrode-less discharge lamp.

6.1.2 Recorder: Any multi-range variable speed recorder that is compatible with the UV detection system is suitable.

6.1.3 Absorption Cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed of Plexiglas tubing, 1 in. O.D X 4.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of a Varian burner cleaning and alignment card. A target area is printed on the top and bottom of the left side of the card connected by a vertical line. The card should be placed so that the vertical line is at the center of the burner slot. Adjust the burner using the horizontal and vertical dials so that the light beam falls within the target area. This alignment gives the maximum transmittance.

6.1.4 Vapor generation assembly complete with flow meter and internal drying tube.

6.1.5 The cold vapor generator is assembled as shown in the Varian owner's manual. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system. Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system either to vent the mercury vapor out through an exhaust hood or to pass the vapor through an absorbing medium such as:

-Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4.
-0.25% Iodine in a 3% KI solution.
-Treated charcoal.

6.2 PE FIMS 100 Flow Injection Mercury Analysis System.

6.2.1 PC with AA WinLab software.

6.2.2 AS 90 Auto sampler.

6.3 Hot Block digestion system with digestion tubes or equivalent.

6.4 Assorted volumetric glassware as needed.

7.0 PROCEDURE

7.1 Preparation of digestion log form and digestion log in LIMS.

7.1.1 Each day the section supervisor prepares a work log. The log lists samples included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN DIGESTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received with a "rush" status will have a chain of custody delivered by the receiving technician to the section supervisor of the department that performs prep.

7.1.1.2 Prepare a written digestion log using the metals digestion logbook that is kept in the digestion area of the laboratory. The following entries must be made in the log.

7.1.1.2.1 Date and time the batch is opened or the date and time the sample(s) is placed on the hot plate.

7.1.1.2.2 All samples included in the digestion batch.

7.1.1.2.3 Volume or weight of samples digested.

7.1.1.2.4 Date and time the digestion is completed.

7.1.1.2.5 Digestion procedure employed.

7.1.1.2.6 The initials of the digestion analyst(s).

7.1.1.2.7 Laboratory number of all reagents used, including spiking standard and acids.

7.1.1.2.8 Final volume of all digestates.

7.1.1.2.9 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.1.1.3.1 Open a Prep Batch in LIMS by double clicking the "Prep" box or by pressing "Ctrl + b."

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS after the prep code is entered.

7.1.1.3.3 Select the Prep Code "7471A_S_P" for soil or "7471A_X_P" for waste from the pull down list. LIMS will automatically assign MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCS and enter the information.

7.1.1.3.4 Enter the technician's name from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this method. Use "User" to select the samples to be included in the batch for desired prep method.

7.1.1.3.6 The samples can be individually selected by highlighting the sample and clicking the "→" arrow or all samples can be selected by clicking the "⇒" arrow.

7.1.1.3.7 Samples that have been selected for re-analysis can be manually added to the sample list following the procedure outlined in section 7.1.1.3.3.

7.1.1.3.8 "Save" the batch by clicking on a previous batch number on the list and then return to the newly created batch.

7.1.2 Table 7-1 indicates the type of samples that comprise an analytical batch. **Note**: NELAC requirements specify that the maximum number of client samples in a prep batch can not exceed 20. Further, a prep batch can not be left "open" for a period that exceeds 24 hours.

Table 7-1
Samples in a NELAC Batch

Method Blank (MB)
LCS (and LCSD if no MS/MSD)
Client Samples
MS and MSD (If enough sample is supplied by client)

7.2 Sample preparation for soils and wastes. (See Tables 7-2 and 7-3 for Sample Preparation Checklists)

7.2.1 Print backlog report for the prep and each test code (7471A_S or 7471A_X).

7.2.2 Pick a sample for QC (Original, MS, and MSD).

7.2.3 Put samples in order and check prep and test info.

7.2.4 Add 0.5g of each sample to digestion vessel and record actual weight. For the sample chosen for MS/MSD, the initial weights used for the unspiked sample, matrix spike and matrix spike duplicate must be within 5% of each other.

7.2.5 Batch samples in LIMS and print a copy of the batch.

7.2.6 Prepare Cal Curve standards by adding appropriate amounts of **first source** 100ug/L Hg working standard to digestion tubes per Table 5-1 and adding DI water to a volume of 40ml. **Standard concentrations are based on the final digestate volume of 50ml**.

7.2.7 Prepare an ICV at 2.0µg/L by adding 1.0 ml of **second source** 100µg/L Hg working standard to a digestion vessel and adding DI water to a volume of 40 ml. **ICV standard must be from a different source than that used for the ICAL standards**.

7.2.8 Prepare the CRA at a concentration of 0.5 ug/L by adding 0.25 mL of 100 ug/L Hg working standard to a digestion vessel and adding DI water to a volume of 40 mL.

7.2.9 Prepare MB and LCS by adding 0.5g of clean sand to appropriate digestion tubes.

7.2.10 Spike LCS, MS and MSD tubes by adding 2.0 ml of the second source 100 µg/L Hg working standard used for the ICV. (**Witness Needed**)

7.2.11 Add approximately 5mL DI water to each tube containing soil or sand, and then add 1.25 mL of concentrated HNO_3 and 2ml of concentrated HCl to each digestion tube. Cover each tube with a watch glass and heat for 2 minutes at 95°C.

7.2.12 Cool then add 25 ml DI water and 7.5 ml of potassium permanganate, $KMnO_4$, to each vessel, re-cover each tube with a watch glass, mix and heat for 30 minutes at 95°C. **Samples must maintain purple color. Check throughout the heating process.** Add small amounts of $KMNO_4$ crystals if sample(s) appears to lose purple color.

7.2.13 Remove from heat and let cool. Add 3 drops of defoamer and one dropper full (approximately 3 mL) of hydroxylamine hydrochloride to reduce the excess permanganate. Bring to 50 mL final volume with DI water and mix thoroughly. If sample(s) still contains permanganate as indicated by purple color, small amounts of hydroxylamine crystals may be added.

7.3 Varian AA (FAA) System Operation.

7.3.1 Turn on the hood over the instrument. Power up the Varian instrument.

7.3.2 Click the Spectra AA icon to start the software. Select "Worksheet" then "New From" and highlight an old run to be used as the template. Name the new file as "Hg MM-DD-YYYYA, B, C, etc." Enter analyst initials and click "OK."

7.3.3 Open the cover and verify that the mercury lamp is on and allow it to warm up for at least 30 minutes. Lamp selection lever must be pointing to the correct lamp. If the lamp is not a Varian lamp, go to Lamp screen and verify the lamp settings, such as current, etc. Light the lamp from the lamp screen.

7.3.4 Attach the VGA 77 sampling system.

7.3.5 Attach Hg cell to screws on the burner and the black tube from the VGA 77 sampling system to the opening on the bottom of the Hg cell. Align the Hg cell with the lamp beam using the procedure outlined in 6.1.3.

7.3.6 Check the condition of the pump tubes and replace if necessary.

7.3.7 Check the sample, reductant, and acid flow by placing tubes and probe in DI water. If flow is consistent, place reductant tube in clear bottle labeled FAA $SnCl_2$, place acid tube in clear bottle labeled FAA Hg 3% Rinse, and place probe in 3% rinse solution on the auto sampler.

7.3.8 Click on the "Labels" tab. Type in your new sequence making sure that the old samples are deleted from the new sequence. When done, click "Analysis".

7.3.9 The analytical sequence is run in the following order:

-Cal Curve Standards
-ICV (Second Source)
-ICB
-CRA
-Up to 9 samples
-CCV
-CCB
-Up to 10 samples
-CCV
-CCB
-Repeat as necessary

7.3.10 Fill the appropriate tubes in the autosampler trays and place the trays on the sampler. Note that the system will not operate if the trays are not positioned properly in the sampler.

7.3.11 Click on "Optimize" icon, then click "OK". When the green bars light up indicating the lamp levels, select “Rescale.” Adjusting the horizontal dial (black knob) and vertical dial (brown knob) one at a time, click “Rescale” until adjusting the dials causes the green bars to be level with each other and close to 1.0. Click “OK” when done, then click “Cancel” on the small Optimize window.

7.3.12 Select the "Select" tool and highlight the sequence information on the screen, if the analysis does not include every sample typed in the sequence. Otherwise, click on "Start" to initiate the run.

7.3.13 The Spectra AA-220 system will automatically generate the calibration curve from the standards that were analyzed and calculate the sample values from the calibration curve.

7.4 FIMS System Operation. (See Table 7-5 for FIMS Loading Diagram)

7.4.1 Turn computer on, turn on the instrument, and double click the “AA Winlab Analyst” icon to start the software.

7.4.2 Click on “File” then “Open” then “Workspace” then double click on “hgfims.fms”. This opens all working windows needed for operation.

7.4.3 Click on “File” then “New” then “Sample Info File”.

7.4.4 Toggle to Batch ID and type in batch ID information as “YYMMDDA, B, C, etc”.

7.4.5 Tab to “Analyst” and type in analyst’s initials.

7.4.6 Tab to “Sample Volume” and enter appropriate value (50ml).

7.4.7 Tab to Auto-sampler location 9 and type in sample IDs beginning with the CRA.

7.4.8 After all samples have been entered, click on “File” then “Save As” then “Sample Info file”, enter file name as “YYMMDD” then the appropriate letter (A,B,C, etc.) then toggle to OK.

7.4.9 Minimize the Sample Info File.

7.4.10 In the Automated Analysis window, double click the test code and select method “7471A_S.” Click on Sample Information Browse and type in the

Sample Information File name and the Results Data Set name. Click on the "Use Entire Sample Info" box in the Automated Analysis Window. Then click on OK.

7.4.11 If you want the lamp to be turned off at the end of the run automatically, check the "Lamp" box.

7.4.12 Click on the "Analyze" tab at the bottom of the Automated Analysis window.

7.4.13 Click on "Analyses" at the top of the program window. Scroll down to "Auto-sampler Loading List" and click. Double check that all samples are present. Click "Print" then exit.

7.4.14 The loading list will appear on the screen as a pdf file. Verify that all samples are present on this list, also. Afterwards, minimize this page as data throughout the analysis will be written to this file.

7.4.15 Fill solution reagent bottles.
-Acid: clear bottle labeled FIMS 3% HCl Rinse with a volume of at least 1L
-Reductant: clear bottle labeled FIMS $SnCl_2$ with a volume of at least 1L.
(**YOU MUST HAVE ENOUGH $SnCl_2$ TO COMPLETE THE RUN. CHANGING OR ADDING $SnCl_2$ WILL REQUIRE RECALIBRATION)**
-DI Water Bottle

7.4.15 Check air filter and replace every month or more frequently if needed. Replace manifold filter daily, or, if changing methods in a single day, change filter before beginning the new method.

7.4.16 Connect the Tygon tubing on the FIMS 100 and snap clamps into place. Adjust clamp tightness if necessary.

7.4.17 Click on the "FIAS" toolbar.

7.4.18 Place the acid tube(blue/yellow) and the reductant tube(red) in a bottle of DI water. Place another container of DI water in wash position on autosampler tray and click "Move Probe Down/Up" to move the probe into the DI water. Click "Pump 1" icon to start the pump. Let the system rinse for about 15 minutes. Check that the flow is continuous for all four tubes. Click the "Pump 1" icon to stop the pump at the end of the rinse period.

7.4.19 Place the reductant tube into the $SnCl_2$ bottle and the acid tube into the 3% HCl bottle and close the FIAS window.

7.4.20 Load the auto-sampler tray with standards, QC and samples.

7.4.21 The analytical sequence is run in the following order:

-Cal Curve Standards
-ICB
-ICV (second source)
-CRA
-Up to 9 samples
-CCV
-CCB
-Up to 10 samples
-CCV
-CCB
-Repeat as necessary

7.4.22 Click "Calibrate" to begin the calibration. After verifying calibration is passing (correlation coefficient >= 0.995), click "Analyze Samples" to start sequence. Monitor the reagent bottles during analysis and refill if necessary. **DO NOT ALLOW THE REAGENT BOTTLES TO GO DRY. DO NOT REFILL $SnCl_2$ BOTTLE DURING ANALYSIS WITHOUT RECALIBRATING.**

7.4.23 After all samples have been analyzed, place the acid tube, reductant tube, and probe in DI Water using the method outlined in 7.4.18. Let the system rinse for about 5 minutes. Remove the lines and the probe from the DI water. Allow the pump to operate for 2-3 minutes to air purge the system. Click on the "Pump 1" icon to stop the pump after the air purge is complete.

7.4.24 Close the FIAS window.

7.4.25 Unsnap the tubing clamps, turn off FIMS, click on "File" then "Exit" then turn off computer.

7.4.26 See Table 7-6 for FIMS Data Exporting Instructions.

Table 7-2
7471A_S_P
(7471A_S)

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (7471A_S)

____ : PICK A SAMPLE FOR QC (ORIGINAL, MS, AND MSD)

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ADD 0.5g OF EACH SAMPLE TO DIGESTION VESSEL. **For the sample chosen for MS/MSD, the initial weights used for the for the unspiked sample, matrix spike and matrix spike duplicate must be within 5% of each other.**

____ : BATCH SAMPLE IN THE LIMS, PRINT A COPY OF THE BATCH

____ : MAKE ICAL STANDARDS FRESH DAILY AT 0.16, 0.5, 1.0, 2.0, 5.0, 10.0 µg/L **BASED ON THE FINAL DIGESTATE VOLUME OF 50 mL** BY ADDING 0.08, 0.25, 0.50, 1.0, 2.5 AND 5.0 mL OF THE 100 µg/L Hg STANDARD AND SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 mL IN THE DIGESTION VESSELS.

____ : PREPARE AN ICV AT 2.0 µg/L BY ADDING 1.0 mL OF SECOND SOURCE 100 µg/L Hg WORKING STANDARD AND SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 mL IN THE DIGESTION VESSEL. **STANDARD MUST BE FROM A DIFFERENT SOURCE THAN THAT USED FOR THE ICAL STANDARDS.**

____ **:** PREPARE THE CRA AT A CONCENTRATION OF 0.5µg/L BY ADDING 0.25mL of 100µg/L Hg WORKING STANDARD TO A DIGESTION VESSEL AND ADDING DI WATER TO A VOLUME OF 40Ml.

____ : ADD 0.5g OF CLEAN SAND TO THE LCS & MB DIGESTION VESSELS.

____ : SPIKE LCS, MS & MSD WITH 2.0 mL OF THE 100 µg/L Hg WORKING STANDARD USED TO PREPARE THE ICV. (WITNESS NEEDED)

____ : ADD APPROX. 5mL DI WATER TO EACH TUBE CONTAINING SOIL OR SAND

____ : ADD 1.25 mL HNO_3 AND 2 mL HCl THEN HEAT FOR 2 MINUTES AT 95°C.

____ : COOL AND ADD 25 mL DI WATERTO EACH TUBE CONGTAINING SOIL OR SAND AND 7.5 mL KMNO HEAT FOR 30 MINUTES AT 95°C. **SAMPLES MUST MAINTAIN PURPLE COLOR**, ADD ADDITIONAL KMNO3 AS CRYSTALS, IF NECESSARY.

____ : REMOVE FROM HEAT AND LET COOL

____ : LINE UP SAMPLES IN ORDER AS THEY APPEAR IN LOG

Batch folders

____ **:** Does it have BACKLOG REPORT FOR THE PREP AND EACH TEST CODE

____ : Is the SAMPLE FOR QC (DUP, MS) adequate

____ : ARE the SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : Check SAMPLE final volume in DIGESTION VESSEL, does it match Recorded VOLUME

____ : CHECK prep log against BATCH IN THE LIMS (sample prep, test code, date time, analysts, check sample ID's, add DUP & MS/MSD or LCSD, check volumes), or print A COPY OF THE BATCH

Table 7-3
7471A_X_P

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (7471A_X)

____ : PICK A SAMPLE FOR QC (ORIGINAL, MS, AND MSD)

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ADD 0.5g OF EACH SAMPLE TO DIGESTION VESSEL AND RECORD ACTUAL WEIGHT. **For the sample chosen for MS/MSD, the initial weights used for the for the unspiked sample, matrix spike and matrix spike duplicate must be within 5% of each other.**

____ : BATCH SAMPLE IN THE LIMS, PRINT A COPY OF THE BATCH

____ : MAKE ICAL STANDARDS FRESH DAILY AT 0.16, 0.5, 1.0, 2.0, 5.0, 10.0 µg/L **BASED ON THE FINAL DIGESTATE VOLUME OF 50 mL** BY ADDING 0.08, 0.25, 0.50, 1.0, 2.5 AND 5.0 mL OF THE 100 µg/L Hg STANDARD AND SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 mL IN THE DIGESTION VESSELS.

____ : PREPARE AN ICV AT 2.0 µg/L BY ADDING 1.0 mL OF SECOND SOURCE 100 µg/L Hg WORKING STANDARD AND SUFFICIENT DI WATER TO BRING THE VOLUME TO 40 mL IN THE DIGESTION VESSEL. **STANDARD MUST BE FROM A DIFFERENT SOURCE THAN THAT USED FOR THE ICAL STANDARDS.**

____ **:** PREPARE THE CRA AT A CONCENTRATION OF 0.5µg/L BY ADDING 0.25mL of 100µg/L Hg WORKING STANDARD TO A DIGESTION VESSEL AND ADDING DI WATER TO A VOLUME OF 40Ml.

____ : ADD 0.5g OF CLEAN SAND TO THE LCS & MB DIGESTION VESSELS. **ADD APPROX. 5mL DI WATER TO MB & LCS VESSELS ONLY.**

____ : SPIKE LCS, MS & MSD WITH 2.0 mL OF THE 100 µg/L Hg WORKING STANDARD USED TO PREPARE THE ICV. (WITNESS NEEDED)

____ : ADD 1.25 mL HNO_3 AND 2 mL HCl THEN HEAT FOR 2 MINUTES AT 95°C.

____ COOL AND ADD 25 mL DI WATER TO EACH TUBE CONTAINING SOIL OR SAND AND 7.5 mL KMNO. HEAT FOR 30 MINUTES AT 95°C. **SAMPLES MUST MAINTAIN PURPLE COLOR**, ADD ADDITIONAL KMNO3 AS CRYSTALS, IF NECESSARY.

____ : REMOVE FROM HEAT AND LET COOL

____ : LINE UP SAMPLES IN ORDER AS THEY APPEAR IN LOG

Batch folders

____ **:** Does it have BACKLOG REPORT FOR THE PREP AND EACH TEST CODE

____ : Is the SAMPLE FOR QC (DUP, MS) adequate

____ : ARE the SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : Check SAMPLE final volume in DIGESTION VESSEL, does it match Recorded VOLUME

____ : CHECK prep log against BATCH IN THE LIMS (sample prep, test code, date time, analysts, check sample IDs, add DUP & MS/MSD or LCSD, check volumes), or print A COPY OF THE BATCH

Table 7-4

DATA REVIEW CHECKLIST- Hg by FIMS/FAA

Batch ID: _____ Data File:_____

QA Analyst

INITIAL RAW DATA REVIEW

☐ ☐: Have prep logs, prep checklists, raw data and run log(s) been printed to pdf and posted to Portal Server
☐ ☐: Are all instrument standards used listed on raw data
☐ ☐: Do calibration curves for all target analytes meet requirements where (≥0.995 Corr.Coef.)
☐ ☐: Are all replicated RSDs≤15% for all values over PQL? **No requires reanalysis and CAR if still out at reanalysis**

GO TO LIMS "MAIN" RUN SCREEN

☐ ☐: Has the LIMS Prep batch been properly entered matching the prep log book and closed
☐ ☐: Are any CARs listed in comments box **(Yes requires determining possible actions before continuing)**
☐ ☐: Are all Sample IDs properly assigned per Backlog Report (Double click each SampleID to verify)
☐ ☐: Are all Test Codes properly assigned per Backlog Report
☐ ☐: Is instrument QC run at the required frequencies
☐ ☐: Are all Sample Types properly assigned
☐ ☐: Are all samples linked to the Prep Batch properly with correct PFac, SpkFac and OFac
☐ ☐: Are dilution factors entered correctly per the raw data Run Log
☐ ☐: Are all Blkref, SPKref, RPDref and CCVref assigned correctly
☐ ☐: Are there any Comments present that require attention? **May require CAR**

GO TO DATA SCREEN

☐ ☐: Calculate Sequence (Cal SEQ tab) to insure LIMS calculations are completed.
☐ ☐: Are there any S or B flags for element of interest on the Instrument QC (ICV, CRA, CCV)? **Yes Requires CAR**
☐ ☐: Are all values for target analytes between <PQL and > -PQL on ICB, CCB and MBs? **No Requires CAR**
☐ ☐: Are there any B flags for elements of interest on any samples and/or Batch QC? **Yes Requires CAR**
☐ ☐: Are there any S and/or R flags for elements of interest on Batch QC (LCS/D, MS/D, PDS)? **Yes Requires CAR except R or S flags on PDS caused by value at or near PQL or insignificant spike.**
☐ ☐: Are there any H flags present for any samples? **Yes Requires CAR**
☐ ☐: Are there any E flags for elements of interest on any samples and/or Batch QC selected for reporting? **Yes Requires CAR**
☐ ☐: Are there any J flags on any target analytes selected to report on diluted sample runs? **Yes Requires reanalysis at lower dilution or CAR to narrate elevated reporting limits.**
☐ ☐: Have readings been entered properly (check at least 2 entries for downloaded data, **ALL** entries for manual data)
☐ ☐: Have at least 2 sample's final results in LIMS been verified by hand calculation
☐ NA: Have **ALL** CARs been closed and narratives written prior to QA?
☐ NA: Have PM, Lab Manager and PM Director been notified for any CAR resulting in reextract and/or due date exceedance?

CAR#: _____ _____ _____ _____

Analyst Signature: _____Date: _____ Time: _____

Reviewer Signature: _____ Date: _____ Time: _____

Table 7-5
FIMS Loading Instruction Diagram

I. To open a "work space"

1. Workspace HgFIMS.fms

or

2. File Open- Workspace- HgFIMS.fms

II. Create a New autosampler loading list

1. File New- Sample info file
Batch ID: YYMMDD "letter A,B,C,…" Analyst: Intials
Sample Vol: 50.0

1
2
3
4
5
6
7
8
9 CRA
10 MB-
11 LCS-
12 Sample (or LCSD, if no MS/MSD)
13 MS (or Sample if using LCSD)
14 MSD (or Sample if using LCSD)
15 PDS (or Sample if using LCSD)

File Save as- Sample info file- YYMMDD "letter A,B,C,…"

III. Run setup

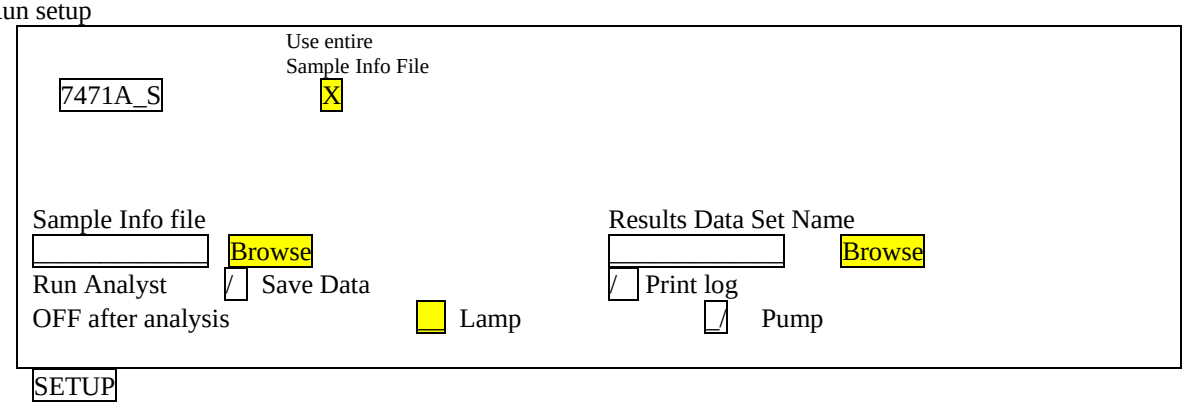
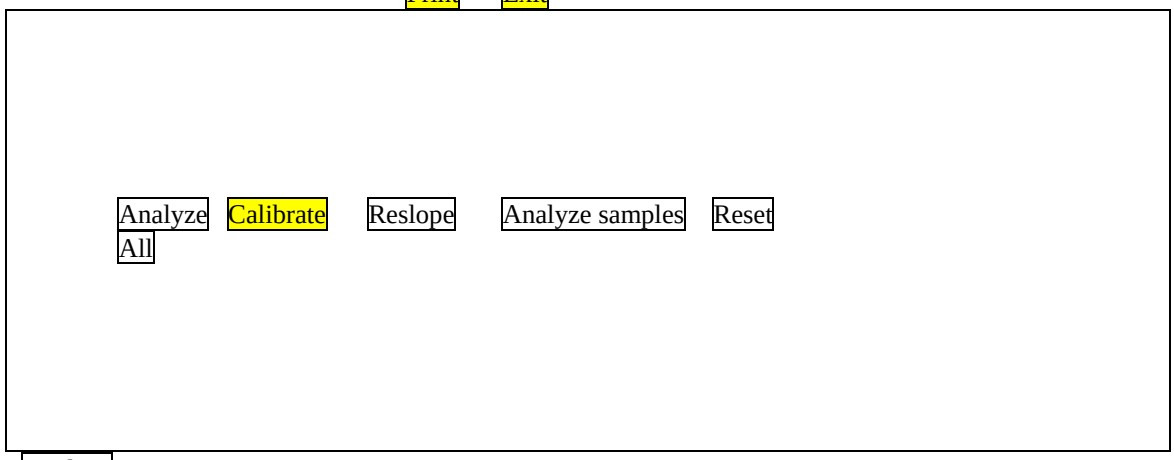


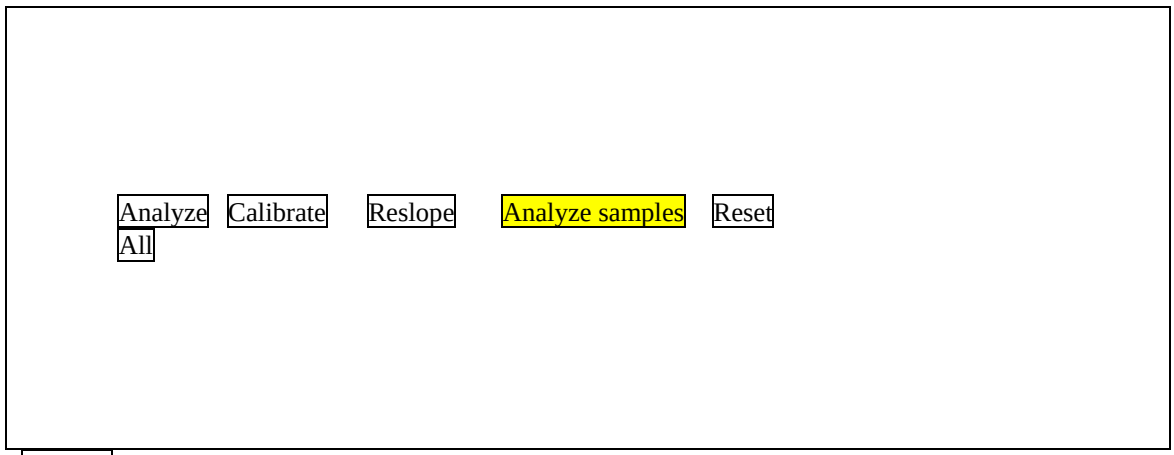
Table 7-5 (Cont)
FIMS Loading Instruction Diagram

1. Under Sample Info file, use Browse to select the autosampler file that you just saved
2. Under Results Data Set, use Browse and then type in the name of the file that you want the data stored in. File name is usually the same as Sample Info File.
3. Click on the box under use entire sample info file
4. If you are putting a run on to finish after you go home, click on the Lamp box

Switch to the Analyze page- Analyses- Autosampler loading list
Print Exit



Analyze
1. Select Calibrate, the program will stop after calibration is complete



Analyze
1. Select Analyze samples after a passing calibration

IV. Adding to Autosampler loading list

1. File Open- Sample Info File- open file that you want to add to and type in the additional samples.
2. File Save

Table 7-6
FIMS Data Exporting Instruction Diagram

I. [START]-PROGRAMS-FIMS-AA Winlab reformat
[open design]
[export]-Double check
[browse]-data set name, type in file name, then select "OK." Change extension to ".TXT."
[save results]
[OK]
X -close window
[OK]

II. My computer-C:/OmegaME/Fims_export
Enable Macros
Run macro (running man)
Type test code(s)-(7471A_S or 7471A_X)
[OK]
Delete curve and unused samples-change IDs-change test codes if needed
Run macro <> (diamond)
[OK]
[OK]
X Exit excel
[YES]

III. Omega SQL - LIMS (Make sure that the prep batch is closed)
[Data entry]
[Add]

Instrument ID:	FIMS
Run Start Date/time:	MM/DD/YYYY
Analyst:	Your name

[Data Import]
Specification
FIMS
C:/omegaME/FimsData
[OK]

Table 7-7
FAA Loading Instructions

1. Double click on the SpectraAA icon on the desktop.
2. Select "Worksheet" then "New From" and highlight the most recent Hg run to be used as the template.
3. Name the new file as "Hg MM-DD-YYYY [A, B, C, etc.]" then enter initials and click OK.
4. Click on the "Labels" tab. Type in the new sequence as follows:
   - ICV
   - ICB
   - CRA 0.5 ppb
   - MB
   - LCS
   - Up to 7 samples
   - CCV
   - CCB
   - Up to 10 samples
   - CCV
   - CCB
   - Repeat as necessary

5. Rows can be added by clicking on "Total Rows…" and typing in required number of rows.
6. Save the loading list by clicking on the "Loading Guide" tab to the left of the screen.
7. The loading list will pop-up. Click the "Print" tab at the bottom.
8. Make sure "Adobe PDF" is selected from the drop down menu and click "OK"
9. Save the file under Desktop → FAA Analysis → Mercury → and save in this format "Hg MM-DD-YYYY [A, B, C, etc.] _Run Log"
10. A PDF file of the loading list will pop-up. At this point you can print the loading list by selecting File→ Print → from the drop down menu. Under "Printer", select HP LaserJet 2100 and click OK.
11. Once the loading list is complete, saved, and printed, select the "Analysis" tab. All of the samples should be highlighted in red as an indication to be analyzed.
    A. To not analyze a sample simply click on the "Select" tab to the left of the screen.
    B. The cursor should change into a highlighter icon. Click on the sample you wish to not analyze and the sample should be a white block.
    C. Once done un-selecting samples click on the "Select" tab again.
12. Begin analysis by clicking the "Start" icon to the left of the screen. Then click "OK" on the next pop-up screen.
13. To re-analyze a sample click the "Pause" icon to the left of the screen. Once the probe has finished its rinse cycle, click on the "Select" icon and then highlight the sample(s) needed to be re-analyzed then click the "Select" icon once again to return to the main analysis screen.
14. At the top of the screen select "Instrument" and click "Start At…"
15. From the drop down menu select "Solution" and click Start.
16. Click "OK" on the "Analysis Checklist" pop-up screen if it appears.

Table 7-8
Exporting FAA Hg Data into LIMS

1. Click reports (printer icon) at the top of the SpectraAA 220 program.
   - Select the data set to be imported.

2. Select the Settings tab and make sure only the following items are checked under Solution Data and Report Contents:

Concentration	Date	Dilution Factors
%RSD	Time	
	Overrange Dilution Factor	

3. Select the Report tab and click Export to PRN file…
   Save the file name same as the worksheet name and click Open and close out of Reports.

4. Open "FAA_Export.xls" on the desktop of the FAA computer. Click Enable Macros.

5. If the spreadsheet contains sample data, click the "Clear Data" button.

6. Click "Import Data" → Double click My Computer → Local Disk: (C) → Varian → sp100 → Data → Scroll over to select data set.

7. Click "Set Test" and enter the appropriate test code for the samples to be imported.

8. Using the row selector (the column of numbered boxes to the left of the cells), select the data not needed. Right-click the selection and select "Delete".

9. If any samples contain dilution information in the Sample ID, enter that dilution under the DF column and delete it from the Sample ID.

10. Using the row selector, select all rows containing samples to be imported into LIMS.

11. Click "Export" to transfer all selected data to the "FAA_DATA.xls" spreadsheet. And click **yes** to replace the file.

12. The FAA_Data.xls spreadsheet will remain open, allowing the user to review the data transferred. If all desired samples are listed, close the FAA_DATA.xls spreadsheet and click **no** when asked to save changes.

13. In LIMS, add an entry under Data Entry. Then click the Data Import button. Select the "FAA" specification and click "Run Import".

14. Click on My Computer → Local Disk: (C) → OmegaME → Open file "FAA_DATA.xls" → Open → OK.

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP# QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Initial Calibration: This is accomplished through a 5-point calibration curve. The points on the curve must meet a 10 %RSD when comparing calibration factors or have linear regression correlation coefficient of $\geq$0.995 to determine if the calibration curve is linear. Initial verification of the curve must be performed using a second source standard. The ICV must recover with $\pm$10% of the true value or recalibration is required. The calibration must be continuously verified by analyzing a CCV and CCB at least every 10 samples and to close the sequence. CCV recovery must be $\pm$10% of true value.

8.1.3 Method Detection Limit Study. The method detection limit is calculated by analyzing at least seven replicates prepared in blank water at concentrations 1 to 5 times higher than the estimated detection limit. Quantitation limits are laboratory derived from the MDL study data set. MDL's are to be performed initially and whenever instrument conditions have changed such that the established MDLs may have been affected.

8.1.4 Method blank. Reagent blank analysis must be performed at the following frequency: Every twenty (20) samples of similar concentration and/or sample matrix or whenever samples are extracted and analyzed by the same procedure at same time, whichever is more frequent. The concentration of the method blank of any analyte of interest should not exceed the laboratory established practical quantitation limit (PQL).

8.1.5 Laboratory Control Sample (LCS). The LCS is used to monitor, assess, and document laboratory method performance and is performed on every batch or twenty samples, whichever occurs more frequently. The recovery of the analytes must meet established laboratory guidelines.

8.1.6 Sample spike. Matrix spikes are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples. The recovery of the analytes must meet established laboratory guidelines.

8.1.7 Duplicate Sample. One sample per analytical batch is digested and analyzed in duplicate. The results are compared and must have an RPD of 0 - 30% for soil samples. The results are only considered valid if the concentration of the sample is at least 4 times the MDL.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP# QA-01000, "Quality Assurance Manual," in Section 5. Included are tables that detail corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance, and any procedure deviations in appropriate logbooks.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 Health and Safety: Safety glasses and latex type gloves must be worn at all times when dealing with any chemicals, samples, or reagents. A lab coat is also highly recommended. Close-toed shoes and clothing that covers the legs (no shorts or dresses) must be worn any time an analyst is working in the laboratory.

9.2 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a health hazard and exposure should be kept as low as reasonably possible. All health and safety concerns for any chemicals are listed in the Material Safety Data Sheets (MSDS) provided by the supplier or manufacturer of these chemicals. A copy of any MSDS is available for review at any time.

9.3 The exhaust hoods should be maintained in a clean condition. Avoid getting kimwipes or other paper inside the hood stack. This is not only a fire hazard but can also decrease the flow within the hood.

9.4 Acids should be handled with care. Always add acids and caustic solutions to water.

10.0 DATA REPORTING

10.1 The LIMS system automatically rounds the data based upon factors that are set up for each test category. Typically, the LIMS reports to two significant figures.

10.2 The reporting limits can be changed in LIMS. Various laboratory personnel including project managers have the rights to change the limits per the requirements of the clients. Reporting limits are based upon the MDLs developed for each test.

10.3 The MDL is defined in 40 CRF Part 136, Appendix B. The default reporting limits indicated in LIMS are outlined in tables in Section 5 of SOP# QA-01000, "Quality Assurance Manual".

10.4 Out-Of-Control Data - Contingencies for handling out-of-control or unacceptable data are included in SOP #QA-01000, "Quality Assurance Manual," of the in-house method manual in Section 5 including corrective actions for failing QC and/or acceptance criteria.

10.5 Calculations

10.5.1 Reagent blanks should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

10.5.2 If dilutions were performed, the appropriate factor must be applied to the sample values.

10.5.3 The instrument will automatically calculate the concentration in the digestate and report it on the report.

10.5.4 Data import to LIMS. Data is automatically entered into the LIMS system.

11.0 FILE MAINTENANCE

11.1 Data is stored on the portal server for a period of 5 years. The data contained in the folders consists of the following.

MB (Digested)
CCV sample and calculated results
LCS/LCSD sample and calculated results
MS/MSD sample and calculated results
Actual samples and calculated results
Daily run log

11.2 Each file folder is labeled with the following information.

Batch number (example – automatically LIMS generated): #8203
Digestion method: 3050B_X where
X = matrix = waste
W = matrix = water
S = matrix = soil

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks. Instrument logbooks must be completed each time that any maintenance is performed on the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as replacement of motors in tower autosamplers. Tables 12-1 and 12-2 are examples of the FIMS and FAA logbook pages.

12.2 Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.3 Routine Maintenance: Typical routine maintenance consists of keeping the system clean and insuring that all generated QC remains acceptable. Examples would be cleaning of the torch and sample introduction lines.

12.4 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as autosampler that will not function. If this type of problem occurs, contact the department manager for assistance.

Table 12-1
Routine Maintenance Log for PE FIMS

AES, Inc.
3785 Presidential Pkwy
Atlanta, GA 30340

February 2008 **FIMS 1429**

Day	1	2	3	4	5	6	7	8
Date	F	SA	SU	M	T	W	H	F
Routine Maintenance								
Daily Visual inspection:								
Tubing								
Filter								
Exhaust fans on								
gas pressure (at least 100psi)								
Regular Maintenance:								
Replace tubing								
Replace filter								
Remake Stannous Chloride solution								
Replace rinse								
Waste discarded (daily)								
Rinse Instrument w/ 8% concentrated acid								
Inspect cell body, make sure no moisture is present								
Weekly:								
Instrument/autosampler wiped down								
Change charcoal								
Monthly Maintenance:								
Inspect lamp (as needed)								
Comments (Please note with an *)								
Analyst								

Table 12-2
Routine Maintenance Log for Varian FAA

Analytical Environmental Services, Inc.
3785 Presidential Parkway
Atlanta, GA 30340

February 2008 **FAA AES1148**

Day	F	SA	SU	M	T	W	H
Date	1	2	3	4	5	6	7
Routine Maintenance							
Daily Visual inspection:							
Pump tubing							
Burner Head							
Exhaust fans on							
Check Acetylene							
Check lamp (no arcing)							
Regular Maintenance:							
Pump tubing conditioned (30 min. minimum)							
Rinse station filled							
Acetylene replaced (if below 100psi)							
For Hg only - rinse lines with DI H_2O after analysis							
Cover exhaust on instrument with cardboard at end of analysis (daily)							
Weekly:							
Instrument/autosampler wiped down							
Waste discarded							
Standard tubes cleaned							
Tubing replaced							
Monthly Maintenance:							
Burner Head cleaned							
Condition of lead lamp checked							
Nebulizer sonicated							
Glass bead checked							
Spray chamber and liquid trap cleaned							
Nebulizer flow optimized							
O-rings checked							
Comments (Please note with an *)							
Analyst							

13.0 METHOD PERFORMANCE

13.1 The method detection limit (MDL) is defined in 40CFR Part 136, Appendix B as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

13.2 This method is recommended for use in the concentration range from the MDL to 100 x MDL.

13.3 For more method performance information, see referenced method.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 SOP HS-03005 Waste Disposal and SOP SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to distilled water or clean soil and the concentration is measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.2 RSD – Relative Standard Deviation

15.3 MS- Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.4 MSD- Matrix Spike Duplicate.

15.5 CCV - Continuing calibration verification standard.

15.6 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot than the calibration standard.

15.7 LCSD - Laboratory Control Sample Duplicate

15.8 Dissolved - Those elements which will pass thorough a 0.45 µm membrane filter

15.9 Suspended - Those elements which are retained by a 0.45 µm membrane filter.

15.10 Total – The concentration determined on an unfiltered sample following vigorous digestion or the sum of the dissolved plus suspended concentrations.

15.11 Total Recoverable – The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, SW-846, Method 7471B, Test Procedures for Evaluating Solid Wastes, Rev. 2, February 2007.

16.2 U.S. Environmental Protection Agency, SW-846, Method 7000B, Flame Atomic Absorption Spectrophotometry, Rev. 2, February 2007.

16.3 U.S. Environmental Protection Agency, SW-846, Method 7010, Graphite Furnace Atomic Absorption Spectrophotometry, Rev. 0, February 2007.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://home/aes/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://home/aes/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

AES, Inc.
3785 Presidential Pkwy.
Atlanta, GA 30340

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DOCUMENT TITLE: STANDARD OPERATING PROCEDURE FOR 1,2-DIBROMOETHANE(EDB) AND 1,2-DIBROMO-3-CHLOROPROPANE(DBCP) BY EPA SW-846 METHOD 8011

DOCUMENT CONTROL NUMBER: Rev. *9*

DOCUMENT DISTRIBUTION NUMBER: OA-11007

ELECTRONIC DOCUMENT LOCATION
AES Portal Server: http://portal/Procedures/Standard Operating Procedures

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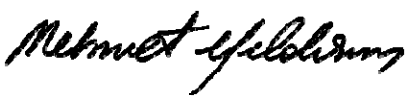
Laboratory Manager:  Date: 2/8/2012

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Department Supervisor:  Date: 2/8/2012

STANDARD OPERATING PROCEDURE FOR
1,2-EDB AND 1,2-DCBP BY EPA SW-846 METHOD 8011

TABLE OF CONTENTS

Page

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentration of the following compounds in drinking and ground water:

Compound Name	CAS No.
1,2-Dibromoethane (EDB)	106-93-4
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8

1.2 The experimentally determined method detection limits (MDL) for EDB and DBCP were calculated to be 0.01 µg/L. The method has been shown to be useful for these analytes over a concentration range of a proximately 0.02 to 200 µg/L. Actual detection limits are highly dependent upon the characteristics of the GC system, sample matrix, and calibration.

1.3 This method is restricted to use by or under the Supervision of analysts experienced in the use of gas chromatography and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 7.0.

1.4 1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane have been tentatively classified known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MES approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

2.0 SUMMARY OF METHOD

2.1 Thirty five- (35) mL of sample are extracted with 2 mL of hexane. 1-5 µl, of the extract are then injected into a gas chromatograph equipped with a linearized electron capture detector (ECD) for separation and analysis. Aqueous matrix spikes are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses.

2.2 The extraction and analysis time is 30 to 50 minutes per sample depending upon the analytical conditions chosen. See Table 7-2.

2.3 Results are confirmed through the use of a second column and separate ECD detector.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contamination in solvents, reagents, glassware, and other sample processing hardware, that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

3.1.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems.

3.1.3 Impurities contained in the extracting solvent (hexane) usually account for the majority of the analytical problems. Reagent blanks should be analyzed as part of each analytical batch.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending on the nature and diversity of the waste being sampled.

3.3 Several instances of accidental sample contamination have been attributed to diffusion of volatile organics through the septum seal into the sample bottle during shipment and storage. Trip blanks must be used to monitor for this problem.

3.4 This liquid/liquid extraction technique extracts a wide boiling range of non-polar organic compounds and, in addition, extracts some polar organic compounds.

3.5 EDB at low concentrations may be masked by very high concentrations of dibromochloromethane (DBCM), a common chlorinated drink water contaminant, when using the confirmation column.

4.0 SAMPLE COLLECTION, PRESERVATION AND HOLDING TIMES

4.1 Water samples must be extracted and analyzed within 14 days of sample collection. Water samples must be stored in the dark at $\leq$6°C.

4.2 Store all extracts at $\leq 6^{o}$ in the dark in Teflon-sealed containers until all analyses are performed.

5.0 REAGENTS AND STANDARDS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall confirm to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of this determination.

5.2 Organic-free reagent water - ASTM type III reagent grade water.

5.3 Methanol, CHOH. Pesticide quality or equivalent. Store apart from other solvents.

5.4 Hexane, C_6H_{14}- Pesticide quality or equivalent. Store apart from other solvents.

5.5 Sodium chloride NaCl - pulverize a batch of NaCl and place it in a muffle furnace at room temperature. Increase the temperature to 400°C and heat overnight. Store in a capped bottle.

5.6 4-Bromofluorobenzene Surrogate Stock Solution, 2000ug/ml. Purchased from Ultra. Stock standards are stored at ≤6°C in the dark in Teflon-sealed containers. Once opened, the stock standard is stable for 6 months.

5.7 Surrogate Working Solution, 7,000ug/L. Dilute 35ul of Surrogate Stock Solution (5.6) to 10.0 ml in methanol. This standard is stored at ≤6°C in the dark in Teflon-sealed containers. Surrogate Working Solution is stable for 6 months or sooner if chromatography indicates degradation. The expiration date of the Surrogate Working Solution cannot exceed the expiration date of the Surrogate Stock Solution.

5.8 Dibromochloromethane, DBCM: Dibromochloromethane, 100 µg/ml purchased from Ultra Scientific. Stock standards are stored at ≤6°C in the dark in Teflon-sealed containers. Once opened, stock standard is stable for four weeks.

5.8.1 DBCM Intermediate stock: Dilute 1mL of the DBCM to 10 ml with methanol. The concentration of this standard is 10,000 µg/l. Intermediate standards are stored at ≤6°C in the dark in Teflon-sealed containers. The expiration date of the standard is four weeks and cannot exceed the expiration date of the opened DBCM standard used to prepare the intermediate mix.

5.8.2 DBCM Working standard. Dilute 35 µl of the Intermediate stock standard to 1.0 ml with methanol. The concentration of this standard is 350µg/l. Working standards must be made fresh daily. 10uL of the working standard added to 35mL of water and extracted as in section 7.2. The final concentration injected will be 0.1ug/L.

5.9 8011 Stock Calibration Mix, 2000 µg/ml each component. Purchased from Ultra. Stock standards are stored at ≤6°C in the dark in Teflon-sealed containers. Once opened, stock standard is stable for four weeks.

5.10 8011 Intermediate Calibration Mix. Dilute 50 µl the Calibration Mix stock standard (5.6) to 10 ml with methanol. The concentration of this standard is 10,000 µg/l of each component. Intermediate standards are stored at ≤6°C in the dark in Teflon-sealed containers. The expiration date of the standard is four weeks and cannot exceed the expiration date of the opened 8011 Stock Calibration Mix used to prepare the intermediate mix.

5.11 8011 Working Calibration Mix. Dilute 35 µl of the Intermediate Calibration Mix standard (5.7) to 1.0 ml with methanol. The concentration of this standard is 350 µg/l of each component. Working standards must be made fresh daily.

5.12 8011 Stock Second Source Mix, 2000 µg/ml each component. Purchased from Restek. Stock standards are stored at ≤6°C in the dark in Teflon-sealed containers. Once opened, stock standard is stable for four weeks.

5.13 8011 Intermediate Second Source Mix. Dilute 50 µl of the Second Source Mix stock standard (5.9) to 10 ml with methanol. The concentration of this standard is 10,000 µg/l of each component. Intermediate standards are stored at ≤6°C in the dark in Teflon-sealed containers. The expiration date of the standard is four weeks and cannot exceed the expiration date of the opened 8011 Stock Second Source Mix used to prepare the intermediate mix.

5.14 8011 Working Second Source Mix. Dilute 35 µl the Intermediate Second Source Mix standard(5.10) to 1.0 ml with methanol. The concentration of this standard is 350 µg/l of each component. Working standards are used immediately and stored in amber Teflon-sealed containers. Working standard must be made fresh daily.

5.15 Calibration Curve. Dilute the 8011 Working Calibration Mix (5.8) as indicated in Table 5-1. **All aqueous standards must be discarded and remade after 8 hours.**

Table 5-1
Calibration Standard Preparation

Amount Stock Standard Added to 35 mL Water	Working Calibration Standard(5.8) Conc. (µg/L)	Initial Calibration Standard Concentration (µg/L)
2.0 µL	350	0.02
3.0 µL	350	0.03
5.0 µL	350	0.05
10 µL	350	0.10
15 µL	350	0.15
20 µL	350	0.20
25 µL	350	0.25

5.16 Final Second Source Standards. Two different second source standards are prepared as follows: (Either standard may be considered an ICV or CCV)

5.16.1 QC Reference Standard. 0.25ug/L. Add 25 uL of Working Second Source Mix (5.11) to 35 ml of DI water and extract per Section 7.

5.16.2 QC Check Standard. 0.10ug/L. Add 10 uL of Working Second Source Mix (5.11) to 35 ml of DI water and extract per Section 7.

5.17 Vendor List

The standards used in this test are purchased through VWR scientific using the catalog numbers and vendors indicated below.

Table 5-2
Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
8011 Calibration Mix	Ultra Scientific	2000 µg/ml	HCM-812
8011 Second Source Mix	Restek	2000 µg/ml	30062
4-BFB Surrogate Solution	Ultra Scientific	2000 µg/ml	STS-110N
Hexane	Baker Ultra	Ultra pure	JT9262-2
Methanol	Omnisolve	P&T Grade	MX0482-6
Sodium Chloride	Fisher	ACS Reagent	S271-3
Dibromochloromethane (DBCM)	Ultra	100ug/mL	HC-100

6.0 APPARATUS AND MATERIALS

6.1 Gas Chromatograph - Hewlett Packard model 5890 Series IIGas Chromatograph, equipped with a Hewlett Packard 7673 or 7673A autosampler or equivalent.

6.1.1 Column 1: J&W DX-3, 30m, 0.32mm ID, 0.25um or equivalent.

6.1.2 Column 2: J&W DB-1, 30m, 0.32mm ID, 0.25um or equivalent.

6.1.3 Hewlett Packard Detectors – Dual Electron Capture Detectors.

6.1.4 Hewlett Packard Chemstation with Enviroquant reporting software or equivalent.

6.2 Balance - Analytical, capable of accurately weighing 0.0001 g.

6.3 Microsyringes - 10 µL, 25 µL, 100 µL, 250 µL, 500 µL, and 1,000 µL.

6.4 Syringe - gas-tight. Various sizes

6.5 2-mL crimp top vial.

6.6 Disposable pipettes- 1 mL.

6.7 Graduated cylinder - 50 mL.

6.8 Volumetric flask - 10 mL.

6.9 Sample container with cap - 43 mL (VOC vial).

6.10 Carrier Gas, Helium - 99.99% or higher purity.

6.11 Make-up Gas (P-5), 5% Methane, 95% Argon

7.0 PROCEDURE

7.1 Preparation of extraction log form and extraction log in LIMS.

7.1.1 Each day the section supervisor prepares a work log. The log lists samples that are included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN EXTRACTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received in a “rush” status will have a chain of custody delivered to the extraction supervisor by sample receiving.

7.1.1.2 Prepare a written extraction log using the 8011 logbook that is kept in the extraction supervisor’s office. The following entries must be made in the log.

7.1.1.2.1 Date and time that the batch is opened or the date and time the liquid/liquid extraction system or Soxhlet extraction system is started.

7.1.1.2.2 All sample(s) included in the extraction batch.

7.1.1.2.3 Volume or weight of samples extracted.

7.1.1.2.4 Date and time that the liquid/liquid extraction or Soxhlet extraction is completed.

7.1.1.2.5 Extraction procedure employed.

7.1.1.2.6 The initials of the extraction analysts.

7.1.1.2.7 Laboratory number of all reagents used including surrogate standard, spiking standard, hexane, methanol, and sodium sulfate used.

7.1.1.2.8 Volume of all reagents used including surrogate standard, spiking standard, hexane and methanol.

7.1.1.2.9 Final volume of all concentrates.

7.1.1.2.10 Date and time the batch is closed.

7.1.1.2.11 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure:

7.1.1.3.1 Open Prep Batch in LIMS by double clicking the "Prep" box.

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS.

7.1.1.3.3 Select the Prep Code "8011_Prep" from the pull down list. The LIMS will automatically assign an MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCSD and enter the information.

7.1.1.3.4 Enter the technician initials from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this preparation method. Note that the list contains all samples requiring extraction by the various methods that have been logged into the LIMS.

7.1.1.3.6 Select the samples to be included in the batch that are assigned the desired prep method. The samples can be individually selected by highlighting the sample and clicking the "$\rightarrow$" arrow or all samples can be selected by clicking the "$\Rightarrow$" arrow.

7.1.1.3.7 Samples that have be selected for re-analysis can be manually added to the sample list following the procedure outlined in 7.1.1.3.3.

7.1.1.3.8 "Save" the batch by clicking a previous batch number on the list and then returning to the newly created batch.

7.1.2 Table 7-1 indicates the number and type of samples that comprise an analytical batch. Note: NELAC requirements specify that the maximum number of client samples in an analytical batch can not exceed 20. Further, a batch can not be left "open" for a time period that exceeds 24 hours.

Table 7-1
Samples required in an Analytical Batch

Method Blank (MB)
LCS (and LCSD if no MSD)
Client Samples
MS and MSD (If supplied by client)
Reference Standard /CCV

7.2 Preparation and Extraction:

7.2.1 Remove the samples to be extracted from storage refrigerator so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.2.1.1 Check the sample backlog list (See section 7.1.1) for directions on the use of LIMS. At times the wrong procedure is logged into LIMS (e.g., 8260_VOC instead of 8011_VOC). If this is the case, contact the section supervisor.

7.2.1.2 The section supervisor will contact the Project Manager to determine the appropriate actions to be followed.

7.2.2 For samples and field blanks contained in 43-mL VOC vials, remove the container cap. Remove and discard approximately 8ml of sample using a transfer pipette or by carefully pouring. Replace the vial cap and weigh the vial and remaining sample to the nearest 0.1g. Record weight on the Log Sheet for each sample.

7.2.3 For calibration standards, check standards, QC reference samples, and blanks measure 35 ml of organic-free reagent water using a graduated cylinder and transfer to clean 43-mL VOC vials. Weighing of these vials is not necessary.

7.2.4 Using a micro syringe, add 25ul of Surrogate Working Solution (5.7) to each vial.

7.2.5 Using a micro syringe, spike the CCV sample vial(s) by adding 10ul of Working Calibration Mix (5.14).

7.2.6 Using a micro syringe, spike the QC Reference sample vial(s) by adding 25ul of Working Second Source Mix (5.11).

7.2.7 Using a micro syringe, spike the QC Check sample vial(s) (also used for LCS/LCSD) by adding 10ul of Working Second Source Mix (5.11).

7.2.8 Using micro syringe, spike the MS and MSD vials (if present) by adding 10ul of Working Second Source Mix (5.11).

7.2.9 Remove the container caps and add 7 g of NaCl to all samples, blanks, and standards.

7.2.10 Recap the sample container and dissolve the NaCl by shaking gently by hand for about 20 seconds.

7.2.11 Remove the cap and using a 2.5ml syringe, add 2.0 mL of hexane to all samples, blanks, and standards. Recap and shake vigorously by hand for 2 minutes. Allow the water and hexane phases to separate. If stored at this stage, keep the container upside down.

7.2.12 Label all 2-ml crimp top GC vials for each calibration standard, blank, and sample. Remove the sample container cap and carefully transfer a sufficient amount (0.5-1.0 mL) of the hexane layer into the labeled vial using a disposable glass pipette and cap tightly.

7.2.13 Transfer the remaining hexane phase, being careful not to include any of the water phase, into a second labeled vial. Store this second vial at 4 °C for reanalysis if necessary.

7.3 Gas chromatographic conditions

Set up operating conditions for the gas chromatograph using the following guidelines, Table 7-2. These conditions may be changed as necessary to improve or maintain analytical conditions.

Table 7-2
GC Conditions for EDB/DBCP Analysis

Condition	Setting	Condition	Setting
Sample Inlet	GC	Injection Source	GC ALS
Front Injector		Back Injector	
Sample Washes	1	No Parameters	
Sample Pumps	1		
Injection Volume	3.0 microliters	Temperature Profiles	
Syringe Size	10.0 microliters	Inlet A	200 C
On Column	Off	Inlet B	50 C
Nanoliter Adaptor	Off	Detector A	300 C
PostInj Solv. A Wash	0	Detector B	300 C
PostInj Solv. B Wash	6	Auxiliary	50 C
Viscosity Delay	1 Second		
Plunger Speed	Fast	Makeup gas	Argon/Methane 95:5%
Carrier Gas	Helium 25cm/sec	Makeup flow rate	30-60 ml/min

Condition	Setting	Condition	Setting
Oven Parameters		Oven Program	
Oven Equilib Time	0.5 minutes	Initial Temp	40 C
Oven Max	300 C	Initial Time	4.0 Minutes
Oven State	On	Level 1	8 C/min→190C, Hold 7.25 Min
Cryo State	Off	Next Run Time	30.00 Minutes
Cryo Blast	Off	Ambient	25 C

Inlet Purge	Initial Value	On Time	Off Time	Splitless Inject
A	Off	1.00	0.00	No
B	Off	1.00	0.00	Yes

Detector	Type	State
A	ECD	On
B	ECD	On

Signal	Source	Peak Width	Data Rate	Start	Stop
1	Det A	0.053	5.000	0.00	1.00
2	Det B	0.053	5.000	0.00	1.00

7.3.1 Preparation of 'Run Log' using Chemstation/Enviroquant software.

7.3.1.1 Open Enviroquant software by "clicking" on Icon.

7.3.1.2 Select "Sequence", then "Edit sample log table".

7.3.1.3 Select sample type for each sample.

7.3.1.4 Number all of the samples

7.3.1.5 Change data file using the following format

41030501

Where:

4 = GC number
Number 1= Year: '01'
0305 = Date in MM DD format
01 = sequential file number

7.3.1.6 Click "OK" when completed

7.3.1.7 On the pull-down menu, select "Sequence", then "Save".

7.3.1.8 When the following prompt appears, change the last part to MM DD format indicating the current day.

C:\HPCHEM\4\Data\410305 where

410305 = Instrument 4, month “03”, day “05”

7.3.1.9 When complete, click “OK”

7.3.1.10 On the pull-down menu, select “Sequence”, then “Run” to start the instrument run.

7.3.2 Editing Sequences

7.3.2.1 Click “File”, then “Edit”

7.3.2.2 Sample information can be directly entered into the pop up box that appears.

7.4 Standardization and Calibration

7.4.1 Retention Time Windows:

7.4.1.1 Before establishing windows, make sure the GC system is within normal operating conditions. Make three (3) injections of all single component mixtures throughout the course of a 72-hour period. Serial injections over a less than the 72-hour period may result in retention time windows that are too tight.

7.4.1.2 Calculate the standard deviation of the three absolute retention times of single component standard.

7.4.1.3 The retention time window is defined as plus or minus (±) three (3) times the standard deviation of the absolute retention times for each standard that is used to define the window. In all instances, however, the experience of the analyst weighs heavily in the interpretation of chromatograms.

7.4.1.4 In those cases where the standard deviation for a particular standard is zero, the analyst may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. The width of the window will be $\pm$0.03 minutes for the default standard deviation for narrow bore columns.

7.4.1.5 The laboratory must calculate retention time windows for each standard GC column and whenever a new GC column is installed.

7.4.1.6 The retention time window center is based on the retention times provided by the daily calibration verification standard. The retention time windows in the analytical method must be updated each working day at the beginning of an analytical sequence.

7.4.1.7 Simultaneous confirmation analysis is performed to validate the presence of a compound. The confirmation column has to be calibrated, and the retention times must be maintained on this column to eliminate the possibility of reporting false positive or false negative data.

7.4.2 Initial Calibration Procedure:

7.4.2.1 If the calibration standards have already been prepared and are in storage, remove them from the refrigerator and allow them to warm to room temperature. **All water based standards must be extracted within 8 hours of preparation.**

7.4.2.2 Place the vials in the GC autosampler tray beginning at position one. Analyze DBCM Retention Time standard to confirm retention time (see Section 3.5). It is recommended that the calibration standards be analyzed from the highest concentration to the lowest. This will ensure that any possible active sites in the instrument will not greatly affect the analysis of the calibration curve.

7.4.2.3 Record the sequence of standards in the GC Instrument Run log and create this sequence in the data acquisition system.

7.4.2.4 Analyze the initial calibration standards described in Table 5-1 by injecting 3 µL of each calibration standard. The lowest concentration level must be equal to the lower reporting limit (LIMS PQL) and the highest concentration must be equal to the LIMS UQL (Upper Quantitation Limit).

7.4.2.5 Using the data system, generate the initial calibration report indicating the calibration factors and the percent relative standard deviation for each analyte at each of the calibration levels analyzed.

7.4.2.6 Calculate the calibration factor for each analyte at each concentration as:

7.4.2.7 Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{\text{CF}} = \frac{\sum_{i-1}^{n} CF_i}{n}$$

Where n is the number of standards analyzed.

7.4.2.8 Calculate the standard deviation (SD) and the RSD of the Calibration factors for each analyte as:

$$RSD = \frac{SD}{\overline{CF}} x\,100$$

$$\mathrm{SD} = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}}$$

7.4.2.9 If the RSD (relative standard deviation) for each analyte is **≤10%,** the initial calibration is considered linear and the average calibration factor can be used to quantitate sample results.

7.4.2.10 In those instances where the RSD for one or more analytes exceeds 10%, a linear regression curve must be constructed with correlation coefficient of 0.995 or greater.

7.5 Calibration Verification and Analysis of Sample Extracts:

7.5.1 Prior to analysis of any standards or samples, several GC parameters should be checked and recorded in order to ensure that operating conditions have not changed significantly from those of prior analyses.

7.5.1.1 The auto-sampler syringe rinse solution should be filled with Hexane and waste bottles should be emptied.

7.5.1.2 Verify calibration by injecting calibration verification standards at 0.10ug/L (8011 QC Check Std.) and 0.25ug/L (8011 QC Reference Std.) prior to conducting any sample analyses. One CCV standard must also be injected at intervals of not less than once every ten (10) samples and at the end of the analysis.

7.5.1.3 All Reference standard, Check Standard and/or CCV recoveries must be within 60-140%. If preparation of new standards does not result in recoveries within 60-140%, new calibration curves must be prepared.

7.5.2 Sample Analysis

7.5.2.1 Remove the sample extracts from their storage location in the Semivolatiles Lab and allow them to warm to room temperature.

7.5.2.2 Place them in the GC autosampler with the appropriate number of calibration check solutions and hexane blanks. Analyzing the Method Blank immediately after the calibration check standard is preferable since this allows early evaluation of these QC samples and prevents any interference from possible carryover from samples previously analyzed.

7.5.2.3 An example sequence is shown below.

1. Hexane blank
2. CCV standard at 0.10 ug/L (8011 QC Check Std.)
3. Method blank
4. LCS (2^{nd} Source at 0.10 ug/L)
5. LCSD (2^{nd} Source at 0.10 ug/L)
6. Reference Std (2^{nd} Source at 0.25 ug/L)
7. 10 Sample extracts and batch QC samples
8. CCV (every 10 injections and at the end of the sequence)

7.5.2.4 Inject 3 μL of final sample extract to the GC system. Record the injection sequence in the GC instrument run logbook.

7.5.2.5 If any compound in any sample exceeds the analytical range of the highest calibration standard, that sample must be diluted.

7.5.2.6 Sample injection may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements.

7.5.3 Data interpretation

7.5.3.1 The qualitative identification of the single compounds determined by this method is based on retention time. Therefore, if any of the standards fall outside their daily retention time windows, the system is deemed out of control. The cause of the RT shifting must be corrected prior to continuing analysis and all affected samples reanalyzed.

7.5.3.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window on both analytical columns. If detection of the analyte is obviously prevented by background interference such as overlapping peaks, negative dips, etc., single column detection may be deemed positive identification after careful review using overlays with daily CCVs.

7.5.3.3 All peaks identified as target analytes in client samples must be carefully compared to standards for peak shape consistency using overlays. Peaks obviously not consistent with the standard peaks may be the result of background interference and/or non-target component interference. If the peaks do not reasonably match those of the standards, further review is required prior to reporting as a positive result. In most cases, these samples will require reporting with elevated RLs and job appropriately narrated.

7.5.3.4 Once a target analyte has been detected on both analytical columns and peak shapes have been confirmed as reasonably consistent with standard peaks, the analyst must evaluate the agreement between the quantitative results on each column.

7.5.3.5 The use of manual integration should be limited. Refer to AES Manual Integration SOP, QC-05014, for guidance on the use and documentation of manual integration.

7.5.3.6 If the relative percent difference (RPD) of the on column values exceeds 40%, check the chromatograms to see if an obviously overlapping peak or other chromatographic anomaly is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

7.5.3.7 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems and peak shapes, as demonstrated using overlays showing target peaks sufficiently consistent with standard peaks, report the higher result, qualify the result with “NC” (not confirmed) at QA review and narrate the affected workorder. This approach is conservative relative to the protection of the environment.

7.5.4 Quantitative analysis

7.5.4.1 Once qualitative analysis is complete and target analytes have been detected, the Chemstation software will use the calibration curve or RF to directly calculate the uncorrected concentration (C1) of the targets in the samples.

7.5.4.2 Calculate the actual sample volume (V1) for each sample as equal to the net sample weight:

7.5.4.2.1 Volume(V1) = initial wt. of vial and sample(g) – vial wt.(g)

7.5.4.3 Enter the V1 value as the SampAmt in the LIMS prep batch.

7.5.4.4 LIMS will calculate the corrected target concentrations from the raw values downloaded and the Prep Factors as:

7.5.4.4.1 Concentration, ug/L=Uncorrected Concentration x 35 / V1

Table 7-3

AES
8011 DATA REVIEW CHECKLIST

Batch ID:_____ Run No.:_____

INITIAL RAW DATA REVIEW
QA Analyst

☐☐: All raw data files and run log(s) been printed to pdf and posted to Portal Server (must include all chromatograms, overlays, screen shots, etc. where applicable) **PROPERLY SCALED CHROMATOGRAMS MUST BE PRESENT SO THAT BASELINES/PEAK INTEGRATIONS CAN BE VISUALLY REVIEWED FROM PORTAL FILES**
☐☐: All instrument standard IDs included on raw data or run log
☐☐: Current and approved cal curve used for quantitation
☐☐: All calibration verification criteria met for both columns **(No requires CAR if reported to LIMS)**
☐☐: All RTs for instrument and batch QC are within RT window **(No requires CAR if reported to LIMS)**
☐☐: All peaks properly integrated with baseline points properly assigned. **ALL TARGET ANALYTE PEAKS MUST BE INTEGRATED IN A MANNER CONSISTANT WITH THE INTEGRATIONS USED FOR THE ICAL STDS**
☐☐: All second column confirmation criteria met for all target analyte hits (≤40% RPD, within RT windows, etc.) **(No requires CAR and handling per SOP)**
☐☐: Background sufficiently low to allow for target identification at the PQL at the dilution run or data turned off for rerun at higher dilution
☐☐: Sample not over-diluted based on background or data turned off for rerun at lower dilution
☐☐: Check duplicate run data for all samples to ensure no compounds are double reported and dilution values match closely with original run.

GO TO LIMS "MAIN" RUN SCREEN
☐☐: All Sample IDs properly assigned per Backlog Report (double click on each SampleID to verify)
☐☐: All Test Codes properly assigned per Backlog Report
☐☐:All instrument QC run at required frequency
☐☐: All Sample Types properly assigned
☐☐: All samples linked to the Prep Batch properly with correct PFac, SpkFac and OFAC
☐☐: All dilution factors entered correctly per the raw data/run logs
☐☐: All Blkref, SPKref, RPDref and CCVref assigned correctly
☐☐: All Comments present are addressed **(May require CAR)**

GO TO "DATA" SCREEN
☐☐: Calculate Sequence to ensure LIMS calculations are complete
☐☐: Are all CCB/hexane blanks below PQL for all target analytes**(No requires CAR if reported to \ LIMS)**
☐☐: Are CCVs at 0.10 and 0.25ug/L(Ref) run at proper frequency and recovered at 60-140% **(No requires CAR)**
☐☐: Are there any B flags for target analytes indicating MB hits above PQLs **(Yes requires CAR)**
☐☐: Are there any S and/or R flags for spike cmpds and/or surrogates in LCS/LCSD **(Yes requires CAR)**
☐☐: Are there any S and/or R flags for spike cmpds and/or surrogates in MS/MSD **(Yes requires CAR)**
☐☐: Are there any H flags present for any samples **(Yes requires CAR)**
☐☐: Are there any E flags for target analytes on samples and/or Batch QC turned on for reporting **(Yes requires CAR)**

☐ ☐: Are there any J flags on any tartget analytes selected to report on diluted sample runs? **(Yes requires reanalysis at lower dilution or CAR to narrate elevated reporting limits)**

☐ ☐: Are there "*" qualifiers for J flagged target analytes? **(Yes requires removal of "*" qualifiers)**

☐ ☐: Are there any target analytes reported as BRL with elevated PQLs due to dilution or reduced prep volume **(Yes requires CAR)**

☐ ☐: Do all LIMS raw values match values on raw data

☐ ☐: Have at least 2 sample's final results been manually calculated to verify LIMS calculations

☐ ☐: Check duplicate run data for all samples to ensure no compounds are double reported and dilution values match closely with original run.

PRIOR TO FINAL QA APPROVAL

☐ **NA**: Have all CARs been closed and narratives written prior to QA.

DO NOT USE MATRIX INTERFERENCE NARRATIVES WITHOUT CHROMATOGRAPHIC EVIDENCE FOR MATRIX INTERFERENCE AND/OR PREP COMMENTS INDICATING MATRIX INTERFERENCE UNLESS CONFIRMED BY REEXTRACTION AND REANALYSIS.

☐ ☐:**ALL HITS FOR EDB AND/OR DBCP MUST BE THOROUGHLY REVIEWED BEFORE REPORTING. REVIEW MUST INCLUDE OVERLAY WITH STDS, COMPARING TO 8260 DATA IF SUFFICIENTLY HIGH, ETC.**

☐ **NA**: Have PM, Lab Manager and PM Director been notified for any CAR resulting in reextract and/or due date exceedance

CAR #: _____ _____ _____ _____ _____

Analyst Signature /Date/Time: _____

Reviewer Signature/Date/Time: _____

Comments: _____

_____

_____

_____

Table 7-4

Calibration Curve Review Checklist for GC Methods

Matrix: _____ Instrument: _____ Date Prepared: _____

Test Method: _____ Curve ID: _____

	Col 1	Col 2
• Does the calibration curve contain a minimum of 5 points.	☐	☐
• If average response factor used in calculation are all compound RSD ±20% for 8000 Methods and FL PRO or 10% for 600 Methods	☐	☐
• If linear regression used in calculation, is the correlation coefficient (r) > 0.995	☐	☐
• Is the lowest data point in each curve at the PQL in LIMS	☐	☐
List any analytes with data point below or above PQL		
• Is the highest data point in each curve at the UQL in LIMS	☐	☐
List any analytes with highest standard below UQL		
• Is the retention time RT of each target analyte in each calibration standard within the RT window.	☐	☐
Comments:		
• Does the ICV pass (% D < = 15% for each analyte or average of all %Ds ≤15% for 8000 Methods; ≤20% for FL PRO; % D ≤15% for each target for 600s)	☐	☐
• Is ICV a second source standard from ICAL standards	☐	☐
• Is the standard preparation information included and properly signed and dated.	☐	☐
• Is run log included and properly signed and dated.	☐	☐
• Are all chromatograms included and properly signed and dated.	☐	☐

Analyst: _____ Date: _____ Time: _____

Primary Reviewer: _____ Date: _____ Time: _____

Secondary Reviewer: _____ Date: _____ Time: _____

Comments:

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP #QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Calibration of the Gas Chromatograph. This is accomplished through a minimum 5-point calibration curve. The points on the curve must meet a 10 % RSD when comparing calibration factors or have linear regression correlation coefficient of 0.995 or greater to verify that the calibration curve is linear. The verification of the curve is also performed using the 8011 Check and Reference standards which must recover within 60-140% of the expected values.

8.1.3 Retention time window. The retention time for each analyte is compared over a 72-hour time period and the average retention time calculated. Daily retention times for all instrument QC samples must fall within specified time windows.

8.1.4 Method Detection Limit Study. The method detection limit is calculated by analyzing at minimum seven replicates prepared in blank water at 0.03 ug/L. Average recovery must be 60-140% and calculated MDL must be less than the spike concentration used. Calculated MDL must be <0.02 ug/L (PQL). MDL's are to be performed annually or whenever instrument conditions have changed that will affect the established detection limits.

8.1.5 Method blank. Reagent blank analysis must be performed at the following frequency: Every twenty (20) samples of similar concentration and/or sample matrix or whenever samples are extracted and analyzed by the same procedure at same time, whichever is more frequent. The concentration of the method blank of any analyte of interest should not be exceeding the laboratory established practical quantitation limit (PQL).

8.1.6 Surrogate Recovery. All samples, blanks and QC samples are fortified with surrogate spiking compound before extraction and injection in order to monitor sample extraction efficiency. The recovery of the surrogate compound must be within the recovery limits established by the laboratory.

8.1.7 Laboratory Control Sample (LCS). The Laboratory Control Sample (LCS) is used to monitor, assess and document laboratory method performance and is performed with every batch or twenty samples, whichever occurs more frequently. The recovery of the analytes must meet established laboratory guidelines.

8.1.8 Laboratory Control Duplicate Sample (LCSD). The Laboratory Control Duplicate Sample (LCSD) is only analyzed when there is insufficient sample volume to perform the MSD. The LCSD is used to monitor, assess and document laboratory method performance and is performed with every batch or twenty samples, whichever occurs more frequently. The recovery of the analytes must meet established laboratory guidelines.

8.1.9 Sample spike and duplicate spike. Matrix spikes and matrix spike duplicates are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples. The recovery of the analytes must meet established laboratory guidelines.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP #QA-01000, "Quality Assurance Manual," of the in-house method manual in Section 5.8, "Procedures For Assessing Out-Of-Control Situations," and in Table 5-6, "Summary of Calibration and Quality Control Procedures for Various Test Types". These tables include corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance and any procedure deviations in appropriate logbooks.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 Health and Safety: Safety glasses and latex type gloves must be worn at all times when dealing with any chemicals, samples, or reagents. A lab coat is also highly recommended. Close-toed shoes and clothing that covers the legs (no shorts or dresses) must be worn at all times an analyst is working in the laboratory.

9.2 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a health hazard and exposure should be as low as reasonably possible. All health and safety concerns for these and any other chemicals are listed in the Material Safety Data Sheets (MSDS) provided by the supplier or manufacturer of these chemicals. A copy of any MSDS is available for review at any time.

9.3 Charcoal traps should be installed on the GC split vents of the instruments with split/splitless injectors to prevent airborne contamination of the work area. All vacuum pumps (Edwards) should contain vapor traps on the outlet pump manifold

9.4 The analyst shall observe all safety precautions provided in the laboratory safety program.

9.5 The ECD detectors on the gas chromatographs must be vented to prevent airborne contamination.

10.0 DATA REPORTING

10.1 The LIMS system automatically rounds the data based upon factors that are set up for each test category.

10.2 Calculation of results using Enviroquant software.

10.2.1 Open Enviroquant software by "clicking" on Icon

10.2.2 On the pull-down menu, click on "Data Analysis"

10.2.3 On the pull-down menu, click on "Load", then "Data file". The path C:\hpchem\2\data should appear.

10.2.4 Using the "arrow" key, scroll down to the desired sequence number. The sequence number represents the GC number, year, month and day in a format 410321. Note that the left side of the box contains all of the samples in the run. Each sample can be selected by positioning the mouse pointer over the sample and double clicking it.

10.2.5 Once a sample has been selected, On the pull-down menu, click on "Load", then "Method". Select the method 8011MA15.M where MA = the month, March and 15 = the date. From this point, the method will remain the same for each sample selection.

10.2.6 To calculate a result, on the pull-down menu, click on "Quant", then "Calculate and generate report". An alternative method is to click on "Quant", then "Int" and "Integrate".

10.2.7 Review the Enviroquant calculated result by clicking on "Quant", then "Q edit". To enlarge the various areas of the chromatogram, place the pointer on the chromatogram and right click the mouse. Drag the mouse over the area to enlarge. Double click the chromatogram to return it to its original size.

10.2.8 Once the chromatogram has been enlarged, the baselines can be redrawn by placing the mouse at one end of the peak and moving it across the bottom while holding down the right mouse. Release the mouse when the line is the correct length.

10.2.9 A general rule for drawing the baseline is that it should be started from the lowest side of the peak so that the baseline makes a "right angle" at the raised side of the peak.

10.2.10 Perform the same procedure on the rest of the chromatograms.

10.3 Completed data is stored in the "C" drive of the acquisition computer under the following directory: "C:\HPCHEM\410305" where 4 is the instrument number, 1 is the year "01", 0305 is the date in MM DD format.

10.3.1 Prior to moving files for final storage, open the completed data folder and change the status of the data to "frozen" using the following procedure.

10.3.1.1 In Enviroquant, click "Tools", then "Change Data State"

10.3.1.2 Click the radio button next to "Frozen"

10.3.1.3 Click "OK". Exit to save the changes

10.3.1.4 Using NT Explorer, Find the file on the "C' drive in the directory "C:\HPChem\1 where 1 = GC 3 and 2 = GC 4

10.3.1.5 Highlight the file and Click "Cut"

10.3.1.6 Using NT Explorer, find and select the folder in the computer called "Storage".

10.3.1.7 Locate the proper directory: GC [#] and click "Save" to save the file to this directory.

10.3.1.8 Periodically, these files are written to a writeable "CD" and stored off site by the VP of Technical Operations.

10.4 Current MDLs for all parameters may be found in Table 5, "Quality Assurance Objectives for Surface Waters, Ground Waters and Waste Waters," and Table 5, "Quality Assurance Objectives for Sediments, Soils and Sludges," found in SOP QA-01000, "Quality Assurance Manual," of the in-house method manual.

10.5 Table 10-1 below summarizes the approximate retention times of the two compounds measured in this test procedure for each column.

Table 10-1

APPROXIMATE RETENTION TIMES FOR DCBP and EDB

Compound	Retention Time (min) DX-3	Retention Time (min) DB-1
DCBP	16.77	12.77
EDB	9.98	6.88
DBCM	10.60	16.77

11.0 FILE MAINTENANCE

11.1 All data are electronically printed as pdf files and stored on the Portal Server.

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto portable hard drives. Two copies are made. One copy is stored on the laboratory premises, the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks must be completed each time that any maintenance is performed upon the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as replacement of motors in tower autosamplers.

12.2 Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.3 Routine maintenance: Typical routine maintenance consists of keeping the system clean and insuring that chromatography remains acceptable. Examples would be peak tailing and degradation of DDT and Dieldrin in pesticide analysis.

12.4 The table below indicates the frequency of routine maintenance for various instruments types within the laboratory.

Table 12-1
Maintenance Frequency

Maintenance Action	Recommended Frequency
Changing injection port liners	Weekly or when chromatography Is effected
Trimming column	Monthly or when chromatography Is effected
Changing GC/HPLC Column	Annually or when other attempts to resolve chromatography fail

12.5 Other types of exceptional maintenance are discussed below.

12.5.1 ECD Background Signal Monitoring

12.5.1.1 Electron Capture Detectors must be regularly monitored for signs of degradation. Monitoring is performed by checking and recording each detector signal from the from GC panel at ambient temperature (40C or less). **BACKGROUND READINGS MUST BE RECORDED IN EACH INSTRUMENT LOG FOR EACH DETECTOR EVERY TIME ANY OTHER MAINTENANCE IS PERFORMED**.

12.5.1.2 The table below from the Agilent Technologies Electron Capture Detector Troubleshooting Tips guidance document describes how signal measurements must be interpreted and appropriate corrective actions.

Table 12-2

5890	6890 ECD	6890 u-ECD	
<10	<20	<200	ECD is likely in a good state of health.
10-40	20-80	200-400	Slightly elevated, no cause for concern at this point. Signal still in "good" range.
40-80	80-150	400-1000	System showing signs of contamination from gases, column, or samples. If the signal increases in response to increased oven temperature, suspect the column.
80-200	150-300	1000-2000	Suspect more severe contamination, follow the troubleshooting guidelines.
>200	>300	>2000	If the following procedures do not work, suspect the ECD Cell.

12.5.1.3 Troubleshooting procedure for ECDs showing more severe contamination.

12.5.1.3.1 Verify that gas supplies are clean. Carrier and makeup gas are recommended to be >99.9995% purity. Even ultra high purity gases must have traps. Check traps for plastic composition or O-ring seals as these can cause contamination.

12.5.1.3.2 Verify that the inlet is clean and inactive and that the column is performing as adequately as indicated by acceptable peak shapes.

12.5.1.3.3 If problem persists after gases, inlet and columns are ruled out as suspect, bake the ECDs as follows:

12.5.1.3.3.1 Remove the column from the the detector and cap the base of the makeup gas adapter. Verify that the makeup gas flow is present. If the signal decreases significantly at this point, suspect contamination prior to the ECD detector. Correct this problem before continuing.

12.5.1.3.3.2 If the signal changes significantly by simply turning the oven fan on and off, suspect a crack or leak in the makeup gas line. **STOP HERE** and evaluate replacing the makeup adapter. Baking the detector with a leak can cause damage to the ECD Cell.

12.5.1.3.3.3 Bake the detector at 350C for 1 hour, taking note of the initial and final signal levels during this period. If the signal does not decrease during the bake time and all leaks in the makeup gas system have been ruled out, bake overnight at 350C. If signal still does not decrease, ECD cells must be replaced or cleaned by removing and sending to outside vendor.

12.6 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as autosampler that will not function. If this type of problem occurs, contact the department manager for assistance.

13.0 METHOD PERFORMANCE

13.1 See referenced method for specific method performance data.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 SOP HS-03005 Waste Disposal and SOP SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade –A dry chemical that has been dried at 250°C for 4 hours cooled and stored in a desiccator.

15.2 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to distilled water or clean soil and the concentration is measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.3 DI water- Deionized water

15.4 RSD – Relative Standard Deviation

15.5 RF – Response factor. Determined as the concentration of a sample divided by the chromatographic area of the peak produced by the sample.

15.6 MS- Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.7 MSD- Matrix Spike Duplicate.

15.8 CCV - Continuing calibration verification standard. Must be varied thoughout the daily runs, that is the concentration must be low, middle, and sometimes at the upper end of the calibration curve.

15.9 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot that the calibration standard.

15.10 LCSD - Laboratory Control Sample Duplicate

16.0 REFERENCES

16.1 "Determinative Chromatographic Separations", USEPA SW-846 Method 8000B, Revision 2, December 1996.

16.2 "1,2-Dibromomethane and 1,2-Dibromo-3-chloropropane by Micro-extraction", USEPA SW-846 Method 8011, Revision 0, December 1992.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://portal/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://portal/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

18.0 SOP REVISION HISTORY

Revision Date	Revision #	Summary of and Reason for Changes/Updates	Responsible for Revision
6/3/2003	5	Update	Greg Jones
9/19/2007	6	Update	Greg Jones
8/12/2008	7	Update	Greg Jones
9/16/2010	8	Update	Dana Till
2/8/2012	9	SC Audit: Updates to Sections 5.0, 7.4.2.2, 7.5.3.5, Tables 5-2, 7-2, 10-1, and addition of Section 18.0.	Dana Till

AES, Inc.
3785 Presidential Pkwy
Atlanta, GA 30340

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DOCUMENT CONTROL NUMBER: Rev. 5

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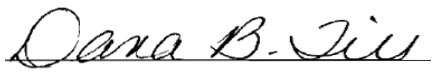
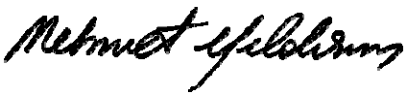
Technical Director:  Date: 2/6/2012

Laboratory Manager:  Date: 2/6/2012

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STANDARD OPERATING PROCEDURE FOR
DIESEL RANGE ORGANICS BY EPA SW-846 METHOD 8015C

TABLE OF CONTENTS

Page

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to nearly all types of samples, regardless of water content, including ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

1.2 This method is applicable to the analysis of petroleum hydrocarbons, including diesel range organics (DROs). DROs correspond to the range of alkanes from C_{l0} to C_{28} and covering a boiling point range of approximately 170^0C - 430^0C. The identification of specific fuel types may be complicated by environmental processes such as evaporation, biodegradation, or when more than one fuel type is present. Methods from other sources may be more appropriate for DROS, since these hydrocarbons are not regulated under RCRA. Consult State and local regulatory authorities for specific requirements.

1.3 This method is restricted for use by, or under the supervision of, analysts experienced in the use of gas chromatographs and skilled in the interpretation of gas chromatograms. In addition, if this method is used for the analysis of petroleum hydrocarbons, it is limited to analysts experienced in the interpretation of hydrocarbon data. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.4 The method can also be used as a screening tool (for semivolatile organics) to obtain semiquantitative data for the prevention of sample overload during quantitative analysis on a GC/MS system. This may be accomplished using an automated (Method 5021) headspace method or by direct injection if a solvent extraction method has been utilized for sample preparation. Single point calibration would be acceptable in this situation. Performance data are not provided for screening.

1.5 This method incorporates the preparative procedures for the extraction of waste, water, and solid samples by SW-846 Methods 3580A (Waste Dilution), 3510C (Separatory Funnel Extraction), 3520C (Continuous Liquid-Liquid Extraction), 3550C (Ultrasonic Extraction), and 3540C (Soxhlet Extraction).

2.0 SUMMARY OF METHOD

2.1 Method 8015C provides gas chromatographic conditions for the detection of certain nonhalogenated semivolatile organic compounds.

2.2 Ground or surface water samples must generally be analyzed in conjunction with methods 3510C, 3520C, or other appropriate preparatory methods to obtain the necessary quantitation limits.

2.3 Diesel range organics (DROS) may be prepared by an appropriate solvent extraction method.

2.4 An appropriate column and temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by a flame ionization detector (FID).

2.5 The method allows the use of packed or capillary columns for the analysis and confirmation of the non-halogenated individual analytes. Columns and conditions listed have been demonstrated to provide separation of those target analytes. Analysts may change these conditions as long as they demonstrate adequate performance.

2.6 Fused silica capillary columns are necessary for the analysis of petroleum hydrocarbons.

3.0 INTERFERENCES

3.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a solvent blank to check for cross contamination.

3.2 Clean syringes or autosamplers by flushing all surfaces that contact samples using appropriate solvents.

3.3 All glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry in an oven at 130^0C for several hours or rinse with methanol and drain. Store dry glassware in a clean environment.

3.4 The flame ionization detector (FID) is a non-selective detector. There is a potential for many non-target compounds present in samples to interfere with this analysis.

3.5 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to bake the injection device prior to subsequent analysis.

3.6 The laboratory where volatile analysis is performed should be completely free of solvents.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

4.1 Sample collection and preservation

4.1.1 Aqueous samples to be analyzed for Method 8015C DRO must be collected in a 1-liter amber jar. It is suggested that two sample jars be filled for each sample to ensure that the laboratory has sufficient sample for re-analysis if the need

arise.

4.1.2 Soil samples to be analyzed for Method 8015C DRO must be collected in 4 oz amber jars.

4.2 Sample holding times and storage

4.2.1 Samples to be analyzed for DRO may be held for 7 days prior to extraction if they are aqueous and 14 days prior to extraction if they are soil.

4.2.2 Extracts may be held at 4°C for up to 40 days prior to analysis by GC.

5.0 REAGENTS AND STANDARDS

5.1 Reagent grade chemicals shall be used whenever possible. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water.

5.3 Methanol, CH_3OH, pesticide quality or equivalent. Store away from other solvents.

5.4 Methylene Chloride, pesticide quality or equivalent. Store away from other solvents.

5.5 Sodium Sulfate, granular anhydrous. Purify by heating to 400^{o}C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

5.6 First Source Diesel Fuel Standard. Purchase from Accustandard, 20,000mg/L. Follow manufacturer's expiration date prior to opening ampule.

5.7 Second Source Diesel Fuel #2 Standard. Purchase from Restek, 50,000 mg/L. Follow manufacturer's expiration date prior to opening ampule.

5.8 Retention Time Window Standard. Florida Pro Spiking Solution Std or equivalent containing C10 and C28. Follow manufacturer's expiration date prior to opening ampule.

5.9 Surrogate standard. Dioctylphthalate, purchased from Aldrich Chemical. Follow manufacturer's expiration date.

5.10 Stock surrogate standard. Dilute 0.5mL of the commercially purchased surrogate standard to 50mL in a volumetric flask with methylene chloride. The concentration of this standard will be 10,000mg/L. Surrogate stock standard is good for 6 months.

5.11 Surrogate spike standard. Dilute 2mL of the stock surrogate standard to 200mL in a volumetric flask with acetone. The concentration of this standard will be 100mg/L. Surrogate spike standard is good for up to 6 months. The expiration date of the surrogate spike standard cannot exceed the expiration date of the surrogate stock standard (5.9).

5.12 Calibration Verification Standards.

5.12.1 Continuing Calibration Verification standards (CCVs) are prepared from the first source diesel fuel standard. The concentration of the standard should be varied throughout the curve, i.e., analyze a low concentration, mid-curve concentration, and high concentration standard.

5.11.2 Initial Calibration Verification (ICV) standard is prepared from the second source DRO standard and is analyzed at a concentration in the mid-range of the curve. To prepare this standard, dilute 40µL of second source diesel stock (50,000 mg/L) and 15µL surrogate (Dioctylpthalate) stock (10,000mg/L) to 1-mL final volume in methylene chloride. The final concentration for DRO is 1000mg/L and the surrogate is 100mg/L. ICV standard is good for up to 6 months. The expiration date of the ICV standard cannot exceed the expiration date of the stock standard solutions.

5.13 Calibration curve. Prepare a calibration curve as indicated in Table 5-1 below.

Table 5-1
DRO Calibration Curve

Diesel Stock (5.5) Used (µL)	Surrogate Stock (5.8) Used (µL)	Final Volume (mL)	Final Surrogate Concentration (mg/L)	Final Diesel Concentration (mg/L)
200ul of 1000std	None	1.0	20	200
500ul of 1000std	None	1.0	50	500
100	10	1.0	100	1000
200	15	1.0	150	2000
500	20	1.0	200	5000
500 (insert)	0	0.5(insert)	0	10000

5.14 Laboratory Control Spike (LCS) and Matrix Spiking (MS) Solution. Purchase Second Source Diesel Fuel #2 Standard from Restek (see Section 5.7). Spiking Solution is a 1,000 mg/L standard made from the 50,000 mg/L stock standard by adding 1 mL of the stock into a 50 mL flask and bring to volume with Acetone. This LCS and MS Spiking Solution has a final concentration of 1,000 mg/L. Spiking solution standard is good for up to 6 months. The expiration date of the spiking solution cannot exceed the expiration date of the stock standard.

5.15 Vendor information. The reagents and chemicals used in this test procedure are purchased from the vendors indicated in Table 5-2

Table 5-2
DRO Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
Methylene Chloride	VWR	Neat	BJ300-4
Sulfuric Acid	EM Science	Neat	SX1244-5
Methanol	JT Baker	Pure	JT9263-3
Diesel Fuel (1st source)	Accustandard	20000 mg/L	FU-009-40X
Diesel Fuel (2nd source)	Restek	50000 mg/L	31258
Florida Pro Spiking Std	Ultra	500 µg/ml each cmpd (17)	SFL-601
Dioctylphthalate	Aldrich Chemical	Neat	D20,115-4
Sodium Sulfate	VWR	Pure	3375-09

6.0 APPARATUS AND MATERIALS

6.1 Gas Chromatograph: Hewlett Packard Model 5890 system complete with an auto-sampler, FID, HP 3365 Series II computer complete with Chemstation version A.03.01.

6.2 Capillary Column: Equity-5, 30M X 0.25 mm or equivalent.

6.3 Ultrasonic preparation - A horn-type device equipped with a titanium tip, or a device that will give equivalent performance, shall be used.

6.3.1 Ultrasonic Disrupter - The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. A device designed to reduce the cavitation sound is recommended. Follow the manufacturers instructions for preparing the disrupter for extraction of samples with low and medium/high concentration.

6.3.2 Use a 3/4" horn for the low concentration method.

6.4 SPE Apparatus:

6.4.1 SPE Extractor (SPE-DEX 4790)

6.4.2 SPE Controller (SPE-DEX Controller for 4790)

6.5 Vacuum Pump

6.6 Dry disk Apparatus (Drydisk Separation Membrane), to dry samples

6.7 Separatory funnel – 2000 ml with teflon stopcock.

6.8 Syringes

6.9 Volumetric flasks, Class A - Appropriate sizes with ground glass stoppers.

6.10 Analytical balance - 0 - 160 g capacity, capable of measuring differences of 0.0001 g.

6.11 Microsyringes - 10- and 25-pL with a 0.006 in. ID needle (Hamilton 702N or equivalent) and 100-µL.

6.12 A 5-ml Luer-Lok glass hypodermic and a 5-ml gas-tight syringe with shutoff valve for volatile analytes.

6.13 Zymark Turbo-Vap II concentrator (for sample concentration)

6.13.1 200 ml concentration vessels with 1.0 ml sensor endpoint

6.14 Concentration tubes – Kuderna Danish (KD), 10 ml graduated.

6.15 Snyder column, Kuderna Danish, three ball macro.

6.16 Beakers, 400 ml.

6.17 2-ml GC crimp top vial with Teflon-lined cap.

6.18 Silicon carbide boiling chips – solvent rinse the chips prior to use.

6.19 Water bath – capable of temperature control (+/- 2°C). The bath should be used in a hood.

6.20 Vials: 5 and 20 ml with screw caps and Teflon-lined septa.

7.0 PROCEDURE

7.1 Preparation of extraction log form and extraction log in LIMS.

7.1.1 Each day the section supervisor prepares a work log. The log lists samples that are included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN EXTRACTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received in a "rush" status will have a chain of custody delivered to the extraction supervisor by the project manager.

7.1.1.2 Prepare a written extraction log using the DRO logbook that is kept in the extraction supervisor's office. The following entries must be made in the log.

7.1.1.2.1 Date and time that the batch is opened or the date and time the liquid/liquid extraction system or Soxhlet extraction system is started.

7.1.1.2.2 All samples included in the extraction batch.

7.1.1.2.3 Volume or weight of samples extracted.

7.1.1.2.4 Date and time that the liquid/liquid extraction or Soxhlet extraction is completed.

7.1.1.2.5 Extraction procedure employed.

7.1.1.2.6 The initials of the extraction analysts.

7.1.1.2.7 Laboratory number of all reagents used including surrogate standard, spiking standard, methylene chloride, and sodium sulfate used.

7.1.1.2.8 Volume of all reagents used including surrogate standard, spiking standard, and methylene chloride.

7.1.1.2.9 Final volume of all concentrates.

7.1.1.2.10 Date and time the batch is closed.

7.1.1.2.11 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.1.1.3.1 Open Prep Batch in LIMS by double clicking the "Prep" box.

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS.

7.1.1.3.3 Select the Prep Code "3510" from the pull down list. The LIMS will automatically assign an MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCSD and enter the information.

7.1.1.3.4 Enter the technician initials from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this preparation method. Note that the list contains all samples requiring extraction by method 3510 not just samples that have been logged into LIMS for DRO analysis.

7.1.1.3.6 Select the samples to be included in the batch that are assigned the desired prep method. The samples can be individually selected by
highlighting the sample and clicking the "→" arrow or all samples can be selected by clicking the "⇒" arrow.

7.1.1.3.7 Samples that have be selected for re-analysis can be manually added to the sample list following the procedure outlined in section 7.1.1.3.3.

7.1.1.3.8 "Save" the batch by clicking a previous batch number on the list and then returning to the newly created batch.

7.1.2 Table 7-1 indicates the number and type of samples that comprise an analytical batch. Note: NELAC requirements specify that the maximum number of client samples in a preparation batch can not exceed 20. Further, a batch can not be left "open" for a time period that exceeds 24 hours.

Table 7-1
Samples in a NELAC Batch

Method Blank (MB)
LCS and LCSD
Client Samples
MS and MSD (If supplied by client)

7.2 Extraction and hydrolysis of high concentration waste samples.

7.2.1 Remove the samples to be extracted from designated storage location so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.2.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS. At times the wrong extraction procedure is logged into LIMS (e.g., Continuous Liquid/Liquid Extraction instead of Separatory Funnel Extraction). If this is the case, contact the section supervisor.

7.2.1.2 The section supervisor will contact the Project Manager to determine the appropriate actions to be followed.

7.2.2 Prepare the Borosilicate culture tubes by rinsing them two times with methylene chloride.

7.2.3 Transfer approximately 1 g of waste sample to a 20-ml vial (record weight to the nearest 0.1 g). Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

7.2.4 Add 2.5-ml surrogate spiking solution to all samples and blanks. For the sample in each analytical batch selected for spiking, add 2.5 ml of the matrix-spiking standard.

7.2.5 Immediately dilute to 10 ml with methylene chloride. If the sample does not dissolves in methylene chloride, try acetone, methanol or hexane.

7.2.6 Add 2 g of anhydrous sodium sulfate to the sample.

7.2.7 Cap and shake the sample for 2 min.

7.2.8 Allow the extract to settle in the 10-ml vial with screw top.

7.2.9 Place a portion of the sample into a GC autosampler vial and seal with a crimp top. Label the vial appropriately.

7.3 Extraction of aqueous samples using separatory funnels.

7.3.1 Remove the samples to be extracted from designated storage location so they can warm to room temperature. If the samples cannot be found in the

refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.3.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS. At times the wrong extraction procedure is logged into LIMS (e.g., Continuous Liquid/Liquid Extraction instead of Separatory Funnel Extraction). If this is the case, contact the section supervisor.

7.3.1.2 The section supervisor will contact the Project Manager to determine the appropriate actions to be followed.

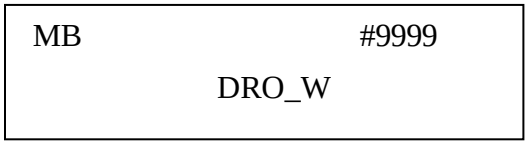
7.3.2 Prepare a 2-liter separatory funnel and a 250mL Zymark concentration tube with filter funnel for each sample in the analytical batch by rinsing two times with 30 ml of methylene chloride. Discard the solvent media after each rinse.

7.3.3 Place a folded filter paper in each funnel and add approximately 10-20 grams of sodium sulfate to each funnel.

7.3.4 Table 7-1 indicates the number and type of samples that comprise a prep batch. Note: NELAC requirements specify that the maximum number of client samples in a prep batch can not exceed 20. Further, a batch can not be left "open" for a period that exceeds 24 hours.

7.3.5 Label each zymark tube using the white label tape and a "Sharpie". White tape is used for DRO analysis. The information included in the label is the sample name, batch number, test type, and sample matrix as indicated in the example below.

Example Label used in Extraction Laboratory



7.3.6 Mark the sample volume on the sample bottle and transfer the sample to a 2-liter separatory funnel. After rinsing the bottle with methylene chloride, fill with water and measure the volume using a graduated cylinder.

7.3.7 Pre-clean a 1-ml syringe by rinsing at least 5 times with methylene chloride.

7.3.8 Using the syringe, add 1.0 ml of the Spike solution to the LCS, LCSD, MS, and MSD samples. Rinse the syringe with methylene chloride as before.

7.3.9 Add 1.0 ml of the dioctylphthalate surrogate spiking solutions to all of the samples.

7.3.10 Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, adjust the pH to < 2 with 1:1 sulfuric acid for extraction.

7.3.11 Add 60 ml of methylene chloride to the sample bottle and rinse both the bottle and the graduated cylinder. Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure.

7.3.12 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, and centrifugation.

7.3.13 Collect the methylene chloride extract in the zymark tubes by pouring the extract through a filter funnel containing dried sodium sulfate crystals.

7.3.14 Extract two more times with additional 60-ml portions of methylene chloride. Collect the extract as before. **After the third extraction, allow the layers to separate for at least 15 minutes to insure complete solvent-aqueous separation**. Remove any additional solvent layer.

7.4 Extraction of aqueous samples by SPE. See Table 7-4

7.5 Extraction of soil samples using horn sonicator.

Extractions are performed with the appropriate solvent, the extraction is performed in the specified pulse mode, and the horn tip is positioned just below the surface of the solvent yet above the sample.

Very active mixing of the sample and the solvent must occur when the ultrasonic pulse is activated. The analyst must observe such mixing at some point during the extraction process.

7.5.1 Remove the samples to be extracted from designated storage location so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.5.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS. At times the wrong extraction procedure is logged into LIMS (e.g., Continuous Liquid/Liquid Extraction instead of Separatory Funnel Extraction). If this is the case, contact the section supervisor.

7.5.1.2 The section supervisor will contact the Project Manager to determine the appropriate actions to be followed.

7.5.2 Prepare a 400-ml jar and 250mL zymark concentration tube with filter funnel for each sample in the analytical batch by rinsing two times with 30 ml of methylene chloride. Discard the solvent media after each rinse. Assemble the apparatus.

7.5.3 Place a folded filter paper in each funnel and add approximately 10-20 grams of acidified sodium sulfate to each funnel.

7.5.4 Table 7-1 indicates the number and type of samples that comprise a prep batch. Note: NELAC requirements specify that the maximum number of client samples in a prep batch can not exceed 20. Further, a batch can not be left "open" for a period that exceeds 24 hours

7.5.5 Label jars and zymark tubes using the white label tape and a "Sharpie". White tape is used for DRO analysis. The information included in the label is the sample name, batch number, test type, and sample matrix as indicated in the example below.

Example Label used in Extraction Laboratory

MB #9999
DRO_S

7.5.6 Remove approximately one-half of the contents of the soil jar. In a clean container, mix the soil in an attempt to make it as homogenous as possible.

7.5.7 Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture **must be mixed** with 60 g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate may be added. After addition of sodium sulfate, the sample should be free flowing

7.5.8 Accurately weigh approximately 30 grams of soil sample and place into the appropriate jar. The MB, LCS, and LCSD samples should be prepared from 30 grams of clean sand. For the sample chosen for MS/MSD, the initial weights for MS and MSD must be within 5% of each other.

7.5.9 Pre-clean a 1-ml syringe by rinsing at least 5 times with methylene chloride.

7.5.10 Using the syringe, add 1.0 ml of the Spike solution to the LCS, LCSD, MS, and MSD samples. Rinse the syringe with methylene chloride as before.

7.5.11 Add 1.0 ml of the dioctylphthalate surrogate spiking solutions to all of the

samples.

7.5.12 Immediately add 100 ml of methylene chloride to each jar.

7.5.13 Place the bottom surface of the tip of the #207 (or equivalent) 3/4 inch disrupter horn about 1/2 inch below the surface of the solvent, but above the sediment layer.

7.5.14 Extract ultrasonically for 3 minutes, with output control knob set at 10 (or sufficiently high enough to produce mixing of the sample) and with mode switch on Pulse (pulsing energy rather than continuous energy). Percent-duty cycle knob must be set at 50% (energy on 50% of time and off 50% of time). **Do not use microtip probe. Do not sonicate with a power setting that is less than 7**.

7.5.15 Decant the extract and filter it through Whatman No. 41 filter paper (or equivalent) in a filter funnel that is attached to a clean 250mL zymark concentration tube.

7.5.16 Repeat the extraction two more times with an additional 100ml portion of solvent. Decant off the solvent after each ultrasonic extraction. On the final ultrasonic extraction, pour the entire sample into the funnel, rinse with extraction solvent, and collect the solvent extract in the zymark tubes. Continue filtration until all visible solvent is removed from the funnel.

7.5.17 Quantitatively transfer the extract to the Erlenmeyer flask. Proceed to Section 7.7 for extract concentration.

7.6 Extraction concentration.

7.6.1 Turbo-Vap Sample Concentration

7.6.1.1 Set the Turbo-Vap concentrator bath temperature to 39°C. Set the Turbo-Vap pressure to 20. Check the water level in the bath and ensure that it is half way up the lower set of perforations in the back of the chamber.

7.6.1.2 Program the Turbo-Vap to stop indicate concentration completion by an audible sound. Note that the sample could go dry because the system continues to operate after the system emits the sound.

7.6.1.3 Place the vessels in the Turbo-Vap and close the lid. Press the start button for the appropriate vessel. Be sure the solvent waste is draining properly into the waste bottle. When the sample is complete, the sensor will sound intermittently until the vessel is removed.

7.6.1.4 Remove the vessel from the Turbo-Vap"' to prevent further evaporation. The outside of the condenser may be covered in water droplets. Be sure that they do not drip into the sample extract. If the sample evaporates to dryness, re-extract the entire sample.

7.6.1.5 Cool the sample, dilute to exactly 1.0 ml with methylene chloride, and transfer to a screw top vial for storage.

7.6.1.6 If the sample will not concentrate to 10 ml or less, is very viscous, or is very dark in color, make comments in the logbook and dilute to a higher volume.

7.7 Gas chromatographic conditions

Set up operating conditions for the gas chromatograph using the following guidelines. These conditions may be changed as necessary to improve or maintain analytical conditions.

These chromatographic conditions should be sufficient to achieve the detection limits in Section 5 of the QA Manual.

7.7.1 Preparation of Gas Chromatographic conditions using Chemstation/Chemstation software and “Methods” function. Typically, the gas chromatographic conditions have been pre-set so as to conform to the method, however, due to unforeseen circumstances, it may be necessary to change these conditions. This task can be performed by following the subsequent instructions.

7.7.1.1 Open Chemstation software.

7.7.1.2 In the pull-down menu, click “Method”. Select the desired method.

7.7.1.3 Click “Edit Entire Method”, then “OK”. The software will prompt the user through the method for edit.

7.7.1.4 Current GC conditions for this method (DRO1.M) are as follows

Table 7-2
Current GC Conditions for DRO Analysis

Condition	Setting	Condition	Setting
Sample Inlet	GC	Injection Source	GC ALS
Front Injector		Back Injector	
Sample Washes	3	No Parameters	
Sample Pumps	6		
Injection Volume	1.0 microliters	Temperature Profiles	
Syringe Size	10.0 microliters	Inlet A	275°C
On Column	Off	Inlet B	
Nanoliter Adaptor	Off	Detector A	325°C
PostInj Solv. A Wash	3	Detector B	Off
PostInj Solv. B Wash	Off	Plunger Speed	Fast
Viscosity Delay	1 Second	Carrier Gas	Helium 20.2 psi, 0 hold, then 99 psi/min to 40 psi, hold 0.0 min

Condition	Setting	Condition	Setting
Oven Parameters		Oven Program	
Oven Equilib Time	0.5 minute	Initial Temp	50°C
Oven Max	325°C	Initial Time	1.0 minute
Oven State	On	Level 1	25°C /min→300°C, Hold 1.00 min
Cryo State	Off		
Ambient	25°C	Next Run Time	16.0 minutes

Inlet Purge	Initial Value	On Time	Off Time	Splitless Inject
A	Off	1.00	0.00	No
B	Off	Off	0.00	

Detector	Type	State
A	FID	On
B	FID	Off

Signal	Source	Peak Width	Data Rate	Start	Stop
1	Det A	0.01	20Hz	2.00	16.0
2	Det B			0.00	

7.7.1.5 Set the output destination to screen, printer, and file.

7.7.1.6 Set the method to “autointergrate”, and not to generate a report during the run (wastes paper).

7.7.1.7 Set reference window to 10%, non-reference window to 5%, correlation window to 0.02 minutes, default multiplier to 1.00, and default sample concentration to 0.00.

7.7.2 Preparation of 'Run Log' using Chemstation/Chemstation software.

7.7.2.1 Open Chemstation software by "clicking" on Icon.

7.7.2.2 Select "Sequence", then "Edit sample log table".

7.7.2.3 Select sample type for each sample. Sample types include the following:

CCB
CCV
MB
Client Samples
LCS

7.7.2.4 Number all of the samples

7.7.2.5 Change data file using the following format

41030501,

where:

4 = GC number
Number 1= Year: '01'
0305 = Date in MM DD format
01 = sequential file number

7.7.2.6 Click "OK" when completed

7.7.2.7 On the pull-down menu, select "Sequence", then "Save".

7.7.2.8 When the following prompt appears, change the last part to MM DD format indicating the current day.

C:\HPCHEM\4\Data\410305,

where:

410305 = Instrument 4, month "03", day "05"

7.7.2.9 When complete, click “OK”

7.7.2.10 On the pull-down menu, select “Sequence”, then “Run” to start the instrument run.

7.7.3 Editing Sequences

7.7.3.1 Click “File”, then “Edit”

7.7.3.2 Sample information can be directly entered into the pop up box that appears.

7.8 Standardization and Calibration

7.8.1 Retention time range.

7.8.1.1 Before establishing the range, make sure that the GC system is operating within optimum conditions.

7.8.1.2 Make three injections of the Florida Pro standard or a standard containing C_{10} and C_{28} throughout the course of a 72-hour time period. Serial injections over less than a 72-hour time period will result in retention time ranges that are too tight.

7.8.1.3 Calculate the standard deviation of the absolute retention times for the mixture.

7.8.1.4 The retention time range for the individual peaks, C_{10} and C_{28} is defined as a plus or minus three times the standard deviation of the absolute retention time for each component.

7.8.1.5 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ±0.05 minutes as a retention time window.

7.8.1.6 New retention time windows must be determined each time a new GC column is installed. Data is kept on file with the other test method records.

7.8.1.7 The retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component.

7.8.2 Daily measurement of the retention time window.

7.8.2.1 Each day that the test is run, the retention time of the retention time window standard is compared to the existing retention time window.

7.8.2.2 If the retention time range is not within the predetermined retention time range as discussed above, a new range must be determined.

7.8.2.3 A retention time standard must be run at the beginning of a sequence, every 12 hours, and at the end of a sequence or every 20 samples. It must be run every time a CCV standard is run.

7.9 Initial Calibration Procedure:

7.9.1 Initial calibration is performed using multi-point calibration at a minimum of 5 concentration levels. A retention time window standard must be analyzed at each initial calibration update to verify intergration range of C10-C28.

7.9.2 If the calibration standards have already been prepared and are in storage, remove them from the refrigerator and allow them to warm to room temperature. Be sure to check for signs of precipitation in the vials. Proceed to the next step to begin calibration.

7.9.3 Place the vials in the GC autosampler tray beginning at position one. It is recommended that standards be analyzed from the highest concentration to the lowest. This will ensure that any possible active sites in the instrument will not greatly affect the analysis of the calibration curve.

7.9.4 Record the sequence of standards in the GC Instrument Run log and create this sequence in the data acquisition system.

7.9.5 For narrow bore analyze the initial calibration standards using injections of 1.0 µL of each calibration standard.

7.9.6 Using the data system, generate the initial calibration report indicating the calibration factors and the percent relative standard deviation for the total area within C_{10} to C_{28} range at each of the calibration levels analyzed and the surrogate compound.

7.9.7 Calculate the calibration factor at each concentration as:

$$CF = \frac{\text{Total Peak Area in Calibration Range}}{\text{Mass of the Compound Injected (in nanograms)}}$$

7.9.8 Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{\text{CF}} = \frac{\sum_{i-1}^{n} CF_i}{n}$$

Where n is the number of standards analyzed.

7.9.9 Calculate the standard deviation (SD) and the RSD of the Calibration factors for each analyte as:

$$RSD = \frac{SD}{\overline{CF}} x\,100$$

$$\text{SD} = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}}$$

7.9.10 If the RSD (relative standard deviation) for each analyte is ≤20%, the initial calibration is considered linear and the mean calibration factor can be used to quantitate sample results.

7.9.11 If the RSD ≥20%, then linearity through the origin cannot be assumed. The analyst must use a linear regression calibration curve with Correlation Coefficient of 0.995 or greater.

7.9.12 Each initial calibration curve must be verified by analyzing a mid-level standard (ICV) containing each target analyte and prepared from a source other than that used for the ICAL. ICV must meet the criteria described in Section 7.10 (CCV).

7.10 Calibration Verification

7.10.1 Prior to analysis of any standards or samples, several GC parameters should be checked and recorded in order to ensure that operating conditions have not changed significantly from those of prior analyses.

7.10.2 The auto-sampler syringe rinse solution should be filled with MeCl2 and waste bottles should be emptied.

7.10.3 The daily measurement of the retention time window as described in 7.8.2 must be performed. Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty (20) samples and at the end of the analysis (after every 10 samples is *recommended* to minimize the number of samples requiring re-injection when QC limits are exceeded). This includes both a retention time standard and a calibration standard.

7.10.4 The % Difference (calibration factor) or % Drift (calculated concentration) for the DRO CCV must be within ±20%. Surrogate % Difference or % Drift may be within ±20%. **For regulatory reporting in South Carolina, averaging of % Drift or % Difference is not allowed and each target analyte must meet the ±20% criteria.**

7.10.5 Calculate the % Difference as:

$$\%\ \text{Difference} = \frac{\text{CF}_v - \overline{CF}}{\overline{CF}} X 100$$

7.10.6 Calculate the % Drift as:

$$\%\ \text{Drift} = \frac{\text{Calculated concentration - Theoretical concentration}}{\text{Theoretical concentration}} X 100$$

7.10.7 If the calibration factor or calculated concentration for the CCV is within ±20% of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may conduct sample analysis. **For regulatory reporting in South Carolina, averaging of % Drift or % Difference is not allowed and each target analyte must meet the ±20% criteria.**

7.11 Setting up a sequence

7.11.1 Open ChemStation software (revision A.06.06).

7.11.2 Open DRO method by clicking on the “method” drop down menu and double clicking on the method that is desired.

7.11.3 Set up a sequence.

7.11.3.1 Select “Sequence”, then “Sequence Parameters”.

7.11.3.2 Check off “Prefix Counter”.

7.11.3.3 Change Prefix to month and day (mm/dd/yy).

7.11.3.4 Set system to shut down if running or else indicate "no".

7.11.3.5 Create or overwrite the sequence. The following sequence should be followed:

CCB (CH_2Cl_2)
Retention Time Standard (FL-PRO)
CCV Standard or second source standard ICV
Method Blank
LCS/LCSD
Sample extracts, MS, and MSD(less than 20 samples).
Closing CCB/CCV (run within 12 hrs, less than 20 samples or at the end of the sequence)

7.11.3.6 Save the sequence

7.11.3.7 At the prompt, name the sequence as mm/dd/yy. For example, "110700".

7.12 Sample Analysis

7.12.1 Analysis is accomplished by injection of a 1-µL sample into a Gas Chromatograph using a FID detector. The sample results are compared to standards in the range of C_{10} to C_{28}.

7.13 Data interpretation

7.13.1 Qualitative analysis

The qualitative identification of compounds determined by this method is based on the retention times of a mixed carbon standard ranging from C_{10} to C_{28}.

7.13.2 The chromatograms of the samples can be compared to the chromatograms of petroleum standards to estimate the type of petroleum contamination.

7.14 Quantitative analysis

7.14.1 When linearity exists, calculate the concentration of each identified analyte in the sample extract is calculated as follows:

$$\text{Concentration(mg/L)} = \frac{Ax}{RFavg * Vo}$$

Where:

Ax = area of the chromatogram from C_{10} to C_{28}.

RF_{avg} = mean relative response factor for compound being measured.
V_o = Volume of sample injected taking into account any dilutions that may have been made.

7.14.2 The chromatogram area is determined by drawing a forced baseline between the retention times of C_{10} to C_{28} and including **ALL** area under the characteristic hydrocarbon "hump". Surrogate concentrations are determined through the use of a valley to valley integration and the areas are subtracted from the total area of the chromatogram within the retention time window.

7.14.3 The concentrations determined in section 7.9 are applied to factors such as initial sample volume (liters) and or initial sample weight (kg) to determine the original concentration in the original sample. This information is automatically determined through the use of the LIMS system.

7.15 Analytical Checklists. Checklists must be completed for each phase of the procedure. These checklists are placed into and become a part of the completed data package. Procedures for data handling are included in this SOP.

Table 7-3
3510_DRO

____: PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3510_DRO AND DRO_W / 8015B_EXT_W)

____: BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____: PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____: ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (SEPARATORY FUNNELS, ZYMARK TUBES, FUNNELS AND SYRINGE)

____: USE WHITE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____: PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____: TRANSFER 1000 ml SAMPLE INTO SEPARATORY FUNNEL. ADD 1000 ml DI WATER FOR MB AND LCS (AND LCSD IF THE SAMPLE AMOUNT IS INSUFFICIENT TO PREP MS/MSD) (IF ONLY 2 BOTTLES HAVE BEEN PROVIDED BY THE CLIENT, USE 500 ml FOR SAMPLE, MS AND MSD FOR THE QC SAMPLE)

____: CHECK pH OF THE SAMPLES, pH SHOULD BE LESS THAN 2

____: ADD 5 ml SULFURIC ACID TO SAMPLES TO ADJUST THE pH TO LESS THAN 2 IF NECESSARY. CHECK THE pH OF THE SAMPLES, ADD MORE ACID IF NECESSARY.

____: SPIKE LCS, MS AND MSD WITH 1.0 ml DRO SPIKE AND ADD 1.0 ml DRO SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE)

____: ADD 60 ml METHYLENE CHLORIDE TO THE SAMPLE, CAP THE SEPARATORY FUNNEL AND SHAKE FOR ABOUT 2 MINUTES

____: LET THE METHYLENE CHLORIDE SETTLE FOR ABOUT 2-3 MINUTES AND FILTER THE BOTTOM METHYLENE CHLORIDE PORTION THROUGH FILTER PAPER WITH SODIUM SULFATE INTO THE ZYMARK TUBE

____: REPEAT THE EXTRACTION 2 MORE TIMES WITH 60 ml METHYLENE CHLORIDE EACH

____: CONCENTRATE THE SAMPLES IN THE TURBOVAP WITH SETTINGS AT 39°C AND 20 PSI

____: CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP BEEPS

____: RINSE THE ZYMARK TUBE WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE ZYMARK TUBE AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____: LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-4
Checklist for 3535_DRO (SPE)

____: PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (Prep Code AND DRO_W/8015B_EXT_W)

____: BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____: REMOVE THE SAMPLE BOTTLES FROM THE FRIDGE AND LET THEM REACH THE ROOM TEMPERATURE. CHECK PREP AND TEST INFO

____: USE WHITE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE BOTTLES

____: PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____: IF THERE ARE ANY SAMPLES THAT HAVE SEDIMENT ON THE BOTTOM, TRANSFER THE LIQUID PORTION INTO A CLEAN 1L AMBER GLASS BOTTLE AND LABEL IT.

____: PUT 1000 ml DI WATER INTO 1L AMBER GLASS BOTTLE FOR MB AND LCS (AND LCSD IF THE SAMPLE AMOUNT IS INSUFFICIENT TO PREP MS/MSD). (IF ONLY 2 BOTTLES HAVE BEEN PROVIDED BY THE CLIENT, USE 500 ml FOR SAMPLE, MS AND MSD FOR THE QC SAMPLE)

____: SPIKE LCS, MS AND MSD WITH 1.0 ml OF 1000 ppm DRO SPIKE AND ADD 1.0 ml OF 100 ppm DRO SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE) (USE 0.5 ml SPIKE AND SURROGATE IF YOU START WITH 500 ml SAMPLE)

____: ADJUST THE PH OF EACH SAMPLE AND QC SAMPLE TO LESS THAN 2 BY ADDING 2-3 ml OF 1:1 HCl. CAP THE BOTTLES AND MIX WELL.

____: LOAD THE SPEEDISK H_20-PHOBIC DVB (JT BAKER 8068-06) FILTER ONTO THE INSTRUMENT.

____: REMOVE CAP FROM THE SAMPLE BOTTLES, PLACE A SMALL PIECE OF ALUMINUM FOIL OVER THE OPENING AND SCREW ON THE BOTTLE CAP ADAPTER.

____: PLACE A CLEAN 40 ml VOA VIAL ONTO THE INSTRUMENT.

____: LOAD THE 1L SAMPLE BOTTLES ONTO THE INSTRUMENT.

____: LOAD METHOD 8270.1 AND PROCESS THE SAMPLES.

____: REMOVE THE VOA VIAL WITH THE EXTRACT FROM THE INSTRUMENT.

____: POUR EXTRACT INTO THE DRYDISK RESERVOIR AND OPEN THE STOPCOCK. ALLOW TO DRAIN ABOUT 30 SECONDS.

____: CONCENTRATE THE SAMPLES IN THE TURBOVAP WITH SETTINGS AT 39°C AND 20 PSI

____: CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP BEEPS

____: RINSE THE ZYMARK TUBE WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE ZYMARK TUBE AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____: LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-5
3550_DRO

____: PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3550_DRO AND DRO_S / 8015B_EXT_S)

____: BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____: PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____: ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (GLASS JARS, ZYMARK TUBES, FUNNELS AND SYRINGE)

____: USE WHITE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____: PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____: WEIGH 30 g OF SAMPLE INTO GLASS JARS. USE 30 g OF SODIUM SULFATE FOR MB AND LCS

____: DRY THE SAMPLES WITH SODIUM SULFATE. SAMPLES SHOULD BE FREE FLOWING. TRY TO MINIMIZE THE PARTICLE SIZE AS MUCH AS POSSIBLE.

____: ADD 100 ml METHYLENE CHLORIDE TO THE SAMPLES

____: SPIKE LCS, MS AND MSD WITH 1.0 ml DRO SPIKE AND ADD 1.0 ml DRO SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE)

____: SONICATE SAMPLES 3 TIMES AT 3 MINUTE INTERVALS WITH PULSE SETTING AT 0.5 SEC. ADD 100ml METHYLENE CHLORIDE BEFORE EACH SONICATION

____: FILTER EXTRACTIONS INTO ZYMARK TUBES THROUGH FILTER PAPER WITH SODIUM SULFATE. RINSE THE SAMPLES WELL WITH METHYLENE CHLORIDE AFTER THE SONICATIONS AND FILTER THAT TO THE ZYMARK TUBE ALSO.

____: CONCENTRATE THE SAMPLES IN THE TURBOVAP WITH SETTINGS AT 39°C AND 20 PSI

____: CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAPBEEPS

____: RINSE THE ZYMARK TUBE WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE ZYMARK TUBE AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____: LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-6
3580A_DRO

____: PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3580A_DRO AND DRO_X)

____: BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____: PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____: USE WHITE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____: PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____: WEIGH 1 g OF SAMPLE INTO LABELED BOROSILICATE CULTURE TUBES

____: SPIKE LCS, MS AND MSD WITH 2.5 ml DRO SPIKE AND ADD 2.5 ml DRO SURROGATE TO EACH SAMPLE AND QC SAMPLE

____: DILUTE THE SAMPLES TO 10.0 ml WITH METHYLENE CHLORIDE

____: SHAKE OR PIPET STIR FOR 2 MINUTES AND ALLOW IT TO SETTLE. (IF THE SAMPLE HAS A BAD AND/OR NOT SEPARATE FROM THE SOLVENT FILTER IT THORUGH A FILTER PAPER WITH SODIUM SULFATE IN IT)

____: TRANSFER 1.0 ml SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL AND CAP THE VIAL TIGHTLY. TRANSFER THE REST OF THE SAMPLE INTO A 10.0 ml VIAL FOR STORAGE

____: LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

TABLE 7-7

AES DATA REVIEW CHECKLIST
GC 8015 (DRO, DAI, TEPH)

Batch ID: _____ Run No.:_____

SAMPLE RECEIPT
QA Analyst

☐☐: Have all sample ID's been recorded into the logbook and do they match exactly the IDs in LIMS
☐☐: Are all initial and final volumes recorded into the logbook and do they match the initial and final volumes in LIMS
☐☐: Narrative added to the work order as to why initial and/or final volumes were modified?
Narrative for elevated RLS due to limited sample volume or reduced volume prepped due to matrix.

INITIAL RAW DATA REVIEW

☐☐: All raw data files and run log(s) been printed to pdf and posted to Portal Server(must include all chromatograms, overlays, screen shots, etc. where applicable) **PROPERLY SCALED CHROMATOGRAMS MUST BE PRESENT SO THAT BASELINES/PEAK INTEGRATIONS CAN BE VISUALLY REVIEWED FROM PORTAL FILES**
☐☐: All instrument standard IDs included on raw data or run log
☐☐: Current and approved cal curve used for quantitation
☐☐: All calibration verification criteria met for both columns **(No requires CAR if reported to LIMS)**
☐☐: Is there opening/closing CCV or one every 12 hours?
☐☐: Retention time check standard run with every CCV **(No requires CAR if reported to LIMS)**
☐☐: All RTs for instrument and batch QC are within RT window **(No requires CAR if reported to LIMS)**
☐☐: All product ranges properly integrated with baseline points properly assigned. **ALL TARGET ANALYTE RANGES MUST BE INTEGRATED IN A MANNER CONSISTANT WITH THE INTEGRATIONS USED FOR THE ICAL STDS**
☐☐: Background sufficiently low to allow for target identification at the PQL at the dilution run or data turned off for rerun at higher dilution
☐☐: Sample not over-diluted based on background or data turned off for rerun at lower dilution
☐☐: Check duplicate run data for all samples to ensure no compounds are double reported and dilution values match closely with original run.

GO TO LIMS "MAIN" RUN SCREEN

☐☐: All Sample IDs properly assigned per Backlog Report (double click on each SampleID to verify)
☐☐: All Test Codes properly assigned per Backlog Report
☐☐: All instrument QC run at required frequency
☐☐: All Sample Types properly assigned
☐☐: All samples linked to the Prep Batch properly with correct PFac, SpkFac and OFAC
☐☐: All dilution factors entered correctly per the raw data/run logs
☐☐: All Blkref, SPKref, RPDref and CCVref assigned correctly
☐☐: All Comments present are addressed **(May require CAR)**

GO TO "DATA" SCREEN

☐☐: Calculate Sequence to ensure LIMS calculations are complete
☐☐: Are all CCB/instrument blanks below PQL **(No requires CAR if reported to LIMS)**
☐☐: Are there any B flags for target analytes indicating MB hits above PQLs **(Yes requires CAR)**
☐☐: Are there any S and/or R flags for spike cmpds and/or surrogates in LCS/LCSD **(Yes requires CAR)**
☐☐: Are there any S and/or R flags for spike cmpds and/or surrogates in MS/MSD **(Yes requires CAR)**
☐☐: Are there any H flags present for any samples **(Yes requires CAR)**

TABLE 7-7 Cont'd.

☐☐: Are there any E flags for target analytes on samples and/or Batch QC turned on for reporting **(Yes requires CAR)**
☐☐: Are there any J flags on any tartget analytes selected to report on diluted sample runs? **(Yes requires reanalysis at lower dilution or CAR to narrate elevated reporting limits)**
☐☐: Are there "*" qualifiers for J flagged target analytes? **(Yes requires removal of "*" qualifiers)**
☐☐: Are there any target analytes reported as BRL with elevated PQLs due to dilution or reduced prep volume **(Yes requires CAR)**
☐☐: Do all LIMS raw values match values on raw data
☐☐: Have at least 2 sample's final results been manually calculated to verify LIMS calculations
☐☐: Check duplicate run data for all samples to ensure no compounds are double reported and dilution values match closely with original run.

PRIOR TO FINAL QA APPROVAL

☐NA: Have all CARs been closed and narratives written prior to QA.

DO NOT USE MATRIX INTERFERENCE NARRATIVES WITHOUT CHROMATOGRAPHIC EVIDENCE FOR MATRIX INTERFERENCE AND/OR PREP COMMENTS INDICATING MATRIX INTERFERENCE UNLESS CONFIRMED BY REEXTRACTION AND REANALYSIS.

☐NA: Have PM, Lab Manager and PM Director been notified for any CAR resulting in re-extract and/or due date exceeded

CAR #: _____ _____ _____ _____ _____

Analyst Signature /Date/Time: _____

Reviewer Signature/Date/Time: _____

Comments: _____

_____

_____

_____

GC_8015_TEPH_Data Review Checklist_01182010.doc Page 2 of 2

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP #QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Calibration of the Gas Chromatograph. This is accomplished through a 5-point calibration curve. The points on the curve must meet a 20 %RSD when comparing calibration factors to determine if the calibration curve is linear. If the RSD ≥20%, then linearity through the origin cannot be assumed. The analyst must use a linear regression calibration curve with Correlation Coefficient of 0.995 or greater. The verification of the curve must meet the criteria described in section 8.1.3 and must be performed using a second source standard.

8.1.3 Calibration Curve Verification (CCV). Each day at the beginning of the sequence, a CCV must be performed. The % Difference (calibration factor) or % Drift (calculated concentration) for the DRO CCV must be within ±20%. Surrogate % Difference or % Drift may be within ±20%. **For regulatory reporting in South Carolina, averaging of % Drift or % Difference is not allowed and each target analyte must meet the ±20% criteria.**

8.1.4 Retention time window. The retention time for each analyte is compared over a 72-hour time period and the average retention time calculated. Daily retention times must fall within specified time windows.

8.1.5 Method Detection Limit Study. The method detection limit is calculated by analyzing at least seven replicates prepared in blank water at concentrations approximately equal to the PQL for each target analyte. Each calculated MDL must be <PQL. MDL's are to be performed annually or whenever instrument conditions have changes that will affect the established detection limits.

8.1.6 Method blank. Reagent blank analyses must be performed at the following frequency: Every twenty (20) samples of similar concentration and/or sample matrix or whenever samples are extracted and analyzed by the same procedure at same time, whichever is more frequent. The concentration of the method blank of any analyte of interest should not exceed the laboratory established practical quantitation limit (PQL). Target analytes detected in Method blanks at levels >PQL must be handled in accordance with Section 8.2.

8.1.7 Surrogate Recovery. All samples, blanks, and QC samples are fortified with surrogate spiking compound before extraction and injection in order to monitor sample extraction efficiency. The recovery of the surrogate compound must be within the recovery limits established by the laboratory. Recovery outside control limits must be handled in accordance with Section 8.2. For SC Samples, surrogate recovery must be within 70 -130%

8.1.8 Laboratory Control Sample (LCS). The Laboratory Control Sample (LCS) is used to monitor, assess and document laboratory method performance and is performed on every batch or twenty samples, whichever occurs more frequently. The recovery of the analytes must meet established laboratory guidelines. Recovery outside control limits must be handled in accordance with Section 8.2. An LCSD may be analyzed in the absence of MS/MSD. For SC Samples, LCS must be within 70 -130%.

8.1.9 Sample spike and duplicate spike. Matrix spikes and matrix spike duplicates are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples. The recovery of the analytes must meet established laboratory guidelines. Recovery outside control limits must be handled in accordance with Section 8.2.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in Section 5 of SOP# QA-01000, “Quality Assurance Manual”. The tables in this section include corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance and any procedure deviations in appropriate logbooks.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available (i.e., gloves, lab coats, goggles, and dust masks). Reference files of OSHA

regulations and MSDS's are available to all personnel involved in the chemical analysis. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

9.2 Work with any of these compounds in high concentrations should be performed in a hood. An NIOSH approved toxic gas respirator should be worn when the analyst handles very high concentrations of these toxic compounds.

9.3 The constituents within the various samples can contain benzene, benzo(a)pyrene, and other carcinogenic compounds as they typically come from petroleum spills. Handling of samples should be performed while wearing protective gloves and within the fume hood.

9.4 Charcoal traps should be installed on the GC split vents of the instruments with split/splitless injectors to prevent airborne contamination of the work area. All vacuum pumps (Edwards) should contain vapor traps on the outlet pump manifold.

10.0 DATA REPORTING

10.1 The LIMS system automatically rounds the data based upon factors that are set up for each test category. Typically, the LIMS reports to two significant figures.

10.2 The reporting limits can be changed in LIMS. Various laboratory personnel including project managers have the rights to change the limits per the requirements of the clients. Reporting limits are based upon the MDLs developed for each test.

10.3 Calculation of results using Chemstation software.

10.3.1 Open Chemstation software by “clicking” on Icon

10.3.2 On the pull-down menu, click on “Data Analysis”

10.3.3 On the pull-down menu, click on “Load”, then “Data file”. The path C:\hpchem\2\data should appear.

10.3.4 Using the “arrow” key, scroll down to the desired sequence number. The sequence number represents the GC number, year, month and day in a format 410321. Note that the left side of the box contains all of the samples in the run. Each sample can be selected by positioning the mouse pointer over the sample and double clicking it.

10.3.5 Once a sample has been selected, On the pull-down menu, click on “Load”, then “Method”. Select the method DRO0225.M where DRO = the method, 02,25 = the date. From this point, the method will remain the same for each sample selection.

10.3.6 To calculate a result, on the pull-down menu, click on "Quant", then "Calculate and generate report". An alternative method is to click on "Quant", then "Int" and "Integrate".

10.3.7 Review the Chemstation calculated result by clicking on "Quant", then "Q edit". To enlarge the various areas of the chromatogram, place the pointer on the chromatogram and right click the mouse. Drag the mouse over the area to enlarge. Double click the chromatogram to return it to its original size.

10.3.8 Once the chromatogram has been enlarged, the baselines can be redrawn by placing the mouse at one end of the peak and moving it across the bottom while holding down the right mouse. Release the mouse when the line is the correct length.

10.4 Completed data is stored in the "C" drive of the acquisition computer under the following directory: "C:\HPCHEM\410305" where 4 is the instrument number, 1 is the year "01", 0305 is the date in MM DD format.

10.4.1 Prior to moving files for final storage, open the completed data folder and change the status of the data to "frozen" using the following procedure.

10.4.1.1 In Chemstation, click "Tools", then "Change Data State"

10.4.1.2 Click the radio button next to "Frozen"

10.4.1.3 Click "OK". Exit to save the changes

10.4.1.4 Using NT Explorer, Find the file on the "C' drive in the directory "C:\HPChem\1 where 1 = GC 3 and 2 = GC 4

10.4.1.5 Highlight the file and Click "Cut"

10.4.1.6 Using NT Explorer, find and select the folder in the computer called "Storage".

10.4.1.7 Locate the proper directory: either GC 2 or GC 4 and click "Save" to save the file to this directory.

10.4.1.8 Periodically, these files are written to a writeable "CD" and stored off site by the VP of Technical Operations.

10.5 Current MDLs for all parameters may be found in tables in Section 5 of SOP# QA-01000, "Quality Assurance Manual".

11.0 FILE MAINTENANCE

11.1 All data are stored on the Portal Server for a period of 5 years.

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto portable hard drives. Two copies are made. One copy is stored on the laboratory premises, while the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks. Instrument logbooks must be completed each time that any maintenance is performed upon the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as replacement of motors in tower autosamplers.

Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.2 Routine Maintenance: Typical routine maintenance consists of keeping the system clean and insuring that chromatography remains acceptable. Examples would be peak tailing and degradation of DDT and Dieldrin in pesticide analysis. The table below indicates the frequency of routine maintenance for various instrument types within the laboratory.

Maintenance Action	Recommended Frequency
Changing injection port liners	Weekly or when chromatography is affected
Trimming column	Monthly or when chromatography is affected
Cleaning GC/MS source	Weekly or when chromatography is affected
Changing GC/HPLC Column	Annually or when other attempts to resolve chromatography fail.

12.3 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as autosampler that will not function. If this type of problem occurs, contact the department manager for assistance.

13.0 METHOD PERFORMANCE

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The reporting limit RL is defined as the concentration of a substance that is above the level of uncertainty. The concentration listed in the table in Section VIII was obtained using reagent water. Similar results can be achieved using representative wastewater. The MDL actually achieved is a given analysis will vary depending on instrument sensitivity and matrix effects.

13.2 This method is recommended for use in the concentration range from the MDL to 1000 x MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000 x MDL.

13.3 Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Tables 13-1 and 13-2 lists single operator precision and accuracy found in SW-846, Method 8015B.

Table 13-1
RESULTS FROM ANALYSIS OF LOW AROMATIC DIESEL BY GC/FID (2 GRAM SAMPLES)
(5 replicates per test)

Spike Concentration	**Analysis Results**
12.5 ppm	ND
75 ppm	54 +/- 7 ppm
105 ppm	90 +/- 15 ppm
150 ppm	125 +/- 12 ppm
1000 ppm	960 +/- 105 ppm

Samples were prepared using 2 gram aliquots of sandy loam soil spiked with known amounts of low aromatic diesel. Extractions were accomplished using methylene chloride as a solvent (Method 3550, high concentration option.

Low aromatic diesel is sold in California. For this study it was purchased at a gas station in San Diego, CA.

Table 13-2
RESULTS FOR ANALYSIS OF LOW AROMATIC DIESEL BY GC/FID (10 GRAM SAMPLES)
(5 replicates per test)

Spike Concentration	**Analysis Results**
25 ppm	51.2 +/- 6.4 ppm
75 ppm	75.9 +/- 7.8 ppm
125 ppm	98.9 +/- 5.2 ppm
150 ppm	162 +/- 10.4 ppm

Samples were prepared using 10 gram aliquots of sandy loam soil spiked with known amounts of regular #2 diesel purchased at a gas station in Northern Virginia. Extractions were accomplished using methylene chloride as a solvent (Method 3550).

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 SOP HS-03005 Waste Disposal and SOP SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade –A dry chemical that has been dried at 250°C for 4 hours cooled and stored in a desiccator.

15.2 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to distilled water or clean soil and the concentration is measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.3 DI water- Deionized water

15.4 RSD – Relative Standard Deviation

15.5 RF – Response factor. Determined as the concentration of a sample divided by the chromatographic area of the peak produced by the sample.

15.6 MS- Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.7 MSD- Matrix Spike Duplicate.

15.8 CCV - Continuing calibration verification standard. Must be varied thoughout the daily runs, that is the concentration must be low, middle, and sometimes at the upper end of the calibration curve.

15.9 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot that the calibration standard.

15.10 LCSD - Laboratory Control Sample Duplicate

15.11 DRO – Diesel Range Organics. Usually a measure of the organics with retention times between C_{10} and C_{28}.

16.0 REFERENCES

16.1 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 8000B,"Determinative Chromatographic Separations", Rev. 2, December 1996.

16.2 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 8015C, "Nonhalogenated Organics using GC/FID", Rev. 3, February 2007.

16.3 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3510C, "Liquid Liquid Extraction", Rev. 3, December 1996.

16.4 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3520C, "Continuous Liquid Liquid Extraction", Rev. 3, December 1996.

16.5 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3550C, "Ultrasonic Disruption", Rev. 3, February 2007.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://portal/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://portal/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

18.0 SOP REVISION HISTORY

Revision Date	Revision #	Summary of and Reason for Changes/Updates	Responsible for Revision
6/3/2003	2	Update	Greg Jones
4/4/2005	3	Update	Greg Jones
3/9/2009	4	SC/MUR Update	Dana Till
2/6/2012	5	SC Audit Update to Sections 5.0, 8.1.7, and 8.1.8.	Dana Till

AES, Inc.
3785 Presidential Pkwy.
Atlanta, GA 30340

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DOCUMENT CONTROL NUMBER: Rev. 12

DOCUMENT DISTRIBUTION NUMBER: OA-11004

ELECTRONIC DOCUMENT LOCATION
AES Portal Server: http://portal/Procedures/Standard Operating Procedures

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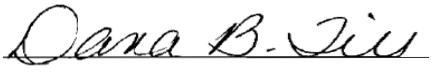
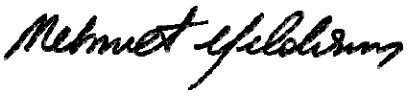
Technical Director:  Date: 2/13/2012

Laboratory Manager:  Date: 2/13/2012

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STANDARD OPERATING PROCEDURE FOR
CHLORINATED HERBICIDES BY GC BY EPA SW-846 METHOD 8151A

TABLE OF CONTENTS

Page

1.0 SCOPE AND APPLICATION

1.1 EPA SW-846 Method 8151A is a capillary gas chromatographic (GC) method for determining certain chlorinated acid herbicides and related compounds in aqueous, soil, and waste matrices. The following compounds can be determined by this method:

Compound Name	Cas No.[a]
2,4-D	94-75-7
2,4-DB	94-28-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2

[a] Chemical Abstract Services Registry Number.

1.2 Because these compounds are produced and used in various forms (acid, salt, ester, etc.), Method 8151 describes a hydrolysis step that can be used to convert herbicide esters into the acid form prior to analysis. Herbicide esters generally have a half-life of less than one week in soil.

1.3 When this procedure is used to analyze unfamiliar samples, compound identification should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column.

1.4 The estimated detection limits for each of the compounds in aqueous and soil matrices are listed in the QA Manual, Section 5. The detection limits for a specific waste sample may differ from those listed, depending upon the nature of the interferences and the sample matrix.

1.5 The following compounds may also be determined using this method:

Compound Name	Cas No.[a]
Acifluorfen	50594-66-6
Bentazon	25057-89-0
Chloramben	133-90-4
DCPA diacid[b]	2136-79-0
3,5-Dichlorobenzoic acid	51-36-5
5-Hydroxydicamba	7600-50-2
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5
Picloram	1918-02-1

[a] Chemical Abstract Services Registry Number.
[b] DCPA monoacid and diacid metabolites included in method scope; DCPA diacid metabolite is used for validation studies. DCPA is a dimethyl ester.

1.6 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatography, and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.7 Only experienced or supervised analysts should be allowed to work with diazomethane due to the potential hazards associated with its use (the compound is explosive and carcinogenic).

1.8 **Because acid herbicides and methyl esters of acid herbicides decompose in the presence of ultraviolet light, the entire extraction, hydrolysis, and derivatization procedure should be carried out uninterrupted from start to finish in its entirety.**

1.9 This method incorporates the preparative procedures for the extraction of waste, water, and solid samples by SW-846 Methods 3580A (Waste Dilution), 3510C (Separatory Funnel Extraction), and Shaker Extraction of solid materials.

2.0 SUMMARY OF METHOD

2.1 This SOP provides the hydrolysis, extraction, derivitization, and gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil and waste samples.

2.2 Water samples are hydrolyzed and extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by GC with and ECD detector. The results are reported as acid equivalents.

2.3 Soil and waste samples are hydrolyzed, extracted, and esterified with diazomethane. The derivatives are determined by GC with and ECD detector. The results are reported as acid equivalents.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contamination in solvents, reagents, glassware, and other sample processing hardware, that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free of interferences, under the conditions of the analysis, by running laboratory reagent blanks.

3.1.1 Glassware must be scrupulously cleaned. As soon as possible after use, clean each piece of glassware by first rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses first with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed

and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending on the nature and diversity of the waste being sampled.

3.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. Although not performed in this laboratory, the determination using pentafluorobenzylation is more sensitive, and thus more prone to interferences from the presence of organic acids or phenols, than methylation.

3.4 Alkaline hydrolysis and subsequent solvent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. **However, if any residual acetone remains from the extraction of solids, hydrolysis may result in the loss of Dinoseb and the formation of aldol condensation products.**

3.5 The herbicides, being strong organic acids react readily with alkaline substances and may be lost during analysis. **Therefore, glassware and glass wool must be acid rinsed, and sodium sulfate must be acidified prior to use to avoid this possibility.**

3.6 **Sample extracts should be dry prior to methylation or poor recoveries will be obtained.**

4.0 SAMPLE COLLECTION, PRESERVATION AND HOLDING TIMES

4.1 Water samples must be extracted within 7 days of sample collection, and must be completely analyzed within 40 days of extraction. Soil/sediment samples must be extracted within 14 days of sample collection and must be completely analyzed within 40 days of extraction.

4.2 Store all extracts in Teflon-sealed containers in the dark at $4^{o}C \pm 2°C$ until all analyses are performed.

5.0 REAGENTS AND STANDARDS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water. All references to water in this method refer to organic-free water, as defined that at the method detection limit of the compounds of interest an interfering substance is not observed.

5.3 Sodium hydroxide solution (10N), NaOH - Dissolve 400 g of NaOH in organic-free reagent water and dilute to 1.0 L. Caution, solution gets hot. Dissolve in ice water.

5.4 Potassium hydroxide solution (37% aqueous solution (W/V)), KOH – Dissolve 37 g of KOH in organic-free reagent water and diluted to 100 mL.

5.5 Phosphate buffer (0.1M), pH=2.5.

5.5.1 Dissolve 12 g sodium phosphate (NaH_2PO_4) in organic-free reagent water and dilute to 1.0 L.

5.5.2 Add phosphoric acid to adjust pH to 2.5.

5.6 N-methyl-N-nitroso-p-toluenesulfonamide (Diazald) – High purity, purchased directly from vendor (see Table 5-2).

5.7 Salicylic acid, H_2SiO_5 – 100-mesh powder, purchased directly from vendor (see Table 5-2). Store at 130°C.

5.8 Sodium sulfate (granular, acidified, anhydrous), Na_2SO_4 purchased directly from vendor (see Table 5-2). Prepare acidified sodium sulfate as follows.

5.8.1 Purify by heating at 400°C for 4 hours in a shallow tray, or by pre-cleaning the sodium sulfate with methylene chloride.

5.8.2 Acidify by slurrying 100-g sodium sulfate with enough diethyl ether to just cover the salt; then add 0.1 mL of concentrated sulfuric acid and mix thoroughly.

5.8.3 Remove the ether under vacuum. Mix 1 g of the resulting salt with 5 mL of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4

5.8.4 **Store the remaining solid at 130°C**.

5.9 Solvents – All solvents should be pesticide quality or equivalent and are purchased directly from vendor (see Table 5-2)

5.9.1 Acetone

5.9.2 Methylene chloride, CH_2Cl_2.

5.9.3 Methanol, CH_3OH.

5.9.4 Diethyl Ether, $C_2H_5OC_2H_5$. Must be free of peroxides as indicated by test strips.

NOTE: Diethyl ether used for this procedure should be stabilized with BHT, not with ethanol, because when ethanol-stabilized ether is used, the methylation reaction may not proceed efficiently, leading to low recoveries of target analytes.

5.9.5 Hexane, C_6H_{14}.

5.9.6 Isooctane, $(CH_3)_3CH_2CH(CH_3)_2$.

5.10 pH Adjustment Solutions

5.10.1 Sodium hydroxide, NaOH, 6N.

5.11 Sulfuric acid, H_2SO_4, 1:1. Mix equal volumes of acid and deionized water. Caution: add acid to water and mix in an ice bath to prevent overheating.

5.12 Stock Standards

5.12.1 Surrogate Stock Solution: Restek Cat. No. 32049, 2,4-Dichlorophenylacetic Acid Standard (Underivatized), 200µg/mL in methanol.

5.12.2 Primary Calibration Mix: Accustandard Underivatized Chlorinated Herbicides Standard Cat. No. M-8150A, 100µg/mL (2,4-D, 2,4-DB, 2,4,5-T, Silvex(2,4,5-TP), Dalapon, Dicamba, Dichloroprop, Dinoseb), 100 ug/mL 2,4-DB (additional), and 10,000µg/mL (MCPP, MCPA) in methanol.

5.12.3 Primary Pentachlorophenol Calibration Standard: Ultra Scientific Cat. No. PH-180-1, Underivatized Pentachlorophenol, 1000µg/mL in methanol.

5.12.4 Second Source Calibration Mix: Accustandard Underivatized Chlorinated Herbicides Standard Cat. No. M-8150A, 100µg/mL (2,4-D, 2,4-DB, 2,4,5-T, Silvex(2,4,5-TP), Dalapon, Dicamba, Dichloroprop, Dinoseb), 100ug/mL 2,4-DB (additional), and 10,000µg/mL (MCPP, MCPA) in methanol. **MUST BE DIFFERENT ACCUSTANDARD AND CHEMSERVICE LOT NO. FROM PRIMARY SOURCE**.

5.12.5 Second Source Pentachlorophenol Calibration Standard: Ultra Scientific Cat. No. EPA-1152, Underivatized Pentachlorophenol, 5000µg/mL in methanol.

5.13 Derivatized Initial Calibration Working Standard:

5.13.1 Add 1.0 mLof Primary Calibration Mix (5.12.2), 0.1mL Primary Pentachlorophenol Calibration Standard (5.12.3) and 0.5 ml Surrogate Stock Solution (5.12.1) to a 10mL volumetric flask and make to volume with hexane.

5.13.2 Derivatize per Section 7.6 for final concentrations of 10µg/mL (2,4-D, 2,4-DB, 2,4,5-T, Silvex(2,4,5-TP), Dalapon, Dicamba, Dichloroprop, Dinoseb, PCP, DCAA) and 1000µg/mL (MCPP, MCPA).

5.13.3 Make dilutions for each ICAL level per Table 5-1 below.

5.14 Derivatized Second Source (ICV) Working Standard:

5.14.1 Add 1.0mL of Second Source Calibration Mix (5.12.4), 0.02mL Second Source Pentachlorophenol Calibration Standard (5.12.5) and 0.5mL Surrogate Stock Solution (5.12.1) to a 10mL volumetric flask and make to volume with hexane.

5.14.2 Derivatize per Section 7.6 for final concentrations of 10µg/mL (2,4-D, 2,4-DB, 2,4,5-T, Silvex(2,4,5-TP), Dalapon, Dicamba, Dichloroprop, Dinoseb, PCP, DCAA) and 1000µg/mL (MCPP, MCPA).

5.14.3 Dilute 500µL of derivitized second source standard to final volume of 10mL in hexane for ICV at concentration equivalent to Level 4 of calibration curve.

Table 5-1
Standard Calibration Curve

Level No.	Working Std (µL) (5.13)	Final Vol (mL)	2,4-D Conc µg/L	2,4-DB Conc µg/L	2,4,5-T Conc µg/L	2,4,5-TP Silvex Conc µg/L	Dalapon Conc µg/L	Dicamba Conc µg/L	Dichlorprop Conc µg/L	Dinoseb Conc µg/L	MCPA Conc µg/L	MCPP Conc µg/L	PCP Conc µg/L	DCAA (Surr) Conc µg/L
1	100	10	100	100	**100**	**100**	100	**100**	100	100	**10000**	**10000**	**100**	100
2	250	10	**250**	**250**	250	250	**250**	250	**250**	**250**	25000	25000	250	250
3	400	10	400	400	400	400	400	400	400	400	40000	40000	400	400
4	500	10	500	500	500	500	500	500	500	500	50000	50000	500	500
5	750	10	750	750	750	750	750	750	750	750	75000	75000	750	750
6	1000	10	1000	1000	1000	1000	1000	1000	1000	1000	100000	100000	1000	1000
7*	1500	10	1500	1500	1500	1500	1500	1500	1500	1500	150000	150000	1500	1500
8*	2000	10	2000	2000	2000	2000	2000	2000	2000	2000	200000	200000	2000	2000

BOLD = Required low standard that must be included in curve.
*Optional level that can be used if linearity requirements are met.

5.14.4 A Standard Preparation Data Sheet and an entry on the Standard Index Sheet in the standard logbook must be completed for each standard prepared.

5.15 Underivatized Spiking Solution (LCS/MS): 5,000/50,000 µg/L

5.15.1 Add 2.5 ml of Primary or Second Source Calibration Mix (5.12.2 or .4) and 0.25mL of Primary Pentachlorophenol Calibration Standard (1000µg/mL) (5.12.5) to a 50mL volumetric flask and make to volume with acetone.

5.16 Stock standard solutions of the derivatized acids must be replaced after 1 year, or sooner if comparison with check standards indicates degradation. Stock standard solutions of the free acids degrade more quickly and should be replaced after two months, or sooner comparison with check standards indicates degradation. Working and intermediate standards must not be assigned an expiration date past the expiration date of the parent solution. Stock standards are stored refrigerated at 4°C ± 2°C.

5.17 Vendor List
The standards used in this test are purchased using the catalog numbers and vendors indicated below.

Table 5-2
Herbicide Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
Herbicide Mix (1st Source)	AccuStandard	100/10000 µg/mL	M8150A
Herbicide Mix (2nd Source)	AccuStandard**(2nd Lot #)**	100/10000 µg/mL	M8150A
2,4-DB-add. (1st Source)	ChemService	100 ug/mL	F958RPS
2,4-DB-add. (2nd Source)	ChemService **(2nd Lot #)**	100 ug/mL	F958RPS
Pentachlorophenol (1st)	Ultra	1000 µg/mL	PH-180-1
Pentachlorophenol (2nd)	Ultra	5000 ug/mL	EPA-1152
DCAA surrogate	Restek	200 µg/mL	32049
Sodium Sulfate	EM Science	Pure	SX0760E-20
Diazald	Sigma-Aldrich	Pure	D28000-25G
Carbitol	Sigma-Aldrich	Pure	537616-100ML
Acetone	EM Science	Pure	AX0116-1
Methanol	B&J	Pure	GZ-230-4
Sodium Hydroxide	JT Baker	Pure	3722-05
Sulfuric Acid	EM Science	Pure	SX1244-5
Potassium Hydroxide	EM Science	Pure	PX1480-1
Diethyl Ether*	B&J	Pure	106-4
Salicylic Acid	Aldrich	Pure	306363-100G
Sodium Chloride	VWR	Pure	VW6430-7
Methylene Chloride	EM Science	Pure	DX0831AE-39
Hexane	EM Science	Pure	HX0296-39
Isooctane	Fisher	Pure	AC32662-0010

* Stabilized

6.0 APPARATUS AND MATERIALS

6.1 Gas Chromatograph - Hewlett Packard Model 5890 Gas Chromatograph, equipped with a Hewlett Packard 7673 or 7673A Autosampler or equivalent. Data System- Hewlett Packard Chemstation with Enviroquant reporting software or equivalent.

6.1.1 Column 1: STX-CLP Pesticides, 30 meter x 0.53 mm ID, 0.5 µm film thickness, Restex Cat. No. 11545.

6.1.2 Column 2: STX_CLP Pesticides II, 30 meter x 0.53 mm ID, 0.42 µm film thickness, Restex Cat. No. 11445.

6.1.3 Hewlett Packard Detector - Electron Capture Detector.

6.2 Flow meter

6.3 250 µl glass inserts for crimp vials.

6.4 Gas tight syringes (1.0 mL, 500 µl, 250 µl, 100 µl, 50 µl, 10 µl)

6.5 10 ml and 20 ml Teflon-sealed screw-cap vials and caps.

6.6 1.5 ml crimp top vials.

6.7 1.5 ml Teflon lined crimp top caps.

6.8 1.5 ml amber screw thread vials.

6.9 Volumetric flasks (Class A) (0.5 ml, 5.0 ml, 10 ml, and 50 ml)

6.10 Diazomethane Bubbler.

6.11 250mL Zymark Turbovap Tubes.

6.12 Beaker – 600-mL, thick-walled.

6.13 Funnel - 75 mm, diameter.

6.14 Separatory funnel - 2000mL, with polytetrafluoroethylene (PTFE) stopcock.

6.15 Erlenmeyer flasks - 250-mLand 500mL, with a ground-glass joint at the neck.

6.16 Pipet- Pasteur, glass, disposable 9140mm x 5mm ID).

6.17 Filter paper – 15 cm diameter (Whatman No. 1 or equivalent).

6.18 Balance – analytical, capable of accurately weighing to 0.0001 g.

6.19 pH paper – wide range

6.20 Wrist shaker

6.21 Drying column – 400mm x 20mm ID Pyrex chromatographic column with Pyrex glass wool at bottom and a PTFE stopcock.

7.0 PROCEDURE

7.1 Preparation of extraction log form and extraction log in LIMS.

7.1.1 Each day the section supervisor prepares a work log. The log lists samples that are included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN EXTRACTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received in a "rush" status will have a chain of custody delivered to the extraction supervisor by the project manager.

7.1.1.2 Prepare a written extraction log using the 8151 logbook that is kept in the extraction supervisor's office. The following entries must be made in the log.

7.1.1.2.1 Date and time that the batch is opened or the date and time the various extraction systems are started.

7.1.1.2.2 All sample(s) included in the extraction batch.

7.1.1.2.3 Volume of samples extracted.

7.1.1.2.4 Date and time that the liquid/liquid extraction is completed.

7.1.1.2.5 Extraction procedure employed.

7.1.1.2.6 The initials of the extraction analysts.

7.1.1.2.7 Laboratory number of all reagents used including surrogate standard, spiking standard, diethyl ether, and sodium sulfate.

7.1.1.2.8 Volume of all reagents used including surrogate standard, spiking standard, and methylene chloride.

7.1.1.2.9 Final volume of all concentrates.

7.1.1.2.10 Date and time the batch is closed.

7.1.1.2.11 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.1.1.3.1 Open Prep Batch in LIMS by double clicking the "Prep" box.

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS.

7.1.1.3.3 Select the Prep Code "3510_HERB" from the pull down list. The LIMS will automatically assign an MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCSD and enter the information.

7.1.1.3.4 Enter the technician's initials from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this method. Note that the list contains all samples requiring extraction by method 3510 not just samples requiring analysis by Method 8151.

7.1.1.3.6 Select the samples to be included in the batch. The samples can be individually selected by highlighting the sample and clicking the "$\rightarrow$" arrow or all samples can be selected by clicking the "$\Rightarrow$" arrow.

7.1.1.3.7 Samples that have be selected for re-analysis can be manually added to the sample list.

7.1.1.3.8 "Save" the batch by clicking a previous batch number on the list and then returning to the newly created batch.

7.1.2 Table 7-1 indicates the type of samples that comprise a preparatory batch. Note: NELAC requirements specify that the maximum number of client samples in a preparatory batch can not exceed 20. Further, a preparatory batch can not be left "open" for a time period that exceeds 24 hours.

Table 7-1

Samples required in a Preparatory Batch

Method Blank (MB)
LCS and LCSD
Client Samples
MS and MSD (If supplied by client)

7.2 Extraction of high concentration waste samples. **This procedure should be carried out in its entirety without delay. The sought after analytes decompose in the presence of ultraviolet light**.

7.2.1 Remove the samples to be extracted from refrigerator R10 so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the subsequent steps.

7.2.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS. At times the wrong extraction procedure is logged into LIMS (e.g., Continuous Liquid/Liquid Extraction instead of Separatory Funnel Extraction). If this is the case, contact the section supervisor.

7.2.1.2 Contact the section supervisor. He will contact the Project Manager to determine the appropriate actions to be followed.

7.2.2 Prepare the Borosilicate culture tubes by rinsing them with a 50% sulfuric acid solution, followed by distilled water, and finally HPLC grade acetone.

7.2.3 Transfer approximately 1 g of waste sample to the tube (record weight to the nearest 0.1 g). Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

7.2.4 Add 1.0-ml of surrogate spiking solution to all samples and blanks. For the sample in each analytical batch selected for spiking, add 1.0 ml of the matrix-spiking standard.

7.2.5 Adjust pH to <2 with HCl.

7.2.6 Add approx. 5 ml with diethyl ether and mix thoroughly. Dewater by passing through acidified NaSO4 (5.8) and acidified glass wool. Concentrate sample to 1 mL in Zymark, Label as in Section 7.2.7, and proceed to Section 7.2.8, Hydrolysis.

7.2.7 Label each sample using the red label tape and a "Sharpie". Red tape is used for 8151 Herbicide analyses. The information included on the label is the sample name, batch number, test type, and sample matrix as indicated in the example below.

Example Label used in Extraction Laboratory

MB #9999 8151A

7.2.8 Hydrolysis

7.2.8.1 Transfer 1.0mL to a 250-mL Erlenmeyer flask with a ground-glass joint at the neck. Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of DI water to the sample. Add boiling chips to the K-D flask. Reflux the mixture on a water bath at 60-65^{o}C until the hydrolysis step is completed (usually 1-2 hours). Remove the flasks from the water bath and cool to room temperature.

CAUTION: The presence of residual acetone will result in the formation of aldol condensation products which will cause GC interference.

7.2.8.2 Transfer the hydrolyzed aqueous solution to a 500-mL separatory funnel and extract the solution three times with 100-mL portions of methylene chloride. Discard the methylene chloride phase. At this point, the basic (aqueous) solution contains the herbicide salts.

7.2.8.3 Adjust the pH of the solution to <2 with cold (4^{o}C) sulfuric acid (1:3) and extract once with 40 mL of diethyl ether and twice with 20-mL portions of ether. Combine the extracts and pour them through a pre-rinsed drying column containing 7 to 10 cm of acidified anhydrous sodium sulfate. Collect the dried extracts in a 500-mL Erlenmeyer flask (with a 24/40 joint) containing 10g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract for a minimum of 2 hours (See **NOTE**). Quantitatively transfer the contents of the flask to a Zymark tube when the extract is known to be dry.

NOTE: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicides recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams

of acidified sodium sulfate and again test by swirling. The 2 hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

7.2.8.4 Sample is ready for extract concentration and derivatization; proceed to **section 7.3.18**.

7.3 Shaker extraction of soil, sediment and other solid samples. **This procedure should be carried out in its entirety without delay. The sought after analytes decompose in the presence of ultraviolet light.**

7.3.1 Print backlog report for the prep and each test code (3550_HERB and 8151_TCL_S).

7.3.2 Batch samples in the LIMS and print a copy of the batch.

7.3.3 Put samples in order and check prep and test information.

7.3.4 Assemble enough appropriate glassware (Zymark tubes and funnels, 250mL Erlenmeyer flasks, and Teflon bottles) and rinse them with solvent.

7.3.5 Use red labels to write the sample information and label the glassware.

7.3.6 Pick a sample for QC (MS and MSD) and homogenize sample.

7.3.7 Weigh 30g of well mixed sample into 250mL Teflon bottle.

7.3.8 Adjust pH to < 2 with HCl. Monitor the pH for 15 minutes with occasional stirring. If necessary, add additional HCl until the pH remains at 2.

7.3.9 Spike LCS, MS and MSD with 1.0mL herbicide spike and put 1.0mL herbicide surrogate to all samples and QC samples.

7.3.10 Add 15ml Acetone, cap the bottle and tumble for 20 minutes. Add 50ml of Diethyl ether, cap the bottle and tumble for 20 minutes. Decant the acetone-ether extract and measure the volume of solvent recovered.

7.3.11 Extract the sample twice more using 15ml Acetone followed by 50 mL of Diethyl ether. After addition of each solvent, the mixture should be shaken with the shaker for 10 minutes and the acetone-ether extract decanted. After the third extraction, the volume of extract recovered should be at least 75% of the volume of added solvent (approximately 146 mL volume of added solvent). If this is not the case, then additional extractions may be necessary.

7.3.12 Combine the extracts in a 2-L separatory funnel containing 250mL of reagent water. If an emulsion forms, slowly add 5 g of acidified sodium sulfate (anhydrous) until the solvent-water mixture separates. A quantity of acidified sodium sulfate equal to the weight of the sample may be added, if necessary.

7.3.13 Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at the desired pH. Gently mix the contents of the separatory funnel for 1 minute and allow the layers to separate.

7.3.14 Collect the aqueous phase in a clean beaker and the extract phase (top layer) in a 500-mL ground glass-stoppered Erlenmeyer flask. Place the aqueous phase in the separatory funnel and re-extract using 25 mL of diethyl ether. Allow the layers to separate and discard the aqueous layer. Combine the ether extracts in a 500 mL K-D flask.

7.3.15 If sample requires additional cleanup, proceed to **section 7.3.16, followed by section 7.3.1.7, hydrolysis**. If the sample does not require additional cleanup, proceed to **7.3.17, hydrolysis**.

7.3.16 Cleanup of <u>non-hydrolyzed</u> herbicides. Use this step if additional cleanup of the non-hydrolyzed herbicides is required.

7.3.16.1 Partition the herbicides by extracting the diethyl ether from **section 7.3.14** three times with 15-mL portions of aqueous base prepared by carefully mixing 30-mL of 37% aqueous potassium hydroxide. Discard the ether phase. At this point the basic (aqueous) solution contains the herbicide salts.

7.3.16.2 Adjust the pH of the solution to <2 with cold 4^{o}C sulfuric acid (1:3) and extract once with 40 mL of diethyl ether and twice with 20-mL portions of ether. Combine the extracts and pour them through a pre-rinsed drying column containing 7-10 cm of acidified anhydrous sodium sulfate. Collect the dried extracts in a 500-mL Erlenmeyer flask (with a 24/40 joint) containing 10g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent and allow the drying agent to remain in contact with the extract for a minimum of 2 hours. See **NOTE** in **section 7.2.8.3** that emphasizes the need for a dry extract prior to esterification. Quantitatively transfer the contents of the flask to a Zymark tube when the extract is known to be dry.

7.3.16.3 Proceed to **hydrolysis** in **section 7.3.17**.

7.3.17 Hydrolysis

7.3.17.1 Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of DI water to the extract. Add boiling chips to the K-D flask. Reflux the mixture on a water bath at 60-65^{o}C until the hydrolysis step is completed (usually 1-2 hours). Remove the flasks from the water bath and cool to room temperature.

CAUTION: The presence of residual acetone will result in the formation of aldol condensation products which will cause GC interference.

7.3.17.2 Transfer the hydrolyzed aqueous solution to a 500-mL separatory funnel and extract the solution three times with 100-mL portions of methylene chloride. Discard the methylene chloride phase. At this point, the basic (aqueous) solution contains the herbicide salts.

7.3.17.3 Adjust the pH of the solution to <2 with cold (4^oC) sulfuric acid (1:3) and extract once with 40 mL of diethyl ether and twice with 20-mL portions of ether. Combine the extracts and pour them through a pre-rinsed drying column containing 7 to 10 cm of acidified anhydrous sodium sulfate. Collect the dried extracts in a 500-mL Erlenmeyer flask (with a 24/40 joint) containing 10g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract for a minimum of 2 hours (See **NOTE**). Quantitatively transfer the contents of the flask to a Zymark tube when the extract is known to be dry.

NOTE: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicides recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2 hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

7.3.17.4 Sample is ready for extract concentration and derivatization; proceed to **section 7.3.18**.

7.3.18 Transfer samples into a Zymark tube and concentrate on turbovap apparatus with settings at 35°C temperature and 4 psi pressure.

7.3.19 Concentrate the sample down to around 1.0mL, turbovap beeps when the sample is just a little less than 1.0mL. Remove the sample when turbovap beeps and add dilute the extract with 1mL of isooctane and 0.5 mL of methanol. Dilute to a final volume of 4.0 mL with diethyl ether.

7.3.20 Mix 2.0mL KOH (43.5g KOH/100 mL DI water), 1.0mL Diethyl Ether, 1.0mL Carbitol (Diethylene Glycol) and 0.3–0.5g of Diazald into a 40mL vial (Solution to use in esterification).

7.3.21 Connect one end of esterification apparatus to the esterification solution and the other end to the sample in the Zymark/culture tube. Adjust the nitrogen flow to approx.10mL/min.

7.3.22 Esterify the sample until its color turns yellow or 5-10 minutes. (Change the esterification solution every 5 samples.)

7.3.23 Let the esterification solution cool down and neutralize it with Silicic Acid. (It is extremely poisonous, do not dump it before you neutralize it.)

7.3.24 Transfer the esterified sample into appropriate 10mL vial and bring the final volume to 10.0mL with hexane.

7.3.25 Transfer about 1.0mL sample aliquot into an autosampler vial and cap the vial tightly.

7.3.26 Label the vials with appropriate sample information.

Example Label used in Extraction Laboratory

MB #9999
8151A

7.4 Preparation of aqueous samples. This procedure is applicable to TCLP extracts. Volumes of organic solvents and other reagents should be adjusted accordingly to account for the reduced volume of TCLP extracts. **This procedure should be carried out in its entirety without delay. The sought after analytes decompose in the presence of ultraviolet light.**

7.4.1 Remove the samples to be extracted from refrigerator R10 so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.4.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS. At times the wrong extraction procedure is logged into LIMS (e.g., Continuous Liquid/Liquid Extraction instead of Separatory Funnel Extraction). If this is the case, contact the section supervisor.

7.4.1.2 Contact the section supervisor. He will contact the Project Manager to determine the appropriate actions to be followed.

7.4.2 Prepare a 250-ml Erlenmeyer flask, 500 ml beaker, and a 2-liter separatory funnel for each sample in the analytical batch by rinsing with 50% sulfuric acid, followed by HPLC grade acetone, and finally two rinses with 30 ml of methylene chloride. Discard the aqueous media and solvent media after each rinse.

7.4.3 Table 7-1 indicates the type of samples that comprise an analytical batch. Note: NELAC requirements specify that the maximum number of client samples in an analytical batch can not exceed 20. Further, a batch can not be left “open” for a time period that exceeds 24 hours.

7.4.4 Label each sample and Erlenmeyer flask using the red label tape and a “Sharpie”. Red tape is used for 8151 Herbicide analysis. The information included on the label

is the sample name, batch number, test type, and sample matrix as indicated in the example below.

Example Label used in Extraction Laboratory

MB	#9999
8151A	

7.4.5 Using a graduated cylinder, transfer a 500-ml sample aliquot to a 600mL beaker.

7.4.6 Pre-clean a 1-ml syringe by rinsing at least 5 times with acetone.

7.4.7 Using the syringe, add 0.5 ml of the spike solution to the LCS, LCSD, MS, and MSD samples. Rinse the syringe with acetone as before.

7.4.8 Add 0.5 ml of the surrogate spiking solution to all of the samples.

7.4.9 Add 125 g of NaCl to the sample and mix to dissolve the salt. The addition of NaCl must be adjusted based upon the amount of sample extracted. Use 250g of NaCl for a 1000mL sample size.

7.4.10 Add 5 ml of 10 N NaOH to the sample and mix. Check the pH of the sample with pH paper. If the sample does not have a pH greater than or equal to 12, adjust the pH by adding more 10N NaOH.

7.4.11 Let the samples sit at room temperature until the hydrolysis step is complete (usually 1-2-hours). After the hydrolysis step is completed, transfer the samples into a 2 L separatory flask

7.4.12 Add 60 ml of methylene chloride to the sample bottle and rinse both the bottle and the graduated cylinder. Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure.

7.4.13 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, and centrifugation. Discard the methylene chloride phase.

7.4.14 Extract two more times by adding 60-ml volume of methylene chloride to the separatory funnel and discarding the methylene chloride layers.

7.4.15 Add 5 ml of cold (4°C) 12 N sulfuric acid to the sample (or hydrolyzed sample), seal, and shake to mix. Check the pH of the sample with pH paper. If the sample does not have a pH less than or equal to 2, adjust the pH by adding more acid.

7.4.16 Add 50-ml diethyl ether to the sample, seal, and extract the sample by vigorously shaking the funnel for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum techniques to complete the phase separation depend upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Remove the aqueous phase to the beaker and collect the ether phase in a 250-mL Erlenmeyer flask containing approximately 10 g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent.

7.4.17 Return the aqueous phase to the separatory funnel, add 25 ml of diethyl ether to the sample, and repeat the extraction procedure a second time, combining the extracts in the 250-ml Erlenmeyer flask.

7.4.18 Perform a third extraction with 25-ml diethyl ether in the same manner. Allow the extract to remain in contact with the sodium sulfate for approximately 2 hours.

> NOTE: **The drying step is very critical to ensuring complete esterification.** Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

7.4.19 Pour the dried extract into a 250mL Zymark Turbovap tube. Use a glass rod to crush any caked sodium sulfate during the transfer. Rinse the Erlenmeyer flask with 20 to 30 ml of diethyl ether to complete the quantitative transfer. Proceed to Section 7.5 for extract concentration.

7.5 Extract concentration

7.5.1 Quantitatively transfer the solvent to a clean dry Turbovap tube.

7.5.2 Place on the Turbovap under the following conditions:

7.5.2.1 Turbovap temperature set at 39°C.

7.5.2.2 Turbovap pressure set at 4 psi.

7.5.3 Concentrate the sample to approximately 0.5 mL.

7.5.4 Remove from Turbovap. Dilute extract with 1mL of isooctane and 0.5mL of methanol. Dilute to a final volume of 4mL with diethyl ether.

7.6 Esterification

7.6.1 Diazomethane derivatization

CAUTION: Diazomethane is a carcinogen and can explode under certain conditions.

The diazomethane derivatization procedures described below will react efficiently with all of the chlorinated herbicides in this method and should be used only by experienced analysts, due to the potential hazards associated with its use.

The following precautions should be taken:

1. Use a safety screen.
2. Use mechanical pipetting aides.
3. Do not heat above 90°C – EXPLOSION may result.
4. Avoid grinding surfaces, ground-glass joints, sleeve bearings, and glass stirrers – EXPLOSION may result.
5. Store away from alkali metals – EXPLOSION may result.
6. Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

7.6.2 Assemble the diazomethane bubbler.

7.6.3 Add diethyl ether to the bottle. Add 1 ml diethyl ether, 1 ml of carbitol, 2.0 mL of 37% KOH, and 0.3-0.5 g of Diazald to the VOC vial. Immediately place the exit tube into the concentrator tube containing the sample extract.

7.6.4 Apply nitrogen flow (10ml/min) to bubble diazomethane through the extract for 10 minutes or until the yellow color of diazomethane persists.

NOTE 1: It can be difficult to determine if a yellow color persists when esterifying yellow or brown colored samples. If the reaction is not complete, low results will occur.

NOTE 2: The amount of Diazald used is sufficient for esterification of approximately three sample extracts.

7.6.5 An additional 0.1-0.2 g of Diazald may be added (after the initial Diazald is consumed) to extend the generation of the diazomethane. There is sufficient KOH present in the original solution to perform a maximum of approximately 20 minutes of total esterification.

7.6.6 Remove the concentrator tube and seal it with a Neoprene or PTFE stopper. Store at room temperature in a hood for 20 minutes.

7.6.7 Destroy any unreacted diazomethane by adding 0.1-0.2 g of silicic acid to the concentrator tube. Allow to stand until the evolution of nitrogen gas has stopped.

7.6.8 Adjust the sample volume to 10.0-ml (5.0 ml for aqueous samples) with hexane and put it in a 12 mL vial. Transfer 1 mL of sample to a GC vial, and store in the refrigerator if further processing will not be performed immediately. Analyze by GC.

7.6.9 NOTE 3: Extracts should be stored at 4°C away from light. Preservation study results indicate that most analytes are stable for 28 days; however, it is recommended that the methylated extracts be analyzed immediately to minimize the trans-esterification and other potential reactions that may occur.

7.7 Gas chromatographic conditions

Set up operating conditions for the gas chromatograph using the following guidelines. These conditions may be changed as necessary to improve or maintain analytical conditions.

These chromatographic conditions should be sufficient to achieve the detection limits in the QA Manual, Section 5.

7.7.1 Preparation of Gas Chromatograph conditions using Chemstation/Enviroquant software and “Methods” function. Typically, the gas chromatograph conditions have been pre-set to conform to the method. However, due to unforeseen circumstances, it may be necessary to change these conditions. This task can be performed by following the subsequent instructions.

7.7.1.1 Open the Enviroquant software.

7.7.1.2 In the pull-down menu, click “Method”. Select the desired method.

7.7.1.3 Click “Edit Entire Method”, then “OK”. The software will prompt the user through the method for edit.

7.7.1.4 Current GC conditions for this method (NEWH.M) are as follows:

Table 7-2
Current GC Conditions for Herbicide Analysis

Condition	Setting	Condition	Setting
Sample Inlet	GC	Injection Source	GC ALS
Front Injector		*Back Injector*	
Sample Washes	2	No Parameters	
Sample Pumps	2		
Injection Volume	3.0 microliters	*Temperature Profiles*	
Syringe Size	10.0 microliters	Inlet A	250°C
On Column	Off	Inlet B	50°C
Nanoliter Adaptor	Off	Detector A	300°C
PostInj Solv. A Wash	5	Detector B	300°C
PostInj Solv. B Wash	5	Auxiliary	50°C
Viscosity Delay	1 Second		
Plunger Speed	Fast	Makeup gas	5% Methane 95% Argon
Carrier Gas	Helium 4-6 ml/min	Makeup flow rate	30-60 ml/min
Oven Parameters		*Oven Program*	
Oven Equilib Time	1 minutes	Initial Temp	60°C
Oven Max	300°C	Initial Time	1.0 Minutes
Oven State	On	Level 1	10°C/min→200°C, Hold 0 Min
Cryo State	Off	Level 2(A)	35°C/min→290°C, Hold 4 Min
Cryo Blast	Off	Level 3(B)	
Ambient	25°C	Next Run Time	21.57 Minutes

Inlet Purge	Initial Value	On Time	Off Time	Splitless Inject
A	Off	1.00	0.00	Yes
B	Off	1.00	0.00	Yes

Detector	Type	State
A	ECD	On
B	ECD	On

Signal	Source	Peak Width	Data Rate	Start	Stop
1	Det A	0.053	5.000	4.00	20
2	Det B	0.053	5.000	4.00	20

7.7.1.5 Set the output destination to screen, printer, and file.

7.7.1.6 Set the method to “autointergrate”, and not to generate a report during the run (wastes paper).

7.7.1.7 Set reference window to 10%, non-reference window to 5%, correlation window to 0.02 minutes, default multiplier to 1.00, and default sample concentration to 0.00.

7.7.2 Preparation of 'Run Log' using Chemstation/Enviroquant software.

7.7.2.1 Open Enviroquant software by "clicking" on Icon.

7.7.2.2 Select "Sequence", then "Edit sample log table".

7.7.2.3 Select sample type for each sample. Sample types include the following:

CCB: Hexane Blank
CCV: Herbicide Continuing Calibration Standard
MBLK: Method Blank for Extraction Batch
LCS: Lab Control Sample
LCSD: Lab Control Sample Duplicate
SAMP: Client Sample
MS: Matrix Spike
MSD: Matrix Spike Duplicate

7.7.2.4 Number all of the samples.

7.7.2.5 Change data file using the following format

41030501 where:

4 = GC number
Number 1= Year: '01'
0305 = Date in MM DD format
01 = sequential file number

7.7.2.6 Click "OK" when complete.

7.7.2.7 On the pull-down menu, select "Sequence", then "Save".

7.7.2.8 When the following prompt appears, change the last part to MM DD format indicating the current day.

C:\HPCHEM\4\Data\410305 where:

410305 = Instrument 4, year "01", month "03", day "05"

7.7.2.9 When complete, click "OK".

7.7.2.10 On the pull-down menu, select “Sequence”, then “Run” to start the instrument run.

7.7.3 Editing Sequences

7.7.3.1 Click “File”, then “Edit”

7.7.3.2 Sample information can be directly entered into the pop up box that appears.

7.8 Standardization and Calibration

7.8.1 Retention Time Windows:

7.8.1.1 Before establishing retention time windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

7.8.1.2 Calculate the standard deviation of the three absolute retention times for each standard.

7.8.1.3 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

7.8.1.4 In those cases where the standard deviation for a particular standard is zero, the analyst may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. The width of the window will be 0.03 minutes for the default standard deviation for narrow bore columns.

7.8.1.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed.

7.8.1.6 The absolute retention time is based on the retention times provided by the daily calibration verification standard. Each absolute retention time in the analytical method must be updated each working day at the beginning of an analytical sequence.

7.8.1.7 Simultaneous second column confirmation analysis is performed to verify the presence of a compound. The confirmation column has to be calibrated, and the retention times must be maintained on this column as per the primary column.

7.9 Initial Calibration Procedure:

7.9.1 Initial calibration is performed using multi-point calibration at a minimum of 5 concentration levels for each target component per Table 5-1.

7.9.2 If the calibration standards have already been prepared and are in storage, remove them from the refrigerator and allow them to warm to room temperature. Be sure to check for signs of precipitation in the vials. Proceed to the next step to begin calibration.

7.9.3 Place the vials in the GC autosampler tray beginning at position one. It is recommended that standards be analyzed from the highest concentration to the lowest. This will ensure that any possible active sites in the instrument will not greatly affect the analysis of the calibration curve.

7.9.4 Record the sequence of standards in the GC Instrument Run log and create this sequence in the data acquisition system.

7.9.5 For narrow bore analyze the initial calibration standards using injections of 3 µL of each calibration standard (1.5 µL split onto each column).

7.9.6 Using the data system, generate the initial calibration report indicating the calibration factors and the percent relative standard deviation for each analyte at each of the calibration levels analyzed.

7.9.7 Calculate the calibration factor for each analyte at each concentration as:

$$\mathrm{CF} = \frac{\text{Peak Area (or Height) of the compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

7.9.8 Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{\mathrm{CF}} = \frac{\sum_{i-1}^{n} CF_i}{n}$$

Where n is the number of standards analyzed.

7.9.9 Calculate the standard deviation (SD) and the RSD of the Calibration factors for each analyte as:

$$RSD = \frac{SD}{\overline{CF}} x\,100$$

$$\mathrm{SD} = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}}$$

7.9.10 If the RSD (relative standard deviation) for each analyte or averaged across all analytes is ≤20%, the initial calibration is considered linear and the mean calibration factor can be used to quantitate sample results. **For regulatory reporting in South Carolina, averaging of RSDs is not allowed and each target analyte RSD must be ≤ 20% if Average RF calibration is to be used.**

7.9.11 In those instances where the RSD for one or more analytes exceeds 20%, the initial calibration may still be acceptable if the following conditions are met **except for regulatory reporting in South Carolina as described in 7.9.10:**

7.9.11.1 The mean of the RSD values for all analytes in the calibration is less than or equal to 20%. The mean RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. If no analyte has an RSD above 20%, then the mean RSD calculation need not be performed.

7.9.11.2 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project. In other words, if the target analyte is part of the calibration standard, its RSD value is included in the evaluation.

7.9.11.3 If all of the conditions in Section 7.9.11 are met, then the average calibration or response factor may be used to determine sample concentrations.

7.9.12 If the RSD ≥20%, then linearity through the origin cannot be assumed. The analyst must use a linear regression calibration curve with Correlation Coefficient of 0.995 or greater.

7.9.13 Each initial calibration curve must be verified by analyzing a mid-level standard (ICV) containing each target analyte and prepared from a source other than that used for the ICAL. ICV must meet the criteria described in Section 7.10.4 (CCV).

7.10 Calibration Verification and Analysis of Sample Extracts:

7.10.1 Prior to analysis of any standards or samples, several GC parameters should be checked and recorded in order to ensure that operating conditions have not changed significantly from those of prior analyses.

7.10.2 The auto-sampler syringe rinse solution should be filled with Hexane and waste bottles should be emptied.

7.10.3 Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. Analysts should alternate the use of high and low concentration mixtures of calibration verification. A calibration standard must also be injected at intervals of not less than once every twenty (20) samples and at the end of the analysis (after every 10 samples is *recommended* to minimize the number of samples requiring re-injection when QC limits are exceeded).

7.10.4 The % Difference (calibration factor) or % Drift (calculated concentration) for the calibration verification standard must be within ±15% for each analyte, or averaged across all analytes before any sample analyses may take place. **For regulatory reporting in South Carolina, averaging of % Drift or % Difference is not allowed and each target analyte must meet the ±15% criteria.**

7.10.5 Calculate the % Difference as:

$$\% \text{ Difference} = \frac{\text{CF}_v - \overline{CF}}{\overline{CF}} X 100$$

Where: CFv = Calibration Factor from the CCV
CF = Mean Calibration Factor from ICAL

7.10.6 Calculate the % Drift as:

$$\% \text{ Drift} = \frac{\text{Calculated concentration - Theoretical concentration}}{\text{Theoretical concentration}} X 100$$

7.10.7 If the calibration factor or calculated concentration for an analyte or averaged across all analytes is within ±15% of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may conduct sample analysis. **For regulatory reporting in South Carolina, averaging of % Drift or % Difference is not allowed and each target analyte must meet the ±15% criteria.**

7.10.8 Remove the sample extracts from their storage location in the Organic Sample Preparation Lab (OSP) and allow them to warm to room temperature.

7.10.9 Place them in the GC autosampler with the appropriate number of calibration check solutions and hexane blanks. Analyzing the Method Blank immediately after the calibration check standard is preferable since this allows early evaluation of these QC samples and prevents any interference from possible carryover from samples previously analyzed.

7.10.10 An example sequence is shown below (See Section 7.8 for SOP on preparation of an analytical sequence).

1. CCB: Hexane blank: must be clean
2. CCV
3. MB: Method blank
4. LCS (LCSD if available)
5. Sample extracts,MS, and MSD(less than 20 samples).
6. Closing CCV (run within 12 hrs, less than 20 samples and at the end of the sequence)

7.10.11 For narrow bore columns inject 3 μL of final sample extract to the GC system. **Note**: only 1.5 μL of extract is injected into each column. Record the injection sequence in the GC instrument run logbook.

7.10.12 If any compound in any sample exceeds the analytical range that sample must be diluted.

7.10.13 Sample injection may continue for as long as the calibration verification standards and blanks continue to meet instrument QC requirements.

7.10.14 Proceed to next sections for qualitative and quantitative analysis.

7.11 Data interpretation

7.11.1 The qualitative identification of single compounds determined by this method is based on retention time.

7.11.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window.

7.11.3 Validation of gas chromatographic system qualitative performance: use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

7.11.4 When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed.

7.12 Quantitative analysis

7.12.1 When linearity exists, calculate the concentration of each identified analyte in the sample as follows:

7.12.1.1 Aqueous Sample:

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

Where:

A_s = Area of the peak for the analyte in the sample.
V_i = Volume of extract injected, µl.
CF = Mean calibration factor for the compound being measured, (area/ng).
V_s = Volume of sample extracted (ml).
V_t = Total volume of the concentrated extract (µl).
D = Dilution Factor.

7.12.1.2 Nonaqueous samples:

$$\text{Concentration } (\mu g/Kg) = \frac{(A_s)(V_t)(D)}{(\overline{CF})(V_i)(W_s)}$$

Where A_s, Vt, D, $\overline{CF}$ and Vi are as described in 7.13.1.1 and
Ws = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data.

7.12.2 When concentrations on both columns have been calculated, the agreement between the quantitative results should be evaluated after the identification has been confirmed.

7.12.3 If the relative percent difference (RPD) of the results exceeds 40%, check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. Carefully examine peak shapes as compared to the closest CCV (Overlays should be used when necessary).

7.12.3.1 Calculate the relative percent difference (RPD) as:

$$RPD = \frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} X 100$$

7.12.4 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result, and qualify the result with “NC” (not confirmed) at QA review and narrate the affected workorder. This approach is conservative relative to the protection of the environment.

Table 7-3
3510_HERB (Prep Code)

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3510_HERB AND 8151_TCL_W/615_W/1311_H)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH SOLVENT

____ : USE RED LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : TRANSFER 500 ml OF SAMPLE AND 500 ml DI WATER (FOR MB AND LCS) INTO GLASS BEAKERS **(USE 50 ml FOR TCLP PREP/1311_H)**

____ : ADD 125 g OF NaCl (ADJUST THE AMOUNT OF NaCl USED BASED UPON THE SAMPLE VOLUME EXTRACTED. USE 250g OF NaCl FOR A 1 L SAMPLE.)

____ : SPIKE LCS, MS AND MSD WITH 0.5 ml HERBICIDE SPIKE AND ADD 0.5 ml HERBICIDE SURROGATE TO EACH SAMPLE AND QC SAMPLE

____ : ADJUST pH TO > 12 WITH NaOH SOLUTION (40 g NaOH/100 ml DI WATER) AND LET THE SAMPLES STAY FOR ABOUT 2 HOURS

____ : TRANSFER SAMPLES TO SEPARATORY FUNNELS

____ : EXTRACT 3 TIMES WITH 60 ML METHYLENE CHLORIDE AND DISCARD THE EXTRACT

____ : ADJUST pH TO < 2 WITH H2SO4

____ : EXTRACT WITH 50 ml DIETHYL ETHER

____ : COLLECT THE ETHER PORTION INTO A 250 ml ERLENMEYER FLASK THAT HAS SOME SODIUM SULFATE IN IT. RECYCLE THE SAMPLE TO THE SEP FUNNEL AND REPEAT THE EXTRACTION 2 MORE TIMES WITH 50 ml ETHER EACH (ETHER PORTION WILL BE FLOATING ON TOP OF THE SAMPLE)

____ : LET THE EXTRACTS STAY ON SODIUM SULFATE FOR ABOUT 2 HOURS

____ : TRANSFER SAMPLES INTO A ZYMARK TUBE AND CONCENTRATE ON TURBOVAP APPARATUS WITH SETTINGS AT 35°C TEMPERATURE AND 4 psi PRESSURE

____ : CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP BEEPS AND DILUTE EXTRACT WITH 1mL OF ISOOCTANE AND 0.5mL OF METHANOL. DILUTE TO A FINAL VOLUME OF 4mL DIETHYL ETHER

____ : MIX 2.0 ml KOH, 1.0 ml DIETHYL ETHER, 1.0 ml CARBITOL (DIETHYLENE GLYCOL) AND 0.3–0.5 g OF DIAZALD INTO A 40 ml VIAL (SOLUTION TO USE IN ESTERIFICATION)

____ : CONNECT ONE END OF ESTERIFICATION APPARATUS TO THE ESTERIFICATION SOLUTION AND THE OTHER END TO THE SAMPLE IN THE ZYMARK TUBE. ADJUST THE NITROGEN FLOW TO 10 ml/min

____ : ESTERIFY THE SAMPLE TILL ITS COLOR TURNS INTO YELLOW OR 5-10 MINUTES (CHANGE THE ESTERIFICATION SOLUTION EVERY 5 SAMPLES)

____ : LET THE ESTERIFICATION SOLUTION COOL DOWN AND NEUTRALIZE IT WITH SILICIC ACID (IT IS EXTREMELY POISIONOUS, DO NOT DUMP IT BEFORE YOU NEUTRALIZE IT)

____ : TRANSFER THE ESTERIFIED SAMPLE INTO A 10 ml VIAL AND BRING THE FINAL VOLUME TO 5.0 ml WITH HEXANE

____ : TRANSFER ABOUT 1.0 ml SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-4
3550_HERB (Prep Code)

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3550_HERB AND 8151_TCL_S)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH SOLVENT

____ : USE RED LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : WEIGH 30 g OF SAMPLE AS RECEIVED INTO 250 ml TEFLON BOTTLES

____ : ADJUST pH TO < 2 WITH 2-3 DROPS OF HCl. MONITOR THE pH FOR 15 MINUTES WITH OCCASIONAL STIRRING. IF NECESSARY, ADD ADDITIONAL HCl UNTIL pH REMAINS AT 2.

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml HERBICIDE SPIKE AND PUT 1.0 ml HERBICIDE SURROGATE TO ALL SAMPLES AND QC SAMPLES

____ : ADD 15ML ACETONE, CAP THE BOTTLES TIGHTLY AND TUMBLE FOR 20 MINUTES.

____ : ADD 50ML OF DIETHYL ETHER, CAP THE BOTTLES TIGHTLY AND TUMBLE FOR 20 MINUTES. DECANT THE ACETONE-ETHER EXTRACT AND MEASURE THE VOLUME OF SOLVENT RECOVERED.

____ : EXTRACT THE SAMPLE **TWICE** MORE USING 15ML ACETONE AND TUMBLE FOR 10 MINUTES FOLLOWED BY 50ML DIETHYL ETHER AND TUMBLE FOR 10 MINUTES AND DECANT THE ACETONE-ETHER EXTRACT INTO CLEAN 250 ML ELEMEYER FLASKS.

____ : REPEAT THE ABOVE STEP ONE MORE TIME. AFTER THE THIRD EXTRACTION, THE VOLUME OF EXTRACT RECOVERED SHOULD BE AT LEAST 75% OF THEVOLUME OF ADDED SOLVENT (APPROXIMATELY 146ML VOLUME OF SOLVENT ADDED). IF THIS IS NOT THE CASE, PERFORM ADDITIONAL EXTRACTIONS MAY BE NECESSARY.

____ : COMBINE THE EXTRACTS IN A 2-L SEPARATORY FUNNEL CONTAINING 250ML REAGENT WATER. IF AN EMULSION FOMS, SLOWLY ADD 5G OF ACIDIFIED SODIUM SULFATE (ANHYDROUS) UNTIL THE SOLVENT-WATER MIXTURE SEPARATES. A QUANTITY OF ACIDIFIED SODIUM SULFATE EQUAL TO THE WEIGHT OF THE SAMPLE MAY BE ADDED, IF NECESSARY.

____ : CHECK THE pH OF THE EXTRACT. IF IT IS NOT AT OR BELOW pH 2, ADD MORE CONCENTRATED HCl UNTIL STABILIZED AT THE DESIRED pH. GENTLY MIX THE CONTENTS OF THE SEPARATORY FUNNEL FOR 1 MINUTE AND ALLOW THE LAYERS TO SEPARATE.

____ : COLLECT THE AQUEOUS PHASE IN A CLEAN BEAKER AND THE EXTRACT PHASE (TOP LAYER) IN A 500ML GROUND GLASS-STOPPERED ERLENMEYER FLASK. PLACE THE AQUEOUS PHASE IN THE SEPARATORY FUNNEL AND RE-ESTRACT USING 25ML OF DIETHYL ETHER. COMBINE THE ETHER EXTRACTS IN A 500ML K-D FLASK.

***IF REQUIRES ADDITIONAL CLEANUP, PROCEED TO CLEANUP FOLLOWED BY HYDROLYSIS AND ESTERIFICATION.**

***IF THE SAMPLE DOES <u>NOT</u> REQUIRE ADDITIONAL CLEANUP, THEN PROCEED TO HYDROLYSIS FOLLOWED BY ESTERIFICATION.**

CLEANUP OF NON-HYDROLYZED HERBICIDES

____ :EXTRACT THE DIETHYL ETHER EXTRACT (SAMPLE) THREE TIMES WITH 15mL PORTIONS OF AQUEOUS BASE PREPARED BY CAREFULLY MIXING 30mL OF 37% AQUEOUS POTASSIUM HYDROXIDE. DISCARD THE ETHER PHASE

____ :ADJUST THE PH OF THE SOLUTION TO <2 WITH COLD (4 DEGREE C) H2SO4 (1:3) AND EXTRACT ONCE WITH 40 mL OF DIETHYL ETHER AND TWICE WITH 20mL PORTIONS OF DIETHYL ETHER

____ :COMBINE THE EXTRACTS AND POUR THEM THROUGH A PRE-RINSED DRYING COLUMN CONTAINING 7-10 CM OF ACIDIFIED ANJYDROUS SODIUM SULFATE

____ :COLLECT THE DRIED EXTRACTS IN A 500 mL ERLENMEYER FLASK CONTAINING 10G OF ACIDIFIED ANHYDROUS SODIUM SULFATE. PERIODICALLY, VIGOROUSLY SHAKE THE EXTRACT FOR A MINIMUM OF 2 HOURS. QUANTITATIVELY TRANSFER THE CONTENTS OF THE FLASK TO A ZYMARK TUBE WHEN THE EXTRACT IS KNOW TO BE DRY

HYDROLYSIS

____ : TRANSFER EXTRACT TO A 250mL ERLENMEYER FLASK WITH A GROUND-GLASS JOING AT THE NECK

____ :ADD 5mL of 37% AQUEOUS POTASSIUM HYDROXIDE AND 30 mL OF WATER TO THE EXTRACT. TRANSFER SAMPLE TO 500mL KD FLASK. ADD BOILING CHIPS TO FLASK. REFLUX THE MIXTURE ON A WATER BATH AT 60-65 DEGREES C UNTIL HYDROLYSIS STEP IS COMPLETED (1-2 HOURS). REMOVE THE FLASKS FROM THE WATER BATH AND COOL TO ROOM TEMPERATURE

____ :TRANSFER THE HYDROLYZED AQUEOUS SOLUTION TO A 500mL SEPARATORY FUNNEL AND EXTRACT THE SOLUTION THREE TIMES WITH 100mL PORTIONS OF METHYLENE CHLORIDE. DISCARD THE METHYLENE CHLORIDE PHASE

____ :ADJUST THE PH OF THE SOLUTION TO <2 WITH COLD (4 DEGREE C) H2SO4 (1:3) AND EXTRACT ONCE WITH 40 mL OF DIETHYL ETHER AND TWICE WITH 20mL PORTIONS OF DIETHYL ETHER.

____ :COMBINE THE EXTRACTS AND POUR TEM THROUGH A PRE-RINSED DRYING COLUMN CONTAINING 7-10 CM OF ACIDIFIED ANJYDROUS SODIUM SULFATE

____ :COLLECT THE DRIED EXTRACTS IN A 500 mL ERLENMEYER FLASK CONTAINING 10G OF ACIDIFIED ANHYDROUS SODIUM SULFATE. PERIODICALLY, VIGOROUSLY SHAKE THE EXTRACT FOR A MINIMUM OF 2 HOURS. QUANTITATIVELY TRANSFER THE CONTENTS OF THE FLASK TO A ZYMARK TUBE WHEN THE EXTRACT IS KNOW TO BE DRY AND PROCEED WITH ESTERIFICATION

ESTERIFICATION

____ : TRANSFER SAMPLES INTO A ZYMARK TUBE AND CONCENTRATE ON TURBOVAP APPARATUS WITH SETTINGS AT 35°C TEMPERATURE AND 4 psi PRESSURE

____ : CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP BEEPS AND DILUTE EXTRACT WITH 1mL OF ISOOCTANE AND 0.5mL OF METHANOL. DILUTE TO A FINAL VOLUME OF 4mL DIETHYL ETHER

____ : MIX 2.0 ml KOH (43.5 g KOH/100 ml DI WATER) , 1.0 ml DIETHYL ETHER , 1.0 ml CARBITOL (DIETHYLENE GLYCOL) AND 0.3–0.5 g OF DIAZALD INTO A 40 ml VIAL (SOLUTION TO USE IN ESTERIFICATION)

____ : CONNECT ONE END OF ESTERIFICATION APPARATUS TO THE ESTERIFICATION SOLUTION AND THE OTHER END TO THE SAMPLE IN THE ZYMARK TUBE. ADJUST THE NITROGEN FLOW TO 10 ml/min

____ : ESTERIFY THE SAMPLE TILL ITS COLOR TURNS INTO YELLOW OR 5-10 MINUTES (CHANGE THE ESTERIFICATION SOLUTION EVERY 5 SAMPLES)

____ : LET THE ESTERIFICATION SOLUTION COOL DOWN AND NEUTRALIZE IT WITH SILICIC ACID (IT IS EXTREMELY POISIONOUS, DO NOT DUMP IT BEFORE YOU NEUTRALIZE IT)

____ : TRANSFER THE ESTERIFIED SAMPLE INTO A 10 ml VIAL AND BRING THE FINAL VOLUME TO 10.0 ml WITH HEXANE

____ : TRANSFER ABOUT 1.0 ml SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-5
3580A_HERB (Prep Code)

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3580A_HERB AND 8151_TCL_X)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH SOLVENT

____ : USE RED LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : WEIGH 1 g OF SAMPLE INTO LABELED BOROSILICATE CULTURE TUBES

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml HERBICIDE SPIKE AND PUT 1.0 ml HERBICIDE SURROGATE TO ALL SAMPLES AND QC SAMPLES

____ : ADJUST pH TO < 2 WITH HCl

____ : ADD APPROX. 5 ML DIETHYL ETHER AND MIX THOROUGHLY. DEWATER BY PASSING THROUGH $NASO_4$ IF NECESSARY.

____ : CONCENTRATE TO 1.0 mL in ZYMARK.

HYDROLYSIS

____ : TRANSFER 1.0mL TO A 250mL ERLENMEYER FLASK WITH A GROUND-GLASS JOING AT THE NECK. (IF A DILUTION OF THE EXTRACT IS NEEDED DUE TO HIGH CONCENTRATIONS OF HERBICIDES, SEE DEPARTMENT SUPERVISOR OR TECHNICAL DIRECTOR BEFORE PROCEEDING

____ :ADD 5mL of 37% AQUEOUS POTASSIUM HYDROXIDE AND 30 mL OF WATER TO THE EXTRACT. TRANSFER SAMPLE TO 500mL KD FLASK. ADD BOILING CHIPS TO FLASK. REFLUX THE MIXTURE ON A WATER BATH AT 60-65 DEGREES C UNTIL HYDROLYSIS STEP IS COMPLETED (1-2 HOURS). REMOVE THE FLASKS FROM THE WATER BATH AND COOL TO ROOM TEMPERATURE

____ :TRANSFER THE HYDROLYZED AQUEOUS SOLUTION TO A 500mL SEPARATORY FUNNEL AND EXTRACT THE SOLUTION THREE TIMES WITH 100mL PORTIONS OF METHYLENE CHLORIDE. DISCARD THE METHYLENE CHLORIDE PHASE

____ :ADJUST THE PH OF THE SOLUTION TO <2 WITH COLD (4 DEGREE C) H2SO4 (1:3) AND EXTRACT ONCE WITH 40 mL OF DIETHYL ETHER AND TWICE WITH 20mL PORTIONS OF DIETHYL ETHER

____ :COMBINE THE EXTRACTS AND POUR THEM THROUGH A PRE-RINSED DRYING COLUMN CONTAINING 7-10 CM OF ACIDIFIED ANJYDROUS SODIUM SULFATE

____ :COLLECT THE DRIED EXTRACTS IN A 500 mL ERLENMEYER FLASK CONTAINING 10G OF ACIDIFIED ANHYDROUS SODIUM SULFATE. PERIODICALLY, VIGOROUSLY SHAKE THE EXTRACT FOR A MINIMUM OF 2 HOURS. QUANTITATIVELY TRANSFER THE CONTENTS OF THE FLASK TO A ZYMARK TUBE WHEN THE EXTRACT IS KNOW TO BE DRY AND PROCEED WITH ESTERIFICATION

ESTERIFICATION

____ : TRANSFER SAMPLES INTO A ZYMARK TUBE AND CONCENTRATE ON TURBOVAP APPARATUS WITH SETTINGS AT 35°C TEMPERATURE AND 4 psi PRESSURE

____ : CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP

BEEPS AND DILUTE EXTRACT WITH 1mL OF ISOOCTANE AND 0.5mL OF METHANOL. DILUTE TO A FINAL VOLUME OF 4mL DIETHYL ETHER

____ : MIX 2.0 ml KOH (43.5 g KOH/100 ml DI WATER), 1.0 ml DIETHYL ETHER, 1.0 ml CARBITOL (DIETHYLENE GLYCOL) AND 0.3–0.5 g OF DIAZALD INTO A 40 ml VIAL (SOLUTION TO USE IN ESTERIFICATION)

____ : CONNECT ONE END OF ESTERIFICATION APPARATUS TO THE ESTERIFICATION SOLUTION AND THE OTHER END TO THE SAMPLE IN THE ZYMARK/CULTURE TUBE. ADJUST THE NITROGEN FLOW TO APPROX. 10 ml/min

____ : ESTERIFY THE SAMPLE TILL ITS COLOR TURNS INTO YELLOW OR 5-10 MINUTES (CHANGE THE ESTERIFICATION SOLUTION EVERY 5 SAMPLES)

____ : LET THE ESTERIFICATION SOLUTION COOL DOWN AND NEUTRALIZE IT WITH SILICIC ACID (IT IS EXTREMELY POISIONOUS, DO NOT DUMP IT BEFORE YOU NEUTRALIZE IT)

____ : TRANSFER THE ESTERIFIED SAMPLE INTO A 10 ml VIAL AND BRING THE FINAL VOLUME TO 10.0 ml WITH HEXANE

____ : TRANSFER ABOUT 1.0 ml SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-6

AES
GC 8151 QC DATA CHECKLIST

Batch ID: _____ Matrix: _____

Data Package

- Is there a valid calibration curve for the test method (check date on chromatograms against list in QA folder)? **Yes**
- Does the package include an instrument run log? **Yes**
- Does the package include a copy of the extraction log (hand written) **Yes**
- Does the package include a copy of the Prep backlog? **Yes**
- Does the package include a copy of the prep log (LIMS)? **Yes**
- Does the package include all the chromatograms in the run log? **Yes**
- Are all chromatograms properly labeled with sample name, dilutions, analyst name, instrument name? **Yes**
- Does the sample IDs in LIMS match the prep log? Run Log? **Yes**
- Are the correct test IDs in LIMS? In the chromatograms? **Yes**
- Is the correct prep date entered in LIMS? **Yes**
- Does the CCV contain all analytes that are being reported? **Yes**
- Is there an opening and closing CCV, or one every 12 hours? **Yes**
- Does the package include MB, LCS, LCSD, MS, and MSD for each extraction batch? **Yes**
- Are there any flags in LIMS that are not resolved with a NCR? **Yes**
- Were samples analyzed (reported) within holding time? If not, needs NCR **Yes**
- Are the analytes properly turned on or off? **Yes**
- Is the proper blank reference indicated? **Yes**
- Is the proper spike reference indicated? **Yes**
- Are any dilution factors accounted for in LIMS? **Yes**
- Is highest result reported for each sample? **Yes**

Analyst: Date: _____ Time: _____

Primary Reviewer: Date: _____ Time: _____

Secondary Reviewer: _____ Date: _____ Time: _____

Comments:

DILUTIONS

Sample ID	Semi-Volatile(s) Needed	Done
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes

REANALYSIS

Sample ID	Semi-Volatile(s) Needed	Done
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes
		Yes

Table 7-7

Calibration Curve Review Checklist for GC Methods

Matrix: _____ Instrument: _____ Date Prepared: _____

Test Method: _____ Curve ID: _____

	Col 1	Col 2
• Does the calibration curve contain a minimum of 5 points.	☐	☐
• If average response factor used in calculation are all compound RSD ±20% for 8000 Methods and FL PRO or 10% for 600 Methods	☐	☐
• If linear regression used in calculation, is the correlation coefficient (r) > 0.995	☐	☐
• Is the lowest data point in each curve at the PQL in LIMS	☐	☐
List any analytes with data point below or above PQL		
• Is the highest data point in each curve at the UQL in LIMS	☐	☐
List any analytes with highest standard below UQL		
• Is the retention time RT of each target analyte in each calibration standard within the RT window.	☐	☐
Comments:		
• Does the ICV pass (% D < = 15% for each analyte or average of all %Ds ≤15% for 8000 Methods; ≤20% for FL PRO; % D ≤15% for each target for 600s)	☐	☐
• Is ICV a second source standard from ICAL standards	☐	☐
• Is the standard preparation information included and properly signed and dated.	☐	☐
• Is run log included and properly signed and dated.	☐	☐
• Are all chromatograms included and properly signed and dated.	☐	☐

Analyst: _____ Date: _____ Time: _____

Primary Reviewer: _____ Date: _____ Time: _____

Secondary Reviewer: _____ Date: _____ Time: _____

Comments:

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data generated. Detailed quality assurance procedures can be found in SOP #QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Calibration of the GC. This is accomplished through a 5-point calibration curve. The points on the curve must meet a 20% RSD when comparing calibration factors to determine if the calibration curve is linear. If RF RSD is >20%, linear regression calibration must be used with Corr. Coeficient $\geq$0.995. Initial verification of the curve is also performed using a second source standard. The calibration verification must also be within ±15% for each analyte, or averaged across all analytes before any sample analyses may take place. **For regulatory reporting in South Carolina, averaging of % Drift or % Difference is not allowed and each target analyte must meet the ±15% criteria.**

8.1.3 Calibration Curve Verification (CCV). Each day at the beginning of the sequence, a CCV must be performed. Then every 12hrs or 20 samples, a CCV need to be performed. The control limit is 15% for each compound or average 15% for all compounds. **For regulatory reporting in South Carolina, averaging of % Drift or % Difference is not allowed and each target analyte must meet the ±15% criteria.**

8.1.4 Retention time window. The retention time for each analyte is compared over a 72-hour time period and the average retention time calculated.

8.1.5 Method Detection Limit Study. The method detection limit is calculated by analyzing at least seven replicates prepared in blank water at concentrations approximately equal to the PQL for each target analyte. Each calculated MDL must be <PQL. MDL's are to be performed annually or whenever instrument conditions have changes that will affect the established detection limits.

8.1.6 Method blank. Reagent blank analyses must be performed at the following frequency: Every twenty (20) samples of similar concentration and/or sample matrix or whenever samples are extracted and analyzed by the same procedure at same time, whichever is more frequent. The concentration of the method blank of any analyte of interest should not exceed the laboratory established practical quantitation limit (PQL). Target analytes detected in Method blanks at levels >PQL must be handled in accordance with Section 8.2.

8.1.7 Surrogate Recovery. All samples, blanks, and QC samples are fortified with surrogate spiking compound before extraction and injection in order to monitor sample extraction efficiency. The recovery of the surrogate compound must be within the recovery limits established by the laboratory. Recovery outside control limits must be handled in accordance with Section 8.2.

8.1.8 Laboratory Control Sample (LCS). The Laboratory Control Sample (LCS) is used to monitor, assess and document laboratory method performance and is performed on every batch or twenty samples, whichever occurs more frequently. The recovery of the analytes must meet established laboratory guidelines. Recovery outside control limits must be handled in accordance with Section 8.2. For SC aqueous samples, recovery ranges for compounds are 70 -130%; Dalapon range is 23.7 - 110%. For SC soil samples, recovery ranges for compounds are 70 -130%; Dinoseb range is 20 -110%.

8.1.9 Sample spike and duplicate spike. Matrix spikes and matrix spike duplicates are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples. The recovery of the analytes must meet established laboratory guidelines. Recovery outside control limits must be handled in accordance with Section 8.2.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP# QA-01000, “Quality Assurance Manual,” in Section 5. Included are tables that detail corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance, and procedure deviations in appropriate logbooks.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 The toxicity and/or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, analysts by wearing gloves, lab coats, and protective eyeglasses must reduce exposure to these chemicals to the lowest level possible. Analysts are responsible for being familiar with regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis.

9.2 The analyst shall observe all safety precautions provided in the laboratory safety program.

9.3 Primary and secondary standards of these toxic compounds must be handled and prepared in a hood.

9.4 The ECD detectors on the gas chromatographs must be vented to prevent airborne contamination.

9.5 Charcoal traps may be installed on the split vent of all instruments with split/splitless injectors to prevent airborne contamination of the work environment.

9.6 Proper disposal of all wastes is essential. Containers are provided for all waste according to type. Section 17 of the Quality Assurance Manual discusses the disposal of various laboratory wastes in greater detail. Also, see Section 14.0 Pollution Management of this SOP.

10.0 DATA REPORTING

10.1 The LIMS system automatically rounds the data based upon factors set up for each test category. Typically, the LIMS reports to two significant figures.

10.2 The reporting limits can be changed in LIMS. Various laboratory personnel including project managers have the rights to change the limits per the requirements of the clients. Reporting limits are based upon the MDLs developed for each test.

10.3 Calculation of results using Enviroquant software.

10.3.1 Open Enviroquant software by “clicking” on Icon.

10.3.2 On the pull-down menu, click on “Data Analysis”.

10.3.3 On the pull-down menu, click on “Load”, then “Data file”. The path C:\hpchem\2\data should appear.

10.3.4 Using the “arrow” key, scroll down to the desired sequence number. The sequence number represents the GC number, year, month and day in a format “410321”. Note that the left side of the box contains all of the samples in the run. Each sample can be selected by positioning the mouse pointer over the sample and double clicking it.

10.3.5 Once a sample has been selected, on the pull-down menu, click on “Load”, then “Method”. Select the method 8151MA15.M, where MA = the month, March and 15 = the date. From this point, the method will remain the same for each sample selection.

10.3.6 To calculate a result, on the pull-down menu, click on “Quant”, then “Calculate and generate report”. An alternative method is to click on “Quant”, then “Int” and “Integrate”.

10.3.7 Review the Enviroquant calculated result by clicking on "Quant", then "Q edit". To enlarge the various areas of the chromatogram, place the pointer on the chromatogram and right click the mouse. Drag the mouse over the area to enlarge. Double click the chromatogram to return it to its original size.

10.3.8 Once the chromatogram has been enlarged, the baselines can be redrawn by placing the mouse at one end of the peak and moving it across the bottom while holding down the right mouse. Release the mouse when the line is the correct length.

10.3.9 A general rule for drawing the baseline is that it should be started from the lowest side of the peak and go straight across so that the baseline makes a "right angle" at the raised side of the peak.

10.3.10 Perform the same procedure on the rest of the chromatograms.

10.4 Completed data is stored in the "C" drive of the acquisition computer under the following directory: "C:\HPCHEM\410305" where 4 is the instrument number, 1 is the year "01", 0305 is the date in MM DD format.

10.4.1 Prior to moving files for final storage, open the completed data folder and change the status of the data to "frozen" using the following procedure.

10.4.1.1 In Enviroquant, click "Tools", then "Change Data State"

10.4.1.2 Click the radio button next to "Frozen"

10.4.1.3 Click "OK". Exit to save the changes

10.4.1.4 Using NT Explorer, Find the file on the "C' drive in the directory "C:\HPChem\1 where 1 = GC 3 and 2 = GC 4

10.4.1.5 Highlight the file and click "Cut".

10.4.1.6 Using NT Explorer, find and select the folder in the computer called "Storage".

10.4.1.7 Locate the proper directory, either GC 2 or GC 4, and click "Save" to save the file to this directory.

10.4.1.8 Periodically, these files are written to a writeable "CD" and stored off site by the VP of Technical Operations.

10.5 Current MDLs for all parameters may be found in tables in Section 5 of SOP# QA-01000, "Quality Assurance Manual".

11.0 FILE MAINTENANCE

11.1 All data are stored on the Portal Server

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto portable hard drives. Two copies are made. One copy is stored on the laboratory premises, while the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks must be completed any time maintenance is performed on the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as replacement of motors in tower autosamplers.

12.2 Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.3 Routine maintenance: Typical routine maintenance consists of keeping the system clean and insuring that chromatography remains acceptable. Examples would be peak tailing and degradation of DDT and Dieldrin in pesticide analysis.

The table below indicates the frequency of routine maintenance for various instruments types within the laboratory.

Maintenance Action	Recommended Frequency
Changing injection port liners	Weekly or when chromatography is affected
Trimming column	Monthly or when chromatography is affected
Cleaning GC/MS source	Weekly or when chromatography is affected
Changing GC/HPLC Column	Annually or when other attempts to resolve chromatography fail

12.4 Non-routine maintenance includes routine maintenance performed when chromatography has been negatively affected as well as other exceptional maintenance to improve chromatography.

12.4.1 Metal (GC) Injector body maintenance (**when poor chromatography is observed**).

12.4.1.1 Turn off the oven and remove the analytical column after the oven has cooled. Remove the glass injection port insert. Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

12.4.1.2 Place a beaker beneath the injector port inside the GC oven. Using a wash bottle, rinse the entire inside of the injector port with acetone and then with toluene, catching the rinsates in the beaker.

12.4.1.3 Prepare a solution of deactivating agent (dichlorodimethylsilane) following the manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivating solution, serially rinse the injector body with toluene, methanol, acetone, and hexane. Reassemble the injector and replace the GC column.

12.5 Peripherals maintenance. This usually includes problems such as the inability to save files to a disk or operational activities such as an autosampler that will not function. If these types of problems occur, contact the department manager for assistance.

13.0 METHOD PERFORMANCE (Per SW-846 Method 8151)

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The reporting limit RL is defined as the concentration of a substance that is above the level of uncertainty. The concentration listed in the table in Section VIII was obtained using reagent water. Similar results can be achieved using representative wastewater. The MDL actually achieved is a given analysis will vary depending on instrument sensitivity and matrix effects.

13.2 This method is recommended for use in the concentration range from the MDL to 1000 x MDL. Direct injection techniques should be used to measure concentration levels above 1000 x MDL.

13.3 General laboratory precision and accuracy are shown in Table 13-1 and 13-2.

Table 13-1
ACCURACY AND PRECISION FOR DIAZOMETHANE DERIVATIZATION ORGANIC-FREE REAGENT WATER MATRIX

Compound	Spike Conc. (µg/L)	Mean[a] %Recovery	Standard Dev. %R
2,4-D	1	131	27.5
2,4-DB	4	87	13.1
2,4,5-TP (Silvex)	0.4	117	16.4
2,4,5-T	0.2	134	30.8
Dalapon	10	100	20.0
Dicamba	0.4	135	32.4
Dichloroprop	2	107	20.3
Dinoseb	0.4	42	14.3
Acifluorfen	0.2	121	15.7
Bentazon	1	120	16.8
Chloramben	0.4	111	14.4
DCPA diacid[b]	0.2	74	9.7
3,5-Dichlorobenzoic acid	0.6	102	16.3
5-Hydroxydicamba	0.2	103	16.5
4-Nitrophenol	1	131	23.6
Pentachlorophenol	0.04	130	31.2
Picloram	0.6	91	15.5

[a] Mean percent recovery calculated from 7-8 determinations of spiked organic-free reagent water.

DCPA monoacid and diacid metabolites included in method scope; DCPA diacid metabolite used for validation studies. DCPA is a dimethyl ester.

Table 13-2
ACCURACY AND PRECISION FOR DIAZOMETHANE DERIVATIZATION CLAY MATRIX

Compound	Mean %R[a]	Linear conc. Range[b] (µg/Kg)	%RSD[c] (n=20)
2,4-D	84.3	1.2-2,440	5.3
2,4-DB	90.7	4.0-8,060	7.6
2,4,5-TP (Silvex)	94.5	0.42-828	5.7
2,4,5-T	83.1	0.42-828	7.3
Dicamba	95.7	0.52-104	7.5
Dichloroprop	97.3	1.5-3,000	5.0
Dinoseb	93.7	0.82-1,620	8.7
MCPP	98.3	620-61,800	3.4
MCPA	96.9	620-61,200	5.3

[a] Mean percent recovery calculated from 10 determinations of spiked clay and clay/sill bottom samples over the linear concentration range.

[b] Linear concentration range was determined using standard solutions and corrected to 50 g solid samples.

[c] Percent relative standard deviation was calculated using standard solutions, 10 samples high in the linear concentration range, and 10 samples low in the range.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analyses generate waste. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 Section 17 of the QA Manual further discusses methods for the disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade – A dry chemical that has been dried at 250°C for 4 hours, cooled, and stored in a desiccator.

15.2 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to distilled water or clean soil (sand) and the concentration is measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.3 DI Water - Deionized water

15.4 RSD – Relative Standard Deviation

15.5 RF – Response Factor. Determined as the concentration of a sample divided by the chromatographic area of the peak produced by the sample

15.6 MS - Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured concentration of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.7 MSD - Matrix Spike Duplicate.

15.8 CCV - Continuing Calibration Verification Standard. Must be varied thoughout the daily runs, that is, the concentrations must be at low, middle, and upper end of the calibration curve.

15.9 ICV – Initial Calibration Verification Standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot that the calibration standard.

15.10 LCSD - Laboratory Control Sample Duplicate

16.0 REFERENCES

16.1 "Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization" USEPA SW-846 Method 8151A, Revision 2, December 1996.

16.2 "Determinative Chromatographic Separations", USEPA SW-846 Method 8000B, Revision 1, December 1996.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://portal/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://portal/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

18.0 SOP Revision History

Revision Date	Revision #	Summary of and Reason for Changes/Updates	Responsible for Revision
5/2/2005	6	Update	Greg Jones
4/10/2009	7	Update	Dana Till
9/16/2010	8	SC Update Sections 7.2.6, 7.4.12, 7.4.14, Table 7-3, Table 7-4, and Table 7-5.	Dana Till
6/29/2011	9 DRAFT	SC Update Sections 2.0, 5.9.6, Table 5-2, 7.2.8, 7.3.15, 7.3.16, 7.3.17, 7.3.19, 7.4.9, 7.5.4, Table 7-3, Table 7-4, and Table 7-5.	Dana Till
7/1/2011	10	SC Update Sections 2.0, 5.9.6, Table 5-2, 7.2.6, 7.2.8, 7.3.15, 7.3.16, 7.3.17, 7.3.19, 7.4.9, 7.5.4, Table 7-3, Table 7-4, and Table 7-5.	Dana Till
11/11/2011	11	Update to Sections 5.12.2, 5.12.4, Table 5-2, 7.4.19, and addition of Section 18.0.	Dana Till
2/13/2012	12	SC Audit: Updates to Sections 5.16, 7.3.8 through 7.3.14, 7.4.19, 8.1.8, Table 7-4 and addition of Section 18.0.	Dana Till

AES, Inc.
3785 Presidential Pkwy
Atlanta, GA 30340

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DOCUMENT CONTROL NUMBER: Rev. 9

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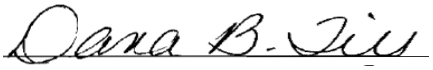
Technical Director:  Date: 6/18/2011

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Department Supervisor:  Date: 6/18/2011

STANDARD OPERATING PROCEDURE FOR
VOLATILE ORGANIC COMPOUNDS BY EPA SW-846 METHOD 8260B/5030/5035

TABLE OF CONTENTS

Page

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to nearly all types of samples, regardless of water content, including ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

1.2 The reporting limit (RL or PQL) of Method 8260 for an individual compound is instrument dependent and, also, dependent upon the choice of sample preparation/introduction method. Using standard quadrapole instrumentation and the purge and trap technique, limits should be approximately 5 µg/Kg (wet weight) for soil/sediment samples, 0.5 mg/Kg (wet weight) for wastes, and 5 µg/L for ground water (see Section 10, Data Reporting). PQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturating the detector. Some states may require lower PQLs than those listed above. Project managers will verify laboratory capabilities prior to commencement of a project.

1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.4 This method is applicable to the analyte list below. Not all of the analytes are reported in every case. The analyst has the opportunity to select varying analyte lists from the test categories contained in the LIMS system. In addition, the analyst can select or de-select individual analytes from a test category (see Section 10, Data Reporting, and Section 11, File Maintenance).

Table 1-1
Method 8260B Analyte List

1,1,1,2-Tetrachloroethane	Ethanol
1,1,1-Trichloroethane	Ethyl acetate
1,1,2,2-Tetrachloroethane	Ethyl Methacrylate
1,1,2-Trichloroethane	Ethylbenzene
1,1-Dichloroethane	Freon-113
1,1-Dichloroethene	Freon-141B
1,1-Dichloropropene	Freon-22
1,2,3-Trichlorobenzene	Hexachlorobutadiene
1,2,3-Trichloropropane	Iodomethane
1,2,4-Trichlorobenzene	iso-Butyraldehyde
1,2,4-Trimethylbenzene	Isobutyl Alcohol
1,2-Dibromo-3-chloropropane	Isopropyl acetate
1,2-Dibromoethane	Isopropyl alcohol

1,2-Dichlorobenzene	Isopropyl ether
1,2-Dichloroethane	Isopropylbenzene
1,2-Dichloropropane	m,p-Xylene
1,3,5-Trimethylbenzene	Methyl acetate
1,3-Dichlorobenzene	Methyl formate
1,3-Dichloropropane	Methyl Methacrylate
1,4-Dichlorobenzene	Methyl tert-butyl ether
1,4-Dioxane	Methylacrylonitrile
2,2-Dichloropropane	Methylcyclohexane
2,3-Dimethylbutane/2-Methylpentane	Methylcyclopentane
2-Butanone	Methylene chloride
2-Chloroethyl vinyl ether	n-Amyl acetate
2-Chlorotoluene	n-Butyl acetate
2-Hexanone	n-Butylbenzene
3-Methylpentane	n-Heptane
4-Chlorotoluene	n-Hexane
4-Isopropyltoluene	n-Propylbenzene
4-Methyl-2-pentanone	Naphthalene
Acetone	o-Xylene
Acetonitrile	Pentachloroethane
Acrolein	Phosgene
Acrylonitrile	Propionitrile
Allyl Chloride	sec-Butylbenzene
Benzene	Styrene
Bromobenzene	tert-Butyl Alcohol
Bromochloromethane	tert-Butylbenzene
Bromodichloromethane	Tetrachloroethene
Bromoform	Tetrahydrofuran
Bromomethane	Toluene
Carbon disulfide	trans-1,2-Dichloroethene
Carbon tetrachloride	trans-1,3-Dichloropropene
Chlorobenzene	trans-1,4-Dichloro-2-butene
Chloroethane	Trichloroethene
Chloroform	Trichlorofluoromethane
Chloromethane	Vinyl acetate
Chloroprene	Vinyl chloride
cis-1,2-Dichloroethene	4-Bromofluorobenzene (SS)
cis-1,3-Dichloropropene	Dibromofluoromethane (SS)

Cyclohexane	Toluene-d8 (SS)
Cyclohexanone	Pentafluorobenzene (IS)
Dibromochloromethane	1,4-Difluorobenzene (IS)
Dibromomethane	Chlorobenzene-d5 (IS)
Dichlorodifluoromethane	1,4-Dichlorobenzene-d4 (IS)
Epichlorohydrin	

IS = Internal Standard SS = Surrogate Standard

Oxygenates Analyte List

Ethanol	tert-Butyl formate
Ethyl tert-butyl alcohol	4-Bromofluorobenzene (SS)
Ethyl tert-butyl ether	Dibromofluoromethane (SS)
Isopropyl ether	Toluene-d8 (SS)
Methyl tert-butyl ether	Pentafluorobenzene (IS)
tert-Amyl alcohol	1,4-Difluorobenzene (IS)
tert-Amyl ethyl ether	Chlorobenzene-d5 (IS)
tert-Amyl methyl ether	1,4-Dichlorobenzene-d4 (IS)
tert-Butyl Alcohol	3,3-Dimethyl-1-butanol

IS = Internal Standard SS = Surrogate Standard

2.0 SUMMARY OF METHOD.

2.1 The volatile compounds are introduced into the gas chromatograph by purge-and-trap Method 5030 (aqueous samples) and Method 5035 (solid and waste oil samples). For all samples, the components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of certified standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard with a five-point calibration curve.

3.0 INTERFERENCES.

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. Analysis of calibration and reagent blanks provides

information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter.

3.1.1 It should be recognized that some laboratory extraction solvents such as methylene chloride can be transported directly into the sample(s) through the ventilation system and/or on clothing worn by analysts who have been in the extractions laboratory. For this reason, it is strongly recommended that VOC chemists avoid this work area.

3.1.2 Subtracting blank values from sample results is not permitted. The laboratory must report the sample result and blank result using the qualifying criteria specified in the QA Manual.

3.2 Cross contamination can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of organic-free reagent water to check for cross contamination. The purge-and-trap system may require extensive bake-out and cleaning after a high-concentration sample. However, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample or are below reporting limits, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling point compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water followed by purge and trap grade methanol, and then dried in an oven at 105°C. Screening the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system.

3.4 The laboratory where volatile analyses are performed should be completely free of solvents.

3.5 Many analytes exhibit low purging efficiencies from a 25-ml sample. This often results in significant amounts of the analytes remaining in the sample purge vessel after analysis. After removing the purged sample aliquot, and rinsing the purge vessel three times with organic-free reagent water, the empty vessel should be subjected to a heated purge cycle before analyzing another sample in the same purge vessel.

3.6 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination by running calibration and reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants or, flow controllers with rubber components in the purging device should be avoided.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES.

4.1 Sample collection and preservation.

4.1.1 Aqueous samples to be analyzed by Method 5030 (Purge and Trap) must be collected in at least two 40-ml vials without any headspace. This is accomplished by filling the vial so that a reverse meniscus of liquid rests on the top prior to closing the vial. The vial is pre-preserved in the laboratory using 1:1 HCl. The final pH of the sample should be <2.

4.1.2 Aqueous samples to be analyzed for compounds Styrene, Vinyl chloride, or 2-Chloroethyl vinyl ether must be collected in at least two 40-mL vials without any headspace and without preservation. Samples collected without preservation have a holding time of 7 days of collection.

4.1.3 If a liquid sample contains residual chlorine, add sodium thiosulfate preservative (10mg/40mL is sufficient for up to 5 ppm chlorine) to the empty sample bottle just prior to shipping to the sampling site. After analysis, the sample may be checked for residual chlorine.

4.1.4 After analysis, the pH of liquid samples is checked to verify that they were preserved to the required pH.

4.1.5 Soil samples with high level VOC concentrations (≥200 µg/Kg VOC) to be analyzed by Method 5035 (Closed System Purge and Trap equivalent to obsolete Method 5030A) must be collected by one of the following methods.

4.1.5.1 Samples are collected in an ENCORE or equivalent vial. Upon receipt in the laboratory, the sample(s) are placed into a 40 ml VOA vial containing purge and trap grade methanol in a ratio of 1 gram of sample to 1 mL of solvent. The sample is analyzed by purging a portion of the methanolic extract diluted in water.

4.1.5.2 Samples are collected in 2-oz or 4-oz jars. A 5 gram sample (or other appropriate weight) is added to a pre-tared 40 mL VOA vial containing purge and trap grade methanol at a ratio of 1 mL solvent to 1 gram of sample. The sample is analyzed by purging a portion of the methanolic extract diluted in water.

4.1.6 Soil samples with low level VOC concentrations (<200µg/Kg VOC) to be analyzed by Method 5035 are collected in VOC vials containing sodium bisulfate and analyzed directly on the autosampler without opening the vial.

4.1.6.1 Prior to sampling, verification of the absence of a reaction between the sample and the sodium bisulfite must be confirmed.

4.1.6.2 If a reaction occurs, the samples must be collected in containers that do not have the preservative or contain only DI, VOC free water. Samples

collected in this manner must be analyzed within 48 hours or frozen within 48 hours and analyzed within 14 days of collection.

4.2 Sample holding times and storage.

4.2.1 All water VOC samples must be stored separately from containers requiring other analyses and kept at 4°C ±2°C, but not frozen.

4.2.2 Water samples may be held up to 14 days prior to analysis if preserved to pH <2 with HCl. Unpreserved samples must be analyzed within 7 days of sampling.

4.2.3 Soil samples collected in bisulfate or methanol preservative must be analyzed within 14 days of collection.

4.2.4 Soil samples for low level VOCs collected in empty VOA vials or vials with water preservative only must be analyzed within 48 hours of collection or frozen and within 14 days of collection.

4.2.5 Soil samples for high level volatiles collected in 2oz. or 4oz. jars must be analyzed within 14 days of collection.

4.2.6 Samples collected in ENCORE samplers must be analyzed within 48 hours or placed in appropriate solvents/preservative and analyzed within 14 days of collection.

5.0 REAGENTS AND STANDARDS

5.1 Reagents.

5.1.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1.2 Organic-free reagent water - All references to water in this procedure refer to organic-free reagent water, defined as ASTM Type III.

5.1.3 Methanol, CH_3OH – Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.1.4 Hydrochloric acid (1:1 v/v), HCl – Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.1.5 Sodium bisulfate, ACS grade or equivalent.

5.1.6 Prepare fresh standards for gases weekly. Reactive compounds, such as 2-chloroethylvinyl ether, may need to be prepared more frequently. All other standards must be replaced after six months. Both gas and liquid standards must be monitored closely by comparison to the initial calibration curve and by comparison to QC check standards. It may be necessary to replace the standards more frequently if either check exceeds 15% difference.

5.1.7 Secondary stock solutions: Secondary stock solutions are prepared from purchased stock standards containing varied concentrations. These stock standards are premixed sets that only require dilution to make the secondary stock standards.

5.1.7.1 8260 Calibration Standard: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 250 µL of Table 5-2 reference numbers 1, 2, and 6. Transfer 500 µL of Table 5-2 reference numbers 3, 4, 5, and 39. Bring to volume with methanol. Concentration will be 50-100 µg/mL (1,4-Dixoane will be 500 µg/mL.)

5.1.7.2 8260 Gases Standard: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 250 µL of Table 5-2 reference number 7. Bring to volume with methanol. Concentration will be 50 µg/mL.

5.1.7.3 8260 Matrix Spike: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 200 µL of Table 5-2 reference number 35. Bring to volume with methanol. Concentration will be 50 µg/mL.

5.1.7.4 MBTEX Calibration Standard: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 250 µL of Table 5-2 reference numbers 2 and 21. Bring to volume with methanol. Concentration will be 50-100 µg/mL.

5.1.7.5 3.3 –Dimethyl-1-butanol Intermediate standard: In a 10 mL volumetric flask, add about 7 mL of methanol, transfer 0.1 grams of Table 5-2 reference number 19. Bring to volume with methanol. Concentration will be 10,000 µg/mL.

5.1.7.6 8260 Oxygenates Standard: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 500 µL of Table 5-2 reference number 18. Then transfer 500 µL of the 3.3 –Dimethyl-1-butanol intermediate standard (5.1.7.5). Bring to volume with methanol. Concentration will be 100-500 µg/mL.

5.1.7.7 8260 Custom (Noramco) Standard #1: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 250 µL of Table 5-2 reference number 23. Bring to volume with methanol. Concentration will be 50 µg/mL.

5.1.7.8 8260 Custom (Noramco) Standard #2: In a 10 mL volumetric flask, add about 7 mL of DI water. Transfer 250 µL of Table 5-2 reference number 24. Bring to volume with DI water and store at 4°C. Concentration will be 50 µg/mL.

5.1.7.9 8260 Appendix IX Standard: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 1 mL of Table 5-2 reference number 14. Transfer 100 µL of Table 5-2 reference number 15. Bring to volume with methanol. Concentrations will be varied.

5.1.7.10 4-Bromofluorobenzene (BFB) standard: In a 5 mL volumetric flask, add about 3 mL of methanol. Transfer 500 µL of Table 5-2 reference number 32. Bring to volume with methanol. Concentration will be 250 µg/mL.

5.1.7.11 8260 Calibration Standard 2nd Source: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 250 µL of Table 5-2 reference numbers 6 (different lot # required), 8, and 12. Transfer 500 µL of Table 5-2 reference numbers 9, 10, 11, and 39 (different lot # required.) Bring to volume with methanol. Concentration will be 50-100 µg/mL (1,4-Dixoane will be 500 µg/mL.)

5.1.7.12 8260 Gases Standard 2nd Source: In a 10 mL volumetric flask, add about 7 mL of methanol. Transfer 250 µL of Table 5-2 reference number 13. Bring to volume with methanol. Concentration will be 50 µg/mL.

5.1.8 Calibration standards - Calibration standards are prepared at a minimum of five concentrations from the secondary stock standards. Prepare these solutions in organic-free reagent water. Prepare calibration standards at the concentration levels shown in table 5-1. Calibration standards must be prepared the day of use.

Table 5-1
8260B Water Calibration Curve Preparation

Calibration Standard Concentration (µg/L)	Secondary Stock Standard Concentration (µg/mL)	Amount of Secondary Standard added to 50mLWater
2-4	50-100	2 µL
5-10	50-100	5 µL
10-20	50-100	10 µL
20-40	50-100	20 µL
50-100	50-100	50 µL
100-200	50-100	100 µL
150-300	50-100	150 µL
200-400	50-100	200 µL

Table 5-1 Continued
8260B Soil Calibration Curve Preparation

Calibration Standard Concentration (µg/L)	Secondary Stock Standard Concentration (µg/mL)	Amount of Secondary Standard added to 5.0mLWater
2-4	50-100	0.2 µL
5-10	50-100	0.5 µL
10-20	50-100	1.0 µL
20-40	50-100	2.0 µL
50-100	50-100	5.0 µL
100-200	50-100	10.0 µL
150-300	50-100	15.0 µL
200-400	50-100	20.0 µL

MBTEX Water Calibration Curve Preparation

Calibration Standard Concentration (µg/L)	Secondary Stock Standard Concentration (µg/mL)	Amount of Secondary Standard added to 50mLWater
1-2	50-100	2 µL
5-10	50-100	5 µL
10-20	50-100	10 µL
50-100	50-100	50 µL
100-200	50-100	100 µL
200-400	50-100	200 µL

MBTEX Soil Calibration Curve Preparation

Calibration Standard Concentration (µg/L)	Secondary Stock Standard Concentration (µg/mL)	Amount of Secondary Standard added to 5.0mLWater
1-2	50-100	0.2 µL
5-10	50-100	0.5 µL
10-20	50-100	1.0 µL
50-100	50-100	5.0 µL
100-200	50-100	10.0 µL
200-400	50-100	20.0 µL

Table 5-1 Continued
Oxygenates Calibration Curve Preparation

Calibration Standard Concentration (µg/L)	Secondary Stock Standard Concentration (µg/mL)	Amount of Secondary Standard added to 50mLWater
1-5	50-250	1 µL
5-25	50-250	5 µL
20-100	50-250	20 µL
50-250	50-250	50 µL
100-500	50-250	100 µL
200-1000	50-250	200 µL
400-2000	50-250	400 µL

5.1.9 Surrogate standards: Dibromofluoromethane, 4-Bromofluorobenzene and Toluene-d_8. Internal standards: Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4.

5.1.10 Internal Standard/Surrogate spiking solution: In a 5 mL volumetric flask, add about 3 mL of methanol. Transfer 500 µL of Table 5-2 reference numbers 36 and 37. Bring to volume with methanol and place in standard reservoir in Archon auto sampler. Concentration will be 250 µg/mL.

5.1.10.1 Each sample undergoing GC/MS analysis must be spiked with 1 µl of the internal standard/surrogate spiking solution prior to analysis utilizing the Archon auto sampler.

5.1.11 Caution, when preparing calibration standards, it is important to keep the methanol concentration at a minimum (i.e. **not to exceed 2%**).

5.1.12 Matrix spike and laboratory control samples (LCS):

5.1.12.1 8260 Water LCS: Add 50 µL of 8260 Matrix Spiking standard (5.1.7.3) to 50 mL of DI water. Concentration will be 50 µg/L.

5.1.12.2 8260 Water MS/MSD: Add 43 µL of 8260 Matrix Spiking standard (5.1.7.3) directly through septum of sample vial. Shake sample to ensure proper mixing. Concentration will be approximately 50 µg/L

5.1.12.3 MBTEX Water LCS: Add 50 µL of MBTEX Calibration standard (5.1.7.4) to 50 mL of DI water. Concentration will be 50-100 µg/L.

5.1.12.4 MBTEX Water MS/MSD: Add 43 µL of MBTEX Calibration standard (5.1.7.4) directly through septum of sample vial. Shake sample to ensure proper mixing. Concentration will be approximately 50-100 µg/L

5.1.12.5 8260 Soil LCS: Add 5 µL of 8260 Matrix Spiking standard (5.1.7.3) to vial containing a stir bar and 5 mL of sodium bisulfate solution at the same concentration as the client samples. Concentration will be 50 µg/Kg.

5.1.12.6 8260 Soil MS/MSD: Add 5 µL of 8260 Matrix Spiking standard (5.1.7.3) to vial containing a stir bar, approximately 5 grams of client soil sample, and 5 mL of sodium bisulfate solution at the same concentration as the client samples. Concentration will be 50 µg/Kg.

5.1.12.7 MBTEX Soil LCS: Add 5 µL of MBTEX Calibration standard (5.1.7.4) to vial containing a stir bar and 5 mL of sodium bisulfate solution at the same concentration as the client samples. Concentration will be 50-100 µg/Kg.

5.1.12.8 MBTEX Soil MS/MSD: Add 5 µL of MBTEX Calibration standard (5.1.7.4) to vial containing a stir bar, approximately 5 grams of client soil sample, and 5 mL of sodium bisulfate solution at the same concentration as the client samples. Concentration will be 50-100 µg/Kg.

5.1.12.9 Oxygenates LCS: Add 200 µL of 8260 Oxygenates standard (5.1.7.6) to 50 mL of DI water. Concentration will be 400-2000 µg/L.

5.1.12.10 Oxygenates MS/MSD: Add 172 µL of 8260 Oxygenates standard (5.1.7.6) directly through septum of sample vial. Shake sample to ensure proper mixing. Concentration will be approximately 400-2000 µg/L.

5.1.13 Continuing calibration verification standard (CCV):

5.1.13.1 8260 Water CCV: Prepare by adding 50 µL of 8260 Calibration standard (5.1.7.1) and 8260 Gases standard (5.1.7.2) to 50 mL of DI water. Concentration will be 50-100 µg/L.

5.1.13.2 MBTEX Water CCV: Prepare by adding 50 µL of MBTEX Calibration standard (5.1.7.4) to 50 mL of DI water. Concentration will be 50-100 µg/L.

5.1.13.3 8260 Soil CCV: Prepare by adding 5 µL of 8260 Calibration standard (5.1.7.1) and 5 µL of 8260 Gasses standard (5.1.7.2) to a vial containing a stir bar and 5 mL of sodium bisulfate solution at the same concentration as the client samples. Concentration will be 50-100 µg/Kg.

5.1.13.4 MBTEX Soil CCV: Prepare by adding 5 µL of MBTEX Calibration standard (5.1.7.4) to a vial containing stir bar and 5 mL of sodium bisulfate solution at the same concentration as the client samples. Concentration will be 50-100 µg/Kg.

5.1.13.5 Oxygenates CCV: Prepare by adding 200 µL of 8260 Oxygenates standard (5.1.7.6) to 50 mL of DI water. Concentration will be 400-2000 µg/L.

5.1.14 Initial calibration verification standard (ICV). This standard is prepared in the same manner as a continuing calibration verification standard **except** that it is prepared from a stock solution purchased from a different vendor or a different lot than the stock solution that is used for the calibration curve. This stock solution is also used in the preparation of standards to be analyzed as part of the IDOC/CDOC (see Section 8.0, Quality Assurance Procedures). Sodium bisulfate must be added to soil standards in the same concentration as the client samples.

5.2 Vendor List. Pertinent information related to the purchase of standards is contained in table 5-2 below. All of the standards are ordered using the catalog numbers and vendors indicated below.

Table 5-2
8260B Standards and Chemicals

Reference Number	Standard Name	Vendor Name	Concentration	Catalog Number
1	502.2 MegaMix (1st Source)	Restek	2000 µg/mL	30431
2	Methyl t-butyl ether (MtBE) (1st Source)	AccuStandard	2000 µg/mL	S-078-10X
3	Acrolein &Acrylonitrile (1st Source)	Supelco	2000 µg/mL	46870-U
4	8260 Additions (1st Source)	Supelco	2000 µg/mL	46831-U
5	trans-1,4-dichloro-2-butene (1st Source)	Restek	2000 µg/mL	30274
6	4.2 Custom 8260 Standard (2 lot #'s required)	Accustandard	2000 µg/mL	S-10776A
7	502.2 Calibration Mix #1 (Gases) (1st Source)	Restek	2000 µg/mL	30042
8	54 Liquid Volatile Comp. (2nd Source)	AccuStandard	2000 µg/mL	M-502A-R-10X
9	8260 Additions (2nd Source)	AccuStandard	2000 µg/mL	M-8260-ADD-10X
10	trans-1,4-Dichloro-2-butene (2nd Source)	AccuStandard	2000 µg/mL	App-9-068-20X
11	Acrolein & Acrylonitrile (2nd Source)	Supelco	2000 µg/mL	4S6870-U
12	Methyl t-butyl ether (MtBE) (2nd Source)	Restek	2000 µg/mL	30402
13	6 Gas Components (2nd Source)	AccuStandard	2000 µg/mL	M502B-10X
14	APP IX Volatile Std. (1st Source)	AccuStandard	Varies	M-8240C-R3
15	Chloroprene (1st Source)	Restek	2000 µg/mL	30238
16	APP IX Volatile Std. (2nd Source)	AccuStandard	Varies	M-8240C-R3-10X
17	Chloroprene (2nd Source)	Accustandard	2000 µg/mL	APP-9-048-R1-10X
18	Oxygenate Mix (2 lot #'s required)	AccuStandard	Varied	S-12689-R1
19	3,3 Dimethyl-1-butanol (1st Source)	Sigma Aldrich	99.9%	183105-1G
20	3,3 Dimethyl-1-butanol (2nd Source)	TCI America	99.9 %	D1333
21	BTEX+MtBE (1st Source)	Restek	2000 µg/mL	30231
22	California Gas. Range Hydrocarbons (BTEX 2nd Source)	AccuStandard	2000 µg/mL	S-603A-10X-PAK
23	Custom Noramco Std #1 (2 lot #'s required)	Accustandard	2000 µg/mL	S-13557
24	Custom Noramco Std #2 (2 lot #'s required)	Accustandard	2000 µg/mL	S-13556
25	Tetrahydrofuran (2 lot #'s required)	Restek	2000 µg/mL	30414
26	Ethyl Acetate (1st Source)	Sigma Aldrich	99.99%	34858-100ML
27	Ethyl Acetate (2nd Source)	Supelco	2000 µg/mL	4-7947
28	Isopropyl Alcohol	EM Science	99.9 %	PX1835P-4
29	1,1-Dichloro-1-Fluorothane (Freon 141B)	Ultra Scientific	100 µg/mL	CFC-250
30	Chlorodifluoromethane (Freon 22)	Ultra Scientific	100 µg/mL	CFC-110
31	1,2-Dichlorotetrafluoroethane (Freon 114)	Restek	100 µg/mL	30476
32	4-Bromofluorobenzene (BFB)	AccuStandard	2500 µg/mL	CLP-004-100X
33	Fluorobenzene	Restek	2000 µg/mL	30030
34	Surrogate Control Std. (BTEX)	AccuStandard	5000 µg/mL	AK-101.0-SS-100X-PAK
35	Matrix Spiking Sol.	AccuStandard	2500 µg/mL	CLP-003-R-10X-PAK
36	8260 Internal Standards	Restek	2500 µg/mL	30074
37	8260 Surrogates	Restek	2500 µg/mL	30073
38	P&T Grade Methanol	EMD	99.99%	MX0482-6
39	1,4-Dioxane (2 lot #'s required)	AccuStandard	10,000 µg/mL	AS-E0480

6.0 APPARATUS AND MATERIALS .

(All listed materials may be substituted for other equivalent materials)

6.1 Purge-and Trap devices.

6.1.1 OI-4560 Concentrator with 25 cm Supelco Carbopack/Carbosieve trap.

6.1.2 OI-4660 Eclipse Concentrator with 25 cm OI #10 trap. OI #7 trap used for low level 1,4-Dioxane analysis only.

6.2 Injection port liners – Splitless liner.

6.3 GC/MS.

6.3.1 Gas chromatograph – Agilent GC 6850.

6.3.2 Gas chromatograph – Agilent GC 6890.

6.3.3 Column – DB-624, 25 m X 0.2 mm ID, 1.12 µm film thickness.

6.3.4 Mass Spectrometer – Hewlett Packard 5973 MSD series.

6.3.5 Mass Spectrometer – Hewlett Packard 5973 N series.

6.4 Data System – Hewlett Packard ChemStation with Enviroquant software.

6.5 Microsyringes – 1 µL, 10 µL, 25 µL, 100 µL, 250 µL, 500 µL, 1,000 µL, and 5000 µL.

6.6 Syringe valve – Two-way, with Luer ends (three each), if applicable to the purging device.

6.7 Syringe – 5 mL and 25 mL, gas-tight with shutoff valve.

6.8 Balances - Analytical, 0.0001 g, and top-loading, 0.1 g.

6.9 Glass scintillation vials – 20 mL and 40 mL, with screw cap and Teflon liner or glass culture tubes with a screw cap and Teflon liner.

6.10 Volumetric flasks, Class A – 5 mL, 10 mL, 25 mL, 50 mL and 100 mL, with ground glass stoppers.

6.11 Vials – 2 mL crimp top.

6.12 Spatula - Stainless steel.

6.13 Disposable pipettes – Pasteur.

6.14 40 mL, screw cap, PTFE lined, septum-sealed vials (VOC vials), preserved with 0.5 ml HCl for aqueous samples.

6.15 40 mL, screw cap, PTFE lined, septum-sealed vials (VOC vials), preserved with 5 mL sodium bisulfate solution, pre-tared, and containing a small stirring bar for soil samples with suspected low concentrations of VOCs.

6.16 40 mL, screw cap, PTFE lined, septum-sealed vials (VOC vials), preserved with 5 mL of purge & trap grade methanol, and pre-tared for soil samples with suspected high concentrations of VOCs.

6.17 Carrier Gas, Helium - 99.99% or higher purity.

6.18 Varian Archon vial autosampler.

6.19 Field sampling equipment (not supplied by the laboratory includes a Model 3780 PT (Associated Design and Manufacturing Company) or EnCore™ sampler). The laboratory supplies EnCore™ sample containers for high concentration samples.

7.0 PROCEDURE

7.1 Sample collection for aqueous samples.

7.1.1 Clients are encouraged to collect a minimum of two VOC vials for each sample. The vials are sent with 0.5 mL of HCl to provide a final pH that is <2.

7.1.2 For aqueous samples to be analyzed for compounds Styrene, Vinyl chloride, or 2-Chloroethyl vinyl ether, clients are encouraged to collect a minimum of two 40-mL vials without any headspace and without preservation. Samples collected without preservation have a holding time of 7 days of collection

7.1.3 Samples are collected so that a reverse meniscus is formed at the top of the vial. When the top is closed, no air bubbles should be present.

7.2 Sample collection for solid samples.

7.2.1 Samples suspected of containing high concentrations of VOCs are collected in duplicate in EnCore samplers and submitted to the laboratory. Within 48 hours of collection, the sample is weighed and placed into a vial with a volume of methanol equal to the weight of the sample.

7.2.2 Samples suspected of containing low concentrations of VOCs are collected in duplicate in tared VOC vials containing 5 mL of sodium bisulfate preservative solution and a stir bar. Samples are re-weighed to determine sample weight and analyzed directly on the Archon autosampler without any further laboratory preparation.

7.2.2.1 A second portion of the soil sample is combined with the sodium bisulfate preservative in the field to determine if any effervescent reaction will occur between the sample and the preservative.

7.2.2.2 The laboratory supplies extra vials without sodium bisulfate preservative for samples that exhibit an effervescent reaction upon mixture with the preservative.

7.3 Preparation of run log form and extraction log in LIMS.

7.3.1 Each day the section supervisor prepares a work log. The log lists samples that are included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.3.1.1 Any samples that are received in a “rush” status will have a chain of custody delivered to the supervisor by sample receiving personnel.

7.3.1.2 Prepare an electronic data sheet in LIMS using the following procedure.

7.3.1.2.1 Open Prep Batch in LIMS by double clicking the “Prep” box..

7.3.1.2.2 To create a new form, click the “add” box. The prep start date, start time, and batch number are automatically created by the LIMS. Make sure that prep start date and time are correct.

7.3.1.2.3 Select the Prep Code “5030” or “5035” from the pull down list. The LIMS will automatically assign an MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCSD and enter the information.

7.3.1.2.4 Enter the technician’s name from the pull down menu.

7.3.1.2.5 Click the “Load Samps” tab to obtain a list of samples that need preparation by this method.

7.3.1.2.6 Select the samples in the batch that are assigned the desired prep method. The samples can be individually selected by highlighting the

sample and clicking the "→" arrow, or all samples can be selected by clicking the "⇒" arrow.

7.3.1.2.7 Samples that have be selected for re-analysis can be manually added to the sample list.

7.3.1.2.8 Enter prep completion date and time.

7.3.1.2.9 "Save" the batch by clicking a previous batch number on the list and then returning to the newly created batch.

7.3.2 Table 7-1 indicates the type of samples that comprise an analytical batch. Note: Laboratory requirements specify that the maximum number of client samples in an analytical batch can not exceed 20.

Table 7-1
Samples in a NELAC Batch

Method Blank (MB)
LCS
Client Samples
MS and MSD (if supplied by client)

7.3.3 The analyst should always place the MS, MSD, MB, and LCS samples in the beginning of the batch. If the batch is continued a second day, early samples may be reported because QC samples have already been analyzed. See the QA Manual for additional information related to batches.

7.4 Soil Sample Preparation.

7.4.1 Low concentration soil method (Approximate concentration range of 5 to 200 µg/Kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.) This procedure is designed for a sample size of approximately 5 g, but other sizes are also applicable as actual sample size is controlled at field sampling.

7.4.1.1 Field preserved sample vials are never to be opened at any time during analysis.

7.4.1.2 Remove the sample vials from storage and allow them to warm to room temperature.

7.4.1.3 Record all sample IDs in the sample log table, carefully wipe off any soil present on the outside of the vial and weigh the vial and contents to the nearest 0.01 g. Record the total weight of vial and contents. Even if final

weight was determined in the field, vials must be weighed and recorded by the lab prior to analysis. Any significant discrepancy between the lab determined weight and the field determined weight must be described in the Case Narrative.

7.4.1.4 For MS/MSD vials supplied by the client, matrix spike is added through the septa using a syringe just prior to loading into the autosampler.

7.4.1.5 If client supplied vials are not available for MS/MSD, vials are prepared by weighing 5.0 g ±0.2 g of soil into pre-preserved vials and immediately sealing the vials. The initial weights used for the matrix spike and matrix spike duplicate must be within 5% of each other. Spike solution is added through the septa using a syringe just prior to loading into the autosampler.

7.4.1.6 Shake the vial gently to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the Archon carousel.

7.4.1.7 Add 5 ml of organic-free reagent water, the internal standards, and the surrogate compounds to all samples. This is carried out using the automated sampler. Prior to purging, heat the sample vial to 40°C for 1.0 minute.

7.4.1.8 For the sample selected for matrix spiking, add the matrix spiking solution. Proceed to Section 7.6, Purge Conditions.

7.4.2 High concentration method for soil samples with concentrations generally greater than 200 µg/Kg. The high concentration method for soil is based on a methanol extraction. The solid sample is either extracted or diluted, depending on sample solubility, in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing surrogates and, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by the appropriate determinative method.

7.4.2.1 When the high concentration sample is not preserved in the field, the sample is the **entire** content of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

7.4.2.2 Add 5 mL of methanol to a pre-tared 40 mL vial. Using a top-loading balance, weigh 5 g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.01 g. Shake the vial for 2 minutes.

7.4.2.3 For soil and solid waste samples that were collected in methanol, weigh the

vial to 0.01 g as a check on the weight recorded in the field, and shake for 2 minutes, as described above.

7.4.2.4 Prior to analysis, the extracts must be stored in the dark at 4°C. Add an appropriate aliquot of the extract to 50 mL of organic-free reagent water in a volumetric flask, add dilution to a VOC vial, and analyze by Method 5030 in conjunction with the appropriate determinative method.

7.4.2.5 If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample (Section 7.7).

7.4.3 High concentration method for oily waste samples. This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol. However, care must be taken to avoid introducing any of the floating oil layers into the instrument. A portion of the diluted sample is then added to 50 ml of organic-free reagent water, purged according to Method 5030, and analyzed.

7.4.3.1 The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.4.3.3.

7.4.3.2 If the waste was not preserved in the field, do the following:

7.4.3.2.1 For solid wastes, weigh 1g (wet weight) of the sample into a pre-tared 40 mL VOC vial. Quickly add 10 mL of methanol and cap the vial. Swirl the vial to mix the contents and then shake vigorously for 2 minutes.

7.4.3.2.2 For liquid wastes, transfer 1 mL of the waste and record the weight on the vial in case the density is needed for reporting in liquid units. Bring the weight up to 1 g and quickly add 10 mL of methanol and cap the vial. Swirl the vial to mix the contents and then shake vigorously for 2 minutes.

7.4.3.3 If the sample was collected in the field in a vial containing methanol, weigh the vial to 0.01 g as a check on the weight recorded in the field. Swirl the vial to mix the contents and then shake vigorously for 2 minutes.

7.4.3.4 Add the appropriate amount of the methanol extract to 50 mL of organic-free reagent water, place into a VOC vial, and analyze by Method 5030, Purge-and-Trap.

7.4.4 Prep Method 3585 using Methanol for highly contaminated or highly complex samples.

7.4.4.1 Transfer approximately 1 g of the oil phase of the sample to a vial or a 10mL volumetric flask. Record the weight to the nearest 0.1 g. Wipe the mouth of the vial with a Kimwipe to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

7.4.4.2 Immediately dilute to volume with methanol.

7.4.4.3 Add surrogate spiking solution, if required, for the analytical method to be employed.

7.4.4.4 Cap and shake the sample for 2 minutes.

7.4.4.5 Analysis by GCMS 8260B.

7.5 Water Sample Preparation.

7.5.1 Remove the sample vial from storage and allow it to warm to room temperature.

7.5.2 Add 43 µL of Matix Spiking Solution to create MS and MSD samples. Add the internal standards and the surrogate compounds to all samples using the automated sampler.

7.6 Sample Concentrator (Purge) Conditions.

7.6.1 Purge the sample with helium at a flow rate of 40 mL/minute for 11 minutes with soil samples being agitated with a magnetic stirring bar or by other mechanical means during the purge cycle. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

7.6.2 After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 220°C without a flow of desorption gas. After the trap has come to temperature, flow the desorption gas at a rate of 10 mL/minute for 2 minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.6.3 After desorbing the sample for 2 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 220°C. After approximately 6 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.6.4 Purge conditions are modified when analyzing for low lever 1,4-Dioxane only as follows:

7.6.4.1 Sample Purge Temperature: 60^{o}C

7.6.4.2 Trap Temperature: 40°C
7.6.4.3 Sample Desorb Temperature: 40°C
7.6.4.4 Sample Desorb Time: 0.5 minutes
7.6.4.5 Trap Bake Time: 10 minutes

7.7 Determination of Dry Weight

7.7.1 If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample.

7.7.2 Weigh 5-10 g of the sample from the 4-oz jar into a tared crucible.

7.7.3 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

7.8 GC/MS Conditions and Operating Parameters.

7.8.1 The recommended GC/MS operating conditions are shown in Table 7-2 below.

Table 7-2
Recommended GC/MS Conditions

Electron energy:	70 volts (nominal)
Mass range:	35-300 amu
Scan time:	2 scan/second
Initial column temperature:	45°C
Initial column holding time:	5 minutes
Column temp. program:	10°C/minute
Final column temperature:	240°C
Final Injector temperature:	250°C
Source temperature:	250°C
Transfer line temperature:	180°C
Carrier gas flow rate:	1 ml/minute

7.8.2 Purge-and-Trap Operating Conditions

Aqueous samples can be purged at temperatures above those recommend as long as all calibration standards, samples, and QC samples are purged at the same temperature, the appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Recommended purge-and-trap conditions are listed below:

Purge Time	11 ± 1 minutes
Purge-Flow Rate	20-40 ml/minute
Sample Desorb Temperature	220°C
Sample Desorb Time	2.0 minute

Note: These operating conditions may differ depending on the analytical system.

7.9 Preparation of "Run Log" using Chemstation/Enviroquant software.

7.9.1 Open Enviroquant software by "clicking" on Icon.

7.9.2 Select "Sequence", then "Edit sample log table".

7.9.3 Select sample type for each sample. Sample types include the following:

BFB = BFB Tune Sample
8260 CCV 050 W = 50 µg/L VOC Standard
MB = Method Blank
LCS = Laboratory Control Sample

7.9.4 Number all of the samples.

Change data file using the following format V7003516 where:

V = volatiles
Number 7 = instrument number
003516 = sequential file number

7.9.5 Click "OK" when completed.

7.9.6 On the pull-down menu, select "Sequence", then "Save".

7.9.7 When the prompt appears, change the last part to MM DD format indicating the current day.

7.9.8 When the following prompt appears, change the last part to MM DD format indicating the current day.

C:\HPCHEM\4\Data\410305, where:
010305 = year "01", month "03", day "05"

7.9.9 When complete, click "OK".

7.9.10 On the pull-down menu, select "Sequence", then "Run" to start the instrument run.

7.10 Editing Sequences.

7.10.1 Click “File”, then “Edit”.

7.10.2 Sample information can be directly entered into the pop up box that appears.

7.11 Initial calibration of the GC/MS System.

7.11.1 Prior to the analysis of any tunes, standards, or samples, several MS and GC parameters should be checked and recorded in order to ensure that the operating conditions have not changed significantly from those of prior analyses. The routine system checks are as follows:

7.11.1.1 The MS vacuum system source pressure should be checked and should be stable. (Acceptable pressure is approximately 50 mtorr or lower when the GC oven temperature is 250°C.).

7.11.1.2 The MS system should be checked for leaks by monitoring for selected ions that indicate the presence of air and water (m/z ratio at 18, 28, 32, & 44).

7.11.1.3 A manual tune should be performed. Record the ion abundances and relative percentages characteristic for the manual tuning compound PFTBA.

7.12 Initial GC/MS calibration curve.

7.13 The initial calibration for SW-846 chromatographic methods involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.

7.13.1.1 The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not appropriate and may lead to significant quantitative errors regardless of the calibration model chosen. Analysts are advised that it may be necessary to prepare calibration standards that cover concentration ranges that are appropriate for specific projects or type of analyses. For instance, the analyst should not necessarily expect to be able to perform a calibration appropriate for sub-ppb level analyses and also use the same calibration data for high-ppb or ppm level samples.

7.13.1.2 Calibration curve must have at least 5 points with the lowest concentration equal to the LIMS PQL and the highest concentration equal to the LIMS UQL.

7.13.2 Calibration using average response with internal standards. For each of the initial calibration standards, calculate the RF values for each target compound relative to the internal standards as follows:

$$RF = \frac{A_s \ X \ C_{is}}{A_{is} \ X \ C_s}$$

where:

A_s = Peak area or height of the analyte or surrogate
A_{is} = Peas area or height of the internal standard
C_s = Concentration of the analyte or surrogate
C_{is} = Concentration of the internal standard

7.13.2.1 SW-846 methods allow the use of both linear and non-linear models for the calibration data. It is AES' policy that non-linear models are not used in any case. In all methods employed by the laboratory, the correlation between response and concentration can be described in a linear method, either average response or linear regression.

7.13.2.2 For average response to be used, %RSD for each target analyte has to be $\leq$15% RSD. If the %RSD for any analyte is >15%, then linear regression is used with a correlation coefficient of $\geq$0.995. For CCC compounds, if % RSD is >15%, then linear regression must be used with a correlation coefficient of $\geq$0.995, but the %RSD for any CCC compound cannot exceed 30% or corrective action per Section 7.14.3.3 must be taken prior to further analysis.

7.13.3 Calibration using linear regressions. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

where:

y = Instrument response (peak area or height)
m = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = The intercept

When internal standards are used, the equation becomes:

$$\frac{As \ Cis}{Ais} = aCs + b$$

where:

A = Area (or height) of the peak for the target analyte in the samples
A = Area (or height) of the peak for the internal standard is
C = Concentration of the target analyte in the calibration standards
C = Concentration of the internal standard is
a = Slope of the line (also called the coefficient of C) s
b = The intercept

7.13.3.1 The analyst should not force the line through the origin, but have the intercept calculated from the five data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0,0) as a sixth calibration point.

7.13.3.2 The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, per AES' policy, r must be greater than or equal to 0.995.

7.13.3.3 The Enviroquant software will automatically calculate the linear regression for any target analyte by selecting "linear regression" in the calculation mode. When in this mode, the software plots the analyte response ratio against the analyte ratio.

7.13.3.3.1 The response ratio is defined as the area of the analyte divided by the area of the internal standard.

7.13.3.3.2 The analyte ratio is defined as the concentration of the analyte divided by the concentration of the internal standard.

7.14 Daily GC/MS calibration

7.14.1 BFB Tune Evaluation. Before analysis and every 12 hours of operation, the hardware tuning for each GC/MS system must be verified as follows:

7.14.1.1 Inject or purge 50 ng of 4-Bromofluorobenzene (BFB) using a 1 µL injection of the 250 µg/mL BFB standard.

7.14.1.2 Spectral information must be obtained using one of the following methods:

7.14.1.2.1 The average of the apex scan plus one scan to the left of the apex and one scan to the right of the apex with background subtraction using a single scan no more that 20 scans from the apex and completely off the BFB peak. Do not subtract part of the BFB peak.

7.14.1.2.2 A single scan within ±3 scans of the apex with background subtraction using a single scan no more than 20 scans from the apex and completely off the BFB peak. Do not subtract part of the BFB peak.

7.14.1.2.3 The average scan across the entire chromatographic peak with background subtraction using a single scan no more than 20 scans from the apex and completely off the BFB peak. Do not subtract part of the BFB peak.

7.14.1.3 Ion abundances for the BFB resulting from 7.14.1 must meet the criteria specified in Table 7-3 below before analysis of samples and/or standards may begin.

7.14.1.4 If ion abundance criteria are not met, proceed with one or both of the following options:

7.14.1.4.1 Make a fresh BFB standard and reinject. If Table 7-3 criteria are now met using evaluation per 7.14.1, analysis may continue.

7.14.1.4.2 If Table 7-3 criteria are not met, instrument maintenance must be performed to restore ion abundance ratios to meet Table 7-3 criteria and/or the instrument must be re-tuned to restore ion abundance rations to meet Table 7-3.

Table 7-3
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	base peak, 100% relative abundance
96	5-9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5-9% of mass 174
176	>95% but <101% of mass 174
177	5-9% of mass 176

7.14.2 Initial and Continuing Calibration Verification (ICV) and (CCV). The ICV is injected to verify the initial calibration. The CCV is injected every 12 hours of operation. The calibration verification standards are mixtures that are composed of compounds designed to measure the following criteria.

7.14.2.1 Verification of the calibration curve.

7.14.2.2 Specific compounds within the mixtures are used to verify system performance. These are the System Performance Check Compounds (SPCCs).

7.14.2.3 Specific compounds within the mixtures are used to verify system integrity. These are the Calibration Check Compounds (CCCs).

7.14.3 After injection of the ICV or CCV, determine the percent difference for all RF calibrated compounds or percent drift for all LR calibrated compounds between the known and the measured concentrations for all compounds within the standard.

7.14.3.1 For all SPCC compounds, calculate the RF from the ICV/CCV and verify that the minimum RF requirements per Table 7-4 have been met. If any RF is less than the required value, corrective actions per Section 7.14.3.4 must be taken prior to further analysis.

7.14.3.1.1 The minimum acceptable average RFs for these compounds are as indicated below.

Table 7-4
SPCC Volatile Compounds

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.14.3.2 After SPCC criteria are met, the CCCs are used to check the validity of the initial and continuing calibration. The value obtained must be less than or equal to 20% difference or drift of the expected value for all CCCs. If any % difference or drift is greater than 20%, corrective action per Section 7.14.3.3 must be taken prior to further analysis.

7.14.3.2.1 The CCC compounds are shown in Table 7-5.

Table 7-5
Calibration Check Compounds (CCCs)

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

7.14.3.3 Corrective actions for failure to meet any of the above criteria may include, but are not limited to, reinjection of freshly prepared standard, inlet maintenance, column maintenance, etc. If corrective actions do not result in all evaluation criteria being met, recalibration is required prior to further analysis.

7.14.3.4 Calculate the percent RSD using the following equation:

$$\%RSD = \frac{SD}{RF_{avg}} \text{ X } 100$$

where:
RF_{avg} = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RFi - RFavg)^2}{N-1}}$$

where:
RF_I = RF for each of 5 calibration levels
N = number of RF values (i.e., 5)

7.14.4 Evaluation of retention times. The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. If the retention time for any internal standard changes by more than 0.06 RRT units from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. Table 7-6 lists the volatile organic compounds, their retention times, and primary and secondary ions. Note that these retention times are approximate and may change if GC/MS conditions are changed.

7.14.5 Further evaluation of retention times. If the retention time for any internal standard in any ICV or CCV changes by more than 30 seconds (0.50 minutes) from that in the mid-point level of the most recent initial calibration, the chromatographic system must be inspected for malfunctions and corrections made as required.

Table 7-6
Retention Times and Characteristic Ions Volatile Compounds

Compound	Retention Time (Minutes)	Primary Ion	Secondary Ion(s)
1,1,1,2-Tetrachloroethane	10.48	131	133, 119

1,1,1-Trichloroethane	5.70	97	99, 61
1,1,2,2-Tetrachloroethane	11.53	83	131, 85
1,1,2-Trichloroethane	9.33	83	97, 85
1,1-Dichloroethane	4.08	63	65, 83
1,1-Dichloroethene	2.73	96	61, 63
1,1-Dichloropropene	5.97	110	75, 77
1,2,3-Trichlorobenzene	14.02	180	182, 145
1,2,3-Trichloropropane	11.56	75	77
1,2,4-Trichlorobenzene	13.70	180	182, 145
1,2,4-Trimethylbenzene	12.06	105	120
1,2-Dibromo-3-chloropropane	13.14	75	155, 157
1,2-Dibromoethane	9.91	107	109, 188
1,2-Dichlorobenzene	12.61	146	111, 148
1,2-Dichloroethane	6.26	62	98
1,2-Dichloropropane	7.43	62	111
1,3,5-Trimethylbenzene	11.77	105	120
1,3-Dichlorobenzene	12.28	146	111, 148
1,3-Dichloropropane	9.53	76	78
1,4-Dichlorobenzene	12.33	146	111, 148
1,4-Dioxane	7.62	88	58, 43, 57
2,2-Dichloropropane	4.96	77	97
2,3-Dimethylbutane/2-Methylpentane	3.32	43	42, 41
2-Butanone	5.06	72	43
2-Chloroethyl vinyl ether	7.43	63	41, 76
2-Chlorotoluene	11.70	91	126
2-Hexanone	9.68	43	58, 100
3-Methylpentane	3.63	57	56
4-Chlorotoluene	11.79	91	126
4-Isopropyltoluene	12.29	119	134, 91
4-Methyl-2-pentanone	8.62	43	58, 85, 100
Acetone	2.77	58	43
Acetonitrile	3.04	41	40, 39
Acrolein	2.64	56	55
Acrylonitrile	3.49	53	52, 51
Allyl Chloride	3.10	76	41, 39
Benzene	6.24	78	77
Bromobenzene	11.55	156	77, 158
Bromochloromethane	5.31	128	49, 130

Bromodichloromethane	7.81	83	85, 127
Bromoform	11.14	173	175, 254
Bromomethane	1.99	94	96
Carbon disulfide	2.97	76	--
Carbon tetrachloride	5.94	119	117
Chlorobenzene	10.40	112	77, 114
Chloroethane	2.08	64	66
Chloroform	5.44	83	85
Chloromethane	1.61	50	52
Chloroprene	4.23	53	88, 90
cis-1,2-Dichloroethene	4.96	96	61, 98
cis-1,3-Dichloropropene	8.42	75	110, 112
Cyclohexane	5.80	56	69, 84
Cyclohexanone	11.51	55	98, 42
Dibromochloromethane	9.78	129	127
Dibromomethane	7.59	93	95, 174
Dichlorodifluoromethane	1.48	85	87
Epichlorohydrin	8.47	57	49, 62
Ethanol	2.44	45	43, 55
Ethyl acetate	5.20	43	45, 61
Ethyl Methacrylate	9.28	69	41, 99, 86
Ethylbenzene	10.52	91	106
Freon-113	2.76	101	151, 85
Freon-141B	2.53	81	83, 61
Freon-22	1.44	51	67
Hexachlorobutadiene	13.81	225	223, 227
Iodomethane	2.88	142	127, 141
iso-Butyraldehyde	3.87	43	41, 72, 39
Isobutyl Alcohol	6.17	43	41, 42, 74
Isopropyl acetate	6.52	43	61, 87
Isopropyl alcohol	2.97	45	43, 44, 59
Isopropyl ether	4.29	45	87, 43
Isopropylbenzene	11.30	105	120
m,p-Xylene	10.63	91	106
Methyl acetate	3.11	43	76
Methyl formate	1.99	60	36
Methyl Methacrylate	7.63	69	41, 100, 39
Methyl tert-butyl ether	3.55	73	57, 41

Methylacrylonitrile	5.28	41	67, 39
Methylcyclohexane	7.41	55	69, 83
Methylcyclopentane	4.95	56	69, 84
Methylene chloride	3.21	84	86, 49
n-Amyl acetate	11.26	43	70, 55, 61
n-Butyl acetate	9.92	43	56, 73
n-Butylbenzene	12.59	91	92, 134
n-Heptane	6.73	43	57, 71, 100
n-Hexane	3.95	57	41, 43, 86
n-Propylbenzene	11.63	91	120
Naphthalene	13.86	128	--
o-Xylene	10.98	91	106
Pentachloroethane	12.08	167	130, 132
Phosgene	5.11	59	63
Propionitrile	5.05	54	52, 55
sec-Butylbenzene	12.19	105	134
Styrene	11.00	104	78
tert-Butyl Alcohol	3.45	59	41, 43
tert-Butylbenzene	12.02	119	91, 134
Tetrachloroethene	9.52	166	168, 129
Tetrahydrofuran	5.48	42	72, 71
Toluene	8.84	92	91
trans-1,2-Dichloroethene	3.55	96	61, 98
trans-1,3-Dichloropropene	9.18	75	77, 110
trans-1,4-Dichloro-2-butene	11.60	124	89, 53
Trichloroethene	7.16	130	95, 97, 132
Trichlorofluoromethane	2.27	101	103, 81, 67
Vinyl acetate	4.20	43	86
Vinyl chloride	1.72	62	64
4-Bromofluorobenzene (SS)	11.52	95	174, 176
Dibromofluoromethane (SS)	5.67	113	--
Toluene-d_8 (SS)	8.76	98	100
Pentafluorobenzene (IS)	5.76	167	--
1,4-Difluorobenzene (IS)	6.79	114	--
Chlorobenzene-d_5 (IS)	10.37	117	--
1,4-Dichlorobenzene-d_4 (IS)	12.31	152	--

Oxygenates

Compound	Retention Time (Minutes)	Primary Ion(s)	Secondary Ion(s)
Ethanol	2.44	45	43, 55
Ethyl tert-butyl alcohol	8.68	59	56, 69, 87
Ethyl tert-butyl ether	4.92	59	57, 41, 87
Isopropyl ether	4.34	45	87, 43
Methyl tert-butyl ether	3.64	73	41, 43, 57
tert-Amyl alcohol	6.47	59	55, 73
tert-Amyl ethyl ether	7.68	59	43, 87, 55
tert-Amyl methyl ether	6.58	73	43, 55, 87
tert-Butyl Alcohol	3.45	59	41, 43
tert-Butyl formate	5.69	41	56
4-Bromofluorobenzene (SS)	11.52	95	174, 176
Dibromofluoromethane (SS)	5.67	113	--
Toluene-d_8 (SS)	8.76	98	100
Pentafluorobenzene (IS)	5.76	167	--
1,4-Difluorobenzene (IS)	6.79	114	--
Chlorobenzene-d_5 (IS)	10.37	117	--
1,4-Dichlorobenzene-d_4 (IS)	12.31	152	--

7.14.6 Internal standard responses. The internal standard responses in all ICVs and CCVs must be evaluated immediately after or during data acquisition. If the peak area for any of the internal standards changes by more than a factor of two (- 50% to + 100%) from that in the mid-point level of the most recent initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made as appropriate. Once corrections have been made, reanalysis of all samples analyzed while the system was malfunctioning is required.

7.14.7 Table 7-7 lists the internal standards with their corresponding analytes that are used for GC/MS volatile analysis.

Table 7-7
Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Pentafluorobenzene	1,4-Difluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene-d_4
1,1,1-Trichloroethane	1,1,2-Trichloroethane	1,1,1,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane
1,1-Dichloroethane	1,1-Dichloropropene	1,2-Dibromomethane	1,2,3-Trichlorobenzene

1,1-Dichloroethene	1,2-Dichloroethane	1,3-Dichloropropane	1,2,3-Trichloropropane
2,2-Dichloropropane	1,2-Dichloropropane	2-Hexanone	1,2,4-Trichlorobenzene
2-Butanone	1,4-Dioxane	4-Bromofluorobenze (SS)	1,2,4-Trimethylbenzene
Acetone	2-Chloroethyl vinyl ether	Bromoform	1,2-Dibromo-3-chloropropane
Acetonitrile	4-Methyl-2-pentanone	Chlorobenzene	1,2-Dichlorobenzene
Acrolein	Benzene	Dibromochloromethane	1,3,5-Trimethylbenzene
Acrylonitrile	Bromodichloromethane	Ethylbenzene	1,3-Dichlorobenzene
Allyl chloride	Carbon tetrachloride	m,p-Xylene	1,4-Dichlorobenzene
Bromochloromethane	cis-1,3-Dichloropropene	o-Xylene	2-Chlorotoluene
Bromomethane	Dibromofluoromethane (SS)	Pentachloroethane	4-Chlorotoluene
Carbon disulfide	Dibromomethane	Styrene	4-Isopropyltoluene
Chloroethane	Ethyl methacrylate	Tetrachloroethene	Bromobenzene
Chloroform	Methyl methacrylate		Hexachlorobutadiene
Chloromethane	Methylcyclohexane		Isopropylbenzene
Chloroprene	n-Amyl acetate		Naphthalene
cis-1,2-Dichloroethene	n-Butyl acetate		n-Butylbenzene
Cyclohexane	Toluene		n-Propylbenzene
Dichlorodifluoromethane	Toluene-d8 (SS)		sec-Butylbenzene
Ethanol	trans-1,3-Dichloropropene		tert-Butylbenzene
Ethyl acetate	Trichloroethene		trans-1,4-Dichloro-2-butene
Freon-113			
Iodomethane			
Isobutyl alcohol			
iso-Butyraldehyde			
Isopropyl acetate			
Isopropyl alcohol			
Isopropyl ether			
Methyl acetate			
Methyl formate			
Methyl tert-butyl ether			
Methylacrylonitrile			
Methylene chloride			
n-Heptane			
n-Hexane			
Propionitrile			
Tetrahydrofuran			
trans-1,2-Dichloroethene			
Trichlorofluoromethane			
Vinyl acetate			
Vinyl chloride			

Oxygenates

Pentafluorobenzene	**1,4-Difluorobenzene**	**Chlorobenzene-d5**	**1,4-Dichlorobenzene-d4**

Ethanol Ethyl tert-butyl alcohol Ethyl tert-butyl ether Isopropyl ether Methyl tert-butyl ether tert-Amyl alcohol tert-Amyl ethyl ether tert-Amyl methyl ether tert-Butyl alcohol tert-Butyl formate	Dibromofluoromethane (SS) Toluene-d_8 (SS)	4-Bromofluorobenzene	

7.15 Data interpretation.

7.15.1 Qualitative analysis.

7.15.1.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method.

7.15.1.2 Clarification on identification and reporting of MtBE (Methyl tert-butyl ether)

7.15.1.2.1 As with any target compound, when MtBE is automatically detected and quanted based on m/z 73 and the qualifier ion m/z 57 is found but outside the expected ration of ±30%, off axis scans of the peak should be examined to determine if pure MtBE can be found and, if it can, the value determined from the manual integration of the area showing pure MtBE should be reported. If no scans can be found with pure MtBE, proceed with the following steps below.

7.15.1.2.1.1 When m/z 73 is present, m/z 57 is present and within expected ion ratios, MtBE is to be reported as quanted on m/z 73 per usual criteria.

7.15.1.2.1.2 When m/z 73 is present, m/z 57 is present, but higher than expected, MtBE is to be reported as quanted on the m/z 73. **Do not Q delete the hit.** This is due to the common interferences from 2- & 3-Methylpentanes which do contribute additional m/z 57 and are constituents in gasoline.

7.15.1.2.1.3 When m/z 73 is present, m/z 57 is not present and MtBE is quanted at **above** the PQL based on m/z 73, the issue

must be forwarded to the TD, QA Manager, or Lab Manager for further review before QAing the data.

7.15.1.2.1.4 When m/z 73 is present, m/z 57 is not present and MtBE is quanted at **below** the PQL based on m/z 73, MtBE is to be reported as quanted on m/z 73 per usual criteria (it will be "J" flagged by LIMS per MDL).

7.15.1.2.2 Clarification on identification and reporting of Trichlorofluoromethane (TCFM).

7.15.1.2.2.1 Ensure any detected hits for TCFM are correctly identified. Freon-141B and Dichlorodifluoromethane elute closely to TCFM and all three compounds have m/z 101, but TCFM will not contain m/z 81 or m/z 67. If m/z 81 or m/z 67 is present, the compound needs to be checked carefully before reporting the hit as TCFM.

7.15.1.3 The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity. Compounds should be identified as present when the criteria below are met.

7.15.2 The intensity patterns of the characteristic ions of a target compound agree with the intensity patterns in the reference spectrum at the same retention time.

7.15.3 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.15.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

7.15.5 Structural isomers that produce very similar mass spectra should be identified as individual analytes if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.15.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles (EICP) of appropriate ions

can aid in the selection of spectra and in the qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum **will** contain extraneous ions contributed by the co-eluting compound.

7.15.7 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

7.15.7.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

7.15.7.2 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20% and 80%.)

7.15.7.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

7.15.7.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or the presence of co-eluting compounds.

7.15.7.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.15.7.6 Computer generated library search routines should not be used. Only after visual comparison of the sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

7.16 Quantitative analysis.

7.16.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte (see Table 7-6).

7.16.2 When linearity exists, calculate the concentration of each identified analyte in the sample as follows:

$$\text{Concentration } (\mu g/L) = \frac{Ax * Qis}{Ais * RFavg * Vo}$$

Where:

Ax =	area of characteristic ion for compound being measured
Q_{Is} =	amount of internal standard injected (ng)
A_{Is} =	area of characteristic ion for the internal standard
RF_{avg} =	mean relative response factor for compound being measured
V_o =	volume of water purged (ml), taking into consideration any dilutions made

7.16.3 Where applicable, an estimate of the concentration for non-calibrated components in the sample should be made. The formula given above should be used with the following modifications: the areas A_x and A_{IS} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine the concentration. Use the nearest internal standard free of interference.

7.17 Sequence Set Up.

7.17.1 Open Enviroquant software.

7.17.2 Develop Log Table.

7.17.2.1 Select “Sequence”, then “Edit sample log table”.

7.17.2.2 Select sample type for each sample.

7.17.2.3 Number all of the samples.

7.17.2.4 Change data file using the following format V7003516, where:

V = volatiles
Number 7 = instrument number
003516 = sequential file number

7.17.2.5 Enter the sample information using the following formats:

MB-8116 = method blank (8116 sequential number from LIMS)
BFB = BFB tune
LCS-8116 = LCS (8116 sequential number from LIMS)
050 = sample concentration
W = sample matrix (water)

Various samples for analysis
Sample weight in grams

7.17.2.6 Enter the method (8260).

7.17.2.7 Save the data in the following directory:

D:\MSDChem\1\Data\010108,

where:

01/01/08 is the date the file is created.

7.17.2.8 Click “Run”, then enter the date of the run. Note that if this is not done, the new analytical batch will overwrite the old batch.

7.17.2.9 Click “Sequence, Position and Run”.

7.17.2.10 Click “Sample”, then click “OK”.

7.17.2.11 Click “Run Sequence”.

7.17.2.12 If the prompt “Process Keywords Before Processing Sequence” appears (it will after modifications are made to existing sequences), click “Yes”.

7.18 Editing Sequences.

7.18.1 Click “File”, then “Edit”.

7.18.2 Sample information can be directly entered into the pop up box that appears.

7.19 Setting up auto-samplers.

7.19.1 Using the left, right, up, and down arrows select Method 01 or 02.

7.19.2 Press “Enter”, then “Edit”.

7.19.3 After each entry, press “Enter”.

7.19.4 The following table indicates the parameters that should be used for operation of the autosampler.

Parameter	Recommended Setting
Method	Either 01 or 02
Sample type	Either soil or water

First and last vial	Enter position number
Sample volume	5 ml
Dilution factor	No
Rinse volume	7 ml
Number of rinses	1
Standard 1 (IS/SS)	Yes
Standard 2	No
Stirring	Yes for soil, No for water
Stir time	0
Settle time	0
Syringe flush	1
Desorb time	2
Cycle timer	0
Average timer	0

7.20 Table 7-8 illustrates the checklist used for data review associated with this test method. Table 7-9 illustrates a calibration curve verification worksheet.

Table 7-8
AES
GC/MS 8260/624 DATA REVIEW CHECKLIST

Batch ID: _____ Run No.:_____

INITIAL RAW DATA REVIEW

QA Analyst

____ ____**:** When reviewing data, be sure to check for carryover in samples that run after a sample with high results for compounds. If a sample is thought to have contamination, then the sample must be re-run to verify results.

____ ____**:** All hits of Acetone, Methylene chloride, and Chloroform should be questioned if not present historically in project and samples are to be re-run as these compounds are common lab contaminants. Soil samples do not need to be re-run for **Acetone** confirmation unless sample is preserved in DI water vials.

Any soil samples containing hits of Carbon disulfide must be re-run for confirmation.

____ ____**:** All hits for Trichlorofluoromethane are to be carefully checked to make sure the hit is correctly identified and not a hit for Freon-141b or Dichlorodifluoromethane.

____ ____**:** Make sure 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, and 1,2-Dichlorobenzene are identified correctly (check retention time on CCV if not sure.)

____ ____**:** All raw data files and run log(s) been printed to pdf and posted to Portal Server (must include all chromatograms, tune files, manual integration files, overlays where applicable)

____ ____**:** All instrument standard IDs included in instrument logbook.

____ ____**:** All tune criteria met.

____ ____**:** Current and approved cal curve used for quantitation.

____ ____**:** All peaks properly integrated with baseline points properly assigned.

____ ____**:** All internal std RTs within 0.5 min of midpoint on ICAL

(No requires CAR if reported to LIMS.)

____ ____**:** CCV internal std areas -50% to +100% of midpoint on ICAL

(No requires CAR if reported to LIMS.)

____ ____**:** All sample internal std areas -50% to +100% of CCV **(No requires CAR if reported to LIMS.)**
____ ____**:** All peaks individually reviewed for spectral match to target with Q delete applied **ONLY** where applicable.
____ ____**:** Background sufficiently low to allow for target identification at the dilution run or data turned off for rerun at higher dilution.
____ ____**:** Sample not over-diluted based on background or data turned off for rerun at lower dilution.
____ ____**:** Check duplicate run data for all samples to ensure no compounds are double reported and dilution values match closely with original run.

GO TO LIMS "MAIN" RUN SCREEN

____ ____**:** All Sample IDs properly assigned per Backlog Report (double click on each SampleID to verify.)
____ ____**:** All Test Codes properly assigned per Backlog Report.
____ ____**:** All Sample Types properly assigned.
____ ____**:** All samples linked to the Prep Batch properly with correct PFac, SpkFac and OFAC.
____ ____**:** All instrument QC run at required frequency.
____ ____**:** All dilution factors entered correctly per the raw data/run logs.
____ ____**:** All Blkref, SPKref, RPDref and CCVref assigned correctly.
____ ____**:** All Comments present are addressed **(May require CAR.)**

GO TO "DATA" SCREEN

____ ____**:** Calculate Sequence to ensure LIMS calculations are complete.
____ ____**:** For 8260 only, do all SPCC cmpds in CCV meet limits for minimum RF **(No requires CAR.)**
____ ____**:** For 8260 only, are all CCC cmpds in CCV all ±20% recovery **(No requires CAR.)**
NOTE: If SPCCs/CCCs are not part of the target analyte list, evaluate using all target analytes per next review step.
____ ____**:** For 8260 and 624, are all target analytes for client samples included in CCV **(No requires CAR.)**
____ ____**:** For 624 only, are all target analytes in CCV within 624 recovery limits **(No requires CAR.)**
____ ____**:** Are there any B flags for target analytes indicating MB hits above PQLs **(Yes requires CAR.)**
____ ____**:** Are there any S and/or R flags for spike cmpds and/or surrogates in LCS **(Yes requires CAR.)**
____ ____**:** Are there any S and/or R flags for spike cmpds and/or surrogates in MS/MSD **(Yes requires CAR.)**
____ ____**:** Are there any S flags for surrogates in samples **(Yes requires CAR.)**
____ ____**:** Are there any H flags present for any samples **(Yes requires CAR.)**
____ ____**:** Are there any E flags for target analytes on samples and/or Batch QC turned on for reporting

(Yes requires CAR.)

____ ____: Are there any J flags on dilutions **(Yes requires running lower dilution.)**

____ ____: Do all LIMS raw values match values on raw data.

____ ____: Have at least 2 sample's final results been manually calculated to verify LIMS calculations.

____ ____: Check all duplicate run data to verify no compounds are double reported and dilution data matches with original run.

PRIOR TO FINAL QA APPROVAL

____ ____: Have all CARs been closed and narratives written prior to QA.

DO NOT USE MATRIX INTERFERENCE NARRATIVES WITHOUT CHROMATOGRAPHIC EVIDENCE FOR MATRIX INTERFERENCE AND/OR PREP COMMENTS INDICATING MATRIX INTERFERENCE UNLESS CONFIRMED BY REEXTRACTION AND REANALYSIS.

____ ____: Have PM, Lab Manager and PM Director been notified for any CAR resulting in reextract and/or due date exceedance.

CAR #: _____ _____ _____ _____ _____

Analyst Signature /Date/Time: _____

Reviewer Signature/Date/Time: _____

Table 7-9
Calibration Curve Review Checklist for 8260B/624

Curve ID_____

1. Does the calibration curve contain a minimum of 5 points. _____
2. If average response factor used in calculation are all non CCC and SPCC compound RSD ≤15% (8260B). _____
3. If response factor is used are all RF RSD <35%. (624) _____
4. If linear regression used in calculation, is the correlation coefficient (r) ≥ 0.995. _____
5. Is the lowest data point in each curve at the PQL in LIMS. _____
   List any analytes with data point above PQL.
   _____
   _____
6. Is the highest data point in each curve at the UQL in LIMS. _____
   List any analytes with data point below UQL.
   _____
   _____

7. Is the peak area for any of the internal standards stay within a factor of two (- 50% to + 100%) of the mid-point standard in the curve. _____
8. Is the relative retention time (RRT) of each target analyte in each calibration. standard within 0.06 RRT units of the other calibration standards. _____
9. Is the average response factor for all CCC compounds within 30% (circle CCC that are above 30%) CCC compounds are: (8260B) _____

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

Comments:_____

10. Is the response factors for the SPCC compounds above 0.050 (Circle SPCC that are below) (8260B) _____

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

Comments:_____

11. Does the ICV pass for 8260B (%D ≤ 20% for CCCs; min RF for SPCC met ; %D ≤20% for all targets). _____
12. Does ICV pass for 624 (all targets within Q Table limits). _____
13. Is ICV second source standard from ICAL standards. _____
14. Is the standard preparation information included and properly signed and dated. _____
15. Is run log included included and properly signed and dated. _____
16. Are all chromatograms included and properly signed and dated. _____

Matrix: _____ Date Prepared: _____ Instrument: _____

Analyst: _____ Analyst Review Date/Time: _____

Supervisor: _____ Supervisor Review Date/Time: _____

Final Reviewer_____ Date/Time: _____

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data generated. Detailed quality assurance procedures can be found in SOP# QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Calibration of the GC/MS. This is accomplished through a 5-point calibration curve. The points on the curve must meet a 15 % RSD or a linear regression coefficient of $\geq$0.995 to determine if the calibration curve is linear. Verification of the curve is also performed, using a second source standard. The response of the calibration verification must be within 15% of the response obtained during the initial calibration.

8.1.3 Retention time window. The retention time for each analyte is evaluated over a 72-hour time period as specified in the method. Daily retention times must fall within specified time windows.

8.1.4 Method Detection Limit Study. The method detection limit is calculated by analyzing at least seven replicates prepared in blank water at 1 to 5 times higher than the estimated detection limit. Quantitation limits are laboratory derived from the MDL study data set. MDL's are to be performed annually or whenever instrument conditions have changes that will affect the established detection limits. Actual MDLs are listed in Tables 5-7 and 5-8 of the AES QA Manual SOP QA-01000.

8.1.5 Method blank. A reagent blank analysis must be performed at the following frequency: every twelve (12) hours and before any samples are analyzed. The concentration of the method blank of any analyte of interest should not exceed the laboratory established practical quantitation limit (PQL).

8.1.6 Surrogate Recovery. All samples, blanks, and QC samples are fortified with surrogate spiking compounds before extraction and injection in order to monitor sample extraction efficiency. The recovery of the surrogate compounds must be within the recovery limits established by the laboratory.

8.1.7 Laboratory Control Sample (LCS). The Laboratory Control Sample (LCS) is used to monitor, assess, and document laboratory method performance and is performed on every batch or twenty samples, whichever occurs more frequently. The recovery of the analytes must meet established laboratory guidelines.

8260 Matrix Spike

Spike Compound	LCS %Recovery Limit
Benzene	70-130%
Chlorobenzene	70-130%
Toluene	70-130%
Trichloroethene	70-130%
1,1-Dichloroethene*	60-140%

*Poor Purge Compound

MBTEX Spike

Spike Compound	LCS %Recovery Limit
Benzene	70-130%
Toluene	70-130%
Ethylbenzene	70-130%
m,p-Xylene	70-130%
o-Xylene	70-130%
Methyl tert-butyl ether	70-130%

Oxygenates Spike

Spike Compound	LCS %Recovery Limit
Ethanol	70-130%
Ethyl tert-butyl ether	70-130%
Isopropyl ether	70-130%
Methyl tert-butyl ether	70-130%
tert-Amyl alcohol	70-130%
tert-Amyl ethyl ether	70-130%
tert-Amyl methyl ether	70-130%
tert-Butyl Alcohol	70-130%
tert-Butyl formate	70-130%
3,3-Dimethyl-1-butanol	70-130%

8.1.8 Sample spike and duplicate spike. Matrix spikes and matrix spike duplicates are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples. The recovery of the analytes must meet established laboratory guidelines.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP #QA-01000, "Quality Assurance Manual," in Section 5. Tables here include corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance, and any procedure deviations in appropriate logbooks.

8.4 All reported results must fall within the instrumental calibration range. LIMS automatically flags data with an "E" qualifier if a reported result exceeds the calibration range. If this type of result is reported, a CAR form must be completed explaining why the sample was not diluted.

8.5 Dilutions must be performed by diluting the sample with Organic-Free DI water when analyzing aqueous samples. Always attempt to dilute samples so that the reported result falls in the mid-range of the calibration curve.

8.6 If the sample is a soil matrix and dilution is required, remove as much sample as available from the ENCORE or other suitable device so as to obtain as representative a sample as possible. Add an equal amount of methanol and analyze the methanolic extract by purge and trap.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 The toxicity or carcinogenicy of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available (i.e., gloves, lab coats, goggles, and dust masks). Reference files of OSHA regulations and MSDS's are available to all personnel involved in the chemical analysis. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

9.2 Work with any of these compounds in high concentrations should be performed in a hood. A NIOSH approved toxic gas respirator should be worn when the analyst handles very high concentrations of these toxic compounds.

9.3 Methanol is a toxic substance whose effects on the body can change daily. It should not be consumed orally as it can cause blindness and death.

9.4 Charcoal traps should be installed on the GC split vents of the instruments with split/splitless injectors to prevent airborne contamination of the work area. All vacuum pumps (Edwards) should contain vapor traps on the outlet pump manifold.

10.0 DATA REPORTING

10.1 The LIMS system automatically rounds the data based upon factors set up for each test category. Typically, the LIMS reports to two significant figures.

10.2 The reporting limits can be changed in LIMS. Various laboratory personnel including project managers have the rights to change the limits per the requirements of the clients.

10.3 Calculation of results using Enviroquant software.

10.3.1 Open Enviroquant software by "clicking" on Icon.

10.3.2 On the pull-down menu, click on "Data Analysis".

10.3.3 On the pull-down menu, click on "Load", then "Data file". The path C:\hpchem\2\data should appear.

10.3.4 Using the "arrow" key, scroll down to the desired sequence number. The sequence number represents the GC number, year, month and day in a format "410321". Note that the left side of the box contains all of the samples in the run. Each sample can be selected by positioning the mouse pointer over the sample and double clicking it.

10.3.5 Once a sample has been selected, on the pull-down menu click on "Load", then "Method". Select the method 8260MA15.M where MA = the month, March and 15 = the date. From this point, the method will remain the same for each sample selection.

10.3.6 To calculate a result, on the pull-down menu, click on "Quant", then "Calculate and generate report". An alternative method is to click on "Quant", then "Int" and "Integrate".

10.3.7 Review the Enviroquant calculated result by clicking on "Quant", then "Q edit". To enlarge the various areas of the chromatogram, place the pointer on the chromatogram and right click the mouse. Drag the mouse over the area to enlarge. Double click the chromatogram to return it to its original size.

10.3.8 Once the chromatogram has been enlarged, the baselines can be redrawn by placing the mouse at one end of the peak and moving it across the bottom while holding down the right mouse. Release the mouse when the line is the correct length.

10.3.9 A general rule for drawing the baseline is that it should be started from the lowest side of the peak and go straight across, so that the baseline makes a "right angle" at the raised side of the peak.

10.3.10 Perform the same procedure on the rest of the chromatograms.

10.4 Completed data is stored in the "C" or local drive of the acquisition computer under the following directory: "C:\HPCHEM\1\DATA\010305" where 01 is the year , 0305 is the date in MM DD format. The Vice President of Technical Operations performs this task.

10.4.1 Prior to moving files for final storage, open the completed data folder and change the status of the data to "frozen" using the following procedure.

10.4.1.1 In Enviroquant, click "Tools", then "Change Data State".

10.4.1.2 Click the radio button next to "Frozen".

10.4.1.3 Click "OK". Exit to save the changes.

10.4.1.4 Using NT Explorer, find the file on the "C' drive in the directory "C:\HPChem\1.

10.4.1.5 Highlight the file and click "Cut".

10.4.1.6 Using NT Explorer, find and select the folder in the computer called "Storage".

10.4.1.7 Locate the proper directory: either MS 3 or MS 4 and click "Save" to save the file to this directory.

10.4.1.8 Periodically, these files are written to a writeable "CD" and stored off site by the VP of Technical Operations. See Section 11.

10.5 Current MDLs for all parameters may be found in Tables in Section 5 of SOP# QA-01000, "Quality Assurance Manual".

11.0 FILE MAINTENANCE

11.1 Data files are printed into electronic .pdf files and stored on the Portal Server under Laboratory, Volatiles, Instrument, Year, and Month. The data contained in the folders consists of the following:

BFB tune(s)
CCV chromatograms and calculated results
MB chromatograms and calculated results
LCS/LCSD chromatograms and calculated results
MS/MSD chromatograms and calculated results

Actual sample chromatograms and calculated results
Daily run log with standard information, pH checks, Chlorine checks, and analyst signature
Data review checklists

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto writeable CD-ROM disks. Two copies are made. One copy of the disks are stored on the laboratory premises and the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument Maintenance logbooks: Instrument Maintenance logbooks must be completed each time any maintenance is performed on the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of the GC/MS, cleaning the ion source. It also includes any non-routine maintenance that may be performed such as replacement of motors in tower autosamplers.

Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.2 Routine Maintenance: Typical routine maintenance consists of keeping the system clean and insuring that the chromatography remains acceptable. Examples of dirty injection ports would be peak tailing and the degradation of DDT and Dieldrin in pesticide analysis. The table below indicates the frequency of routine maintenance for various instrument types within the laboratory.

Maintenance Action	Recommended Frequency
Changing injection port liners	Weekly or when chromatography is affected
Trimming column	Monthly or when chromatography is affected
Cleaning GC/MS source	Quarterly or when chromatography is affected
Changing GC/HPLC Column	Annually or when other attempts to resolve chromatography fail

12.3 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as an autosampler that will not function. If this type of problem occurs, contact the department manager for assistance.

13.0 METHOD PERFORMANCE

13.1 The method detection limit (MDL) is defined in 40CFR Part 136, Appendix B as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The reporting limit (RL) is defined as the concentration of a substance that is above the level of uncertainty.

13.2 The concentrations listed in tables in Section 5 of SOP# QA-01000, "Quality Assurance Manual", were obtained using reagent water. Similar results can be achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

13.3 This method is recommended for use in the concentration range from the MDL to 100 x MDL.

13.4 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 8.0 to 500 µg/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and, essentially, independent of the sample matrix. Table 13-1 lists single operator precision and accuracy found in SW-846, Method 8260B.

Table 13-1
Single Operator Precision and Accuracy-Method 8260B

Compound	Range for Q (µg/L)	Limit for s(µg/L)	Range for X (µg/L)	Range for P, Ps (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-142	D-242
Carbon tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
Chloroethane	7.6-32.4	11.4	8.4-40.4	14-230
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190

Table 13-1 (Continued)
Single Operator Precision and Accuracy-Method 8260B

Compound	Range for Q (µg/L)	Limit for s(µg/L)	(µg/L)	Range for X Ps (%)	Range for P,
1,1-Dichloroethane	14.5-25.5	5.1		14.2-28.5	59-155
1,2-Dichloroethane	13.6-26.4	6.0		14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1		3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7		13.6-28.5	54-156
1,2-Dichloropropane	6.8-33.2	13.8		3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8		3.8-36.2	D-210
trans-1,3-Dichloropropene	10.0-30.0	10.4		7.6-32.4	17-183
Ethyl benzene	11.8-28.2	7.5		17.4-26.7	37-162
Methylene chloride	12.1-27.9	7.4		D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4		13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0		17.0-26.6	64-148
Toluene	14.9-25.1	4.8		16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6		13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5		14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6		18.6-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0		8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0		D-43.5	D-251

Q = Concentration measured in QC check sample, in µg/L
S = Standard deviation of four recovery measurements, in µg/L
X = Average recovery of four recovery measurements, in µg/L
P, Ps = Percent recovery measured as average recovery/concentration in check sample.
D = Detected; result must be greater than zero.
* Criteria were calculated assuming a QC check sample concentration of 20 µg/L

13.5 Recovery limits for surrogates used in this method are based on historical data. The limits are updated frequently and can be found in the LIMS and in tables in Section 5 of SOP# QA-01000, "Quality Assurance Manual".

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates waste. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 SOP WM-17001 Waste Disposal and SOP SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade – A dry chemical that has been dried at 250°C for 4 hours, cooled, and stored in a desiccator.

15.2 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to distilled water or clean soil and the concentration is measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.3 DI water - Deionized water

15.4 RSD – Relative Standard Deviation

15.5 RF – Response factor. Determined as the concentration of a sample divided by the chromatographic area of the peak produced by the sample.

15.6 MS - Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.7 MSD - Matrix Spike Duplicate.

15.8 CCV - Continuing calibration verification standard.

15.9 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot than the initial calibration standards used.

15.10 LCSD - Laboratory Control Sample Duplicate.

15.11 BFB Tune – A comparison of the mass fragments of p-bromofluorobenzene after it has been

injected into a GC/MS. The ratios of the various mass fragments must comply with certain criteria.

15.12 Tuning a GC/MS. The physical procedure in which instrument conditions are varied so that the BFB injection will pass the method criteria.

15.13 m/z – mass to charge ratio.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, Physical Chemical Methods, April 1998, Method 8000B,"Determinative Chromatographic Separations", Revision 2, December 1996.

16.2 Test Methods for Evaluating Solid Waste, Physical Chemical Methods, April 1998, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)", Revision 2, December 1996.

16.3 Test Methods for Evaluating Solid Waste, Physical Chemical Methods, April 1998, Method 5000, "Sample Preparation for Organic Volatile Compounds", Revision 0, December 1996.

16.4 Test Methods for Evaluating Solid Waste, Physical Chemical Methods, April 1998, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December 1996.

16.5 Test Methods for Evaluating Solid Waste, Physical Chemical Methods, April 1998, Method 5035, "Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples", Revision 0, December 1996.

16.6 Test Methods for Evaluating Solid Waste, Physical Chemical Methods, April 1998, Method 3585, "Waste Dilution for Volatile Organics", Revision 0, December 1996.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://portal/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://portal/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

APPROVAL OF ATTACHED DOCUMENT FOR IMPLEMENTATION

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DOCUMENT CONTROL NUMBER: Rev. 9

DOCUMENT DISTRIBUTION NUMBER: OA-11011

ELECTRONIC DOCUMENT LOCATION
AES Portal Server: http://portal/Procedures/Standard Operating Procedures

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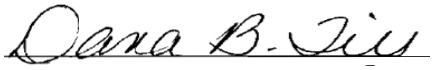
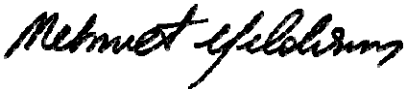
Technical Director:  Date: 2/13/2012

Laboratory Manager:  Date: 2/13/2012

Quality Assurance Manager:  Date: 2/13/2012

Department Supervisor:  Date: 2/13/2012

STANDARD OPERATING PROCEDURE FOR
SEMIVOLATILE ORGANICS BY SW-846 GC/MS METHOD 8270D
PREP METHODS 3510C/3535A/3540C/3550C/3580A

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water.

1.2 This procedure can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include PAH, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitroamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1-1 for a list of compounds and their characteristic ions that can be analyzed by the specified GC/MS system.

1.3 The following compounds may require special treatment when being determined by this method.

1.3.1 Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor.

1.3.2 Under the alkaline conditions of the extraction step, α-BHC, γ-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.

1.3.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.3.4 N-nitrosodimethylamine is difficult to separate form the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated form diphenylamine.

1.3.5 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6,-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid. 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling point material.

1.4 The reporting limit of Method 8270D for determining an individual compound is approximately 1 mg/Kg (wet weight) for soil/sediment samples, 1-200 mg/Kg (wet weight) for wastes, and 10 ug/L for ground water (see the tables in the QA Manual). The reporting limits will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.6 This method is used to analyze for the following compounds. Not all of the compounds will be reported in any given instance.

Table 1-1
RETENTION TIMES AND CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention[a] Time (min.)	Primary Ion	Secondary Ion (s)	Minimum Response Factor (RF) for most common target ions
1,1-Biphenyl		154	153,76	0.010
1,2,4,5-Tetrachlorobenzene	6.62	216	214, 179, 108, 143, 218	0.010
1,2,4-Trichlorobenzene	5.49	180	182, 145	
1,2-Dichlorobenzene	4.12	146	148, 111	
1,2-Diphenylhydrazine	8.43	77	105, 182	
1,3,5-Trinitrobenzene	8.92	75	74, 213, 120, 91, 63	
1,3-Dichlorobenzene	3.81	146	148, 111	
1,3-Dinitrobenzene	7.10	168	76, 50, 75, 92, 122	
1,4-Dichlorobenzene	3.92	146	148, 111	
1,4-Dichlorobenzene-d_4 (IS)	3.98	152	150, 115	
1,4-Naphthoquinone	7.29	158	104, 102, 76, 50, 130	
1-Chloronaphthalene	7.00	162	127, 164	
1-Methylnaphthalene	6.52	142	141	
1-Naphthylamine	8.05	143	115, 89, 63	
2,3,4,6-Tetrachlorophenol	8.03	232	131, 230, 166, 234, 168	0.010
2,4,5-Trichlorophenol	6.87	196	198, 97, 132, 99	0.200
2,4,6-Tribromophenol (surr.)	8.51	330	332, 141	
2,4,6-Trichlorophenol	6.81	196	198, 200	0.200
2,4-Dichlorophenol	5.45	162	164, 98	0.200
2,4-Dimethylphenol	5.26	122	107, 121	0.200
2,4-Dinitrophenol	7.79	184	63, 154	0.010
2,4-Dinitrotoluene	7.92	165	63, 89	0.200
2,6-Dinitrotoluene	7.46	165	63, 89	0.200
2-Acetylaminofluorene	12.02	181	180, 223, 152	
2-Chloronaphthalene	6.98	162	127, 164	0.800
2-Chlorophenol	3.66	128	64, 130	0.800
2-Fluorobiphenyl (surr.)	6.87	172	171	
2-Fluorophenol (surr.)	2.44	112	64	
2-Methylnaphthalene	6.41	142	141	0.400
2-Methylphenol	4.39	107	108, 77, 79, 90	0.700
2-Naphthylamine	7.86	143	115,116	
2-Naphthylamine	7.96	143	115, 116	
2-Nitroaniline	7.17	65	92, 138	0.010
2-Nitrophenol	5.11	139	109, 65	0.100

Compound	Retention[a] Time (min.)	Primary Ion	Secondary Ion (s)	Minimum Response Factor (RF) for most common target ions
2-Picoline	1.81	93	66, 92	
3,3'-Dichlorobenzidine	12.21	252	126	0.010
3,3'-Dimethylbenzidine	11.72	212	106, 196, 180	
3-Methylcholanthrene	13.93	268	252, 253, 126, 134, 113	
3-Methylphenol	4.62	107	108, 77, 79, 90	
3-Nitroaniline	7.65	138	108, 92	0.010
4,6-Dinitro-2-methylphenol	8.37	198	51, 105	0.010
4-Aminobiphenyl	9.11	169	168, 170, 115	
4-Bromophenyl phenyl ether	8.80	248	250, 141	0.100
4-Chloro-3-methylphenol	6.40	107	144, 142	0.200
4-Chloroaniline	5.71	127	129, 65, 92	0.010
4-Chlorophenyl phenyl ether	8.26	204	206, 141	0.400
4-Methylphenol	4.62	107	108, 77, 79, 90	0.600
4-Nitroaniline	8.36	138	65, 108, 92, 80, 39	0.010
4-Nitrophenol	7.98	139	109, 65	0.010
4-Nitroquinoline-1-oxide	10.30	174	101, 128, 75, 116	
5-Nitro-o-toluidine	8.40	152	77, 79, 106, 94	
7,12-Dimethylbenz(a)anthracene	13.39	256	241, 239, 120	
a,a-Dimethylphenethylamine	5.77	58	91, 65, 134, 42	
Acenaphthene	7.64	154	153, 152	0.900
Acenaphthene-d_{10} (IS)	7.60	164	162, 160	
Acenaphthylene	7.44	152	151, 153	0.900
Acetophenone	4.50	105	71, 51, 120	0.010
Allethrin	10.73	123	79, 107, 136	
Aniline	3.51	93	66, 65	
Anthracene	9.35	178	176, 179	0.700
Aramite	11.17	185	191, 319, 334, 197, 321	
Atrazine	9.07	200	173,215	0.010
Baygon	8.49	110	152	
Benz(a)anthracene	12.18	228	229, 226	0.800
Benzaldehyde	3.36	77	105,106	0.010
Benzidine	10.83	184	92, 185	
Benzo(a)pyrene	13.61	252	253, 125	0.700
Benzo(b)fluoranthene	13.31	252	253, 125	0.700
Benzo(g,h,i)perylene	14.79	276	138, 277	0.500
Benzo(k)fluoranthene	13.33	252	253, 125	0.700
Benzoic acid	5.52	122	105, 77	
Benzyl alcohol	4.18	108	79, 77	
Bis(2-chloroethoxy)methane	5.35	93	95, 123	0.300
Bis(2-chloroethyl) ether	3.62	93	63, 95	0.700
Bis(2-chloroisopropyl) ether	4.35	45	77, 121	0.010
Bis(2-ethylhexyl)phthalate	12.32	149	167, 279	0.010

Compound	Retention[a] Time (min.)	Primary Ion	Secondary Ion (s)	Minimum Response Factor (RF) for most common target ions
Butyl benzyl phthalate	11.66	149	91, 206	0.010
Carbazole	9.99	167	139	0.010
Caprolactam	6.19	113	55,56	0.010
Carbaryl	9.76	144	115, 116, 201	
Chlorobenzilate	11.39	251	139, 253, 111, 141	
Chrysene	12.23	228	226, 229	0.700
Chrysene-d_{12} (IS)	12.19	240	120, 236	
Diallate (cis or trans)	8.81	86	234, 43, 70	
Diazinon	9.41	137	179, 199, 304	
Dibenz(a, j)acridine	14.45	279	280, 277, 250	0.400
Dibenz(a,h)anthracene	14.60	278	139, 279	
Dibenzofuran	7.84	168	139	0.800
Diethylphthalate	8.19	149	177, 150	0.010
Dimethoate	9.05	87	93, 125, 143, 229	
Dimethylaminoazobenzene	11.24	225	120, 77, 105, 148, 42	
Dimethylphthalate	7.39	163	194, 164	0.010
Di-n-butyl phthalate	10.01	149	150, 104	0.010
Di-n-octyl phthalate	13.00	149	167, 43	0.101
Diphenylamine	8.70	169	167	
Disulfoton	9.41	88	97, 89, 142, 186	
Dursban	10.31	97	197, 314, 29	
Ethyl methanesulfonate	3.05	79	109, 97, 45, 65	
Famphur	11.67	218	125, 93, 109, 217	
Fenvalerate	13.94	125	167, 225, 419	
Fluoranthene	10.60	202	101, 203	0.600
Fluorene	8.22	166	165, 167	0.900
Hexachlorobenzene	8.83	284	142, 249	0.100
Hexachlorobutadiene	5.74	225	223, 227	0.010
Hexachlorocyclopentadiene	6.59	237	235, 272	0.050
Hexachloroethane	4.53	117	201, 199	0.300
Hexachlorophene	13.75	196	198, 209, 211, 406, 408	
Hexachloropropene	5.74	213	21, 215, 117	
Indeno(1,2,3-c,d)pyrene	14.59	276	138, 227	0.500
Isodrin	10.49	193	66, 195, 263, 265, 147	
Isophorone	5.02	82	95, 138	0.200
Isosafrole	7.04	162	131, 104, 77, 51	
Kepone	11.65	272	274, 237, 178, 143, 270	
Methapyrilene	10.39	97	50, 191, 71	
Methyl methanesulfonate	2.27	80	80, 79, 65, 95	
Methyl parathion	9.83	109	125, 263, 79, 93	

Compound	Retention[a] Time (min.)	Primary Ion	Secondary Ion (s)	Minimum Response Factor (RF) for most common target ions
MGK-264	10.53	164	66, 111, 210	
Naphthalene	5.56	128	129, 127	0.700
Naphthalene-d_8 (IS)	5.54	136	68	
Nitrobenzene	4.70	77	123, 65	0.200
Nitrobenzene-d_5 (surr.)	4.68	82	128, 54	
N-Nitrosodibutylamine	6.14	84	57, 41, 116, 158	
N-Nitrosodiethylamine	2.72	102	42, 57, 44, 56	
N-Nitrosodimethylamine	1.21	42	74, 44	
N-Nitrosodi-n-propylamine	4.53	70	42, 101, 130	0.500
N-Nitrosodiphenylamine	8.42	169	167	0.010
N-Nitrosomethylethylamine	2.02	88	42, 88, 43, 56	
N-Nitrosomorpoline	4.64	56	86,116	
N-Nitrosopiperidine	4.92	114	42, 55, 56, 41	
N-Nitrosopyrrolidine	4.59	100	41, 42, 68, 69	
o-Toluidine	4.62	106	107, 77, 51, 79	
Parathion	10.27	109	97, 291, 139, 155	
Pentachlorobenzene	7.80	250	252, 108, 248, 215, 254	
Pentachloronitrobenzene	9.09	237	142, 214, 249, 295, 265	
Pentachlorophenol	9.11	266	264, 268	0.050
Perylene-d_{12} (IS)	13.66	264	260, 265	
Phenacetin	8.84	108	179, 109, 137, 80	
Phenanthrene	9.29	178	179, 176	0.700
Phenanthrene-d_{10} (IS)	9.26	188	94, 80	
Phenol	3.61	94	65, 66	0.800
Phenol-d_6 (surr.)	3.59	99	42, 71	
Phorate	8.82	75	121, 97, 93, 260	
Piperonyl butoxide	12.08	176	177, 149, 338	
Pronamide	9.22	173	175, 145, 109, 147	
Pyrene	10.85	202	200, 203	0.600
Pyrethrins	11.72	123	162, 105	
Resmethrin	12.10	123	171, 338	
Safrole	6.43	162	104, 77, 103, 135	
Terphenyl-d_{14} (surr.)	11.07	244	122, 212	
Tetraethyl dithiopyrophosphate (sulfotep)	8.82	202	97, 322	
Tetramethrin	12.34	164	123, 79	
Thionazine	8.35	107	96, 97, 143, 79, 68	

IS = Internal standard.
surr. = surrogate.
[a] Estimated retention times.

2.0 SUMMARY OF METHOD

2.1 Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract and for their qualitative and quantitative analysis by mass spectrometry.

2.2 This procedure includes specific calibration and quality control steps that replace the general requirements in SW-846 Method 8000B.

Table 2-1
SAMPLE PREPARATION METHODS

Matrix	**Extraction Method Code***
Water	3510C, 3535A
Soil/Sediment	3540C, 3550C
Waste	3540C, 3550C, 3580A
***Method Code**	**Description**
3510C	Separatory Funnel Liquid-Liquid Extraction
3535A	Solid Phase Extraction
3540C	Soxhlet Extraction
3550C	Ultrasonic Extraction
3580A	Waste Dilution

3.0 INTERFERENCES

3.1 Method interferences may be caused by contamination in solvents, reagents, glassware, and other sample processing hardware. Method interferences may lead to discrete artifacts and/or elevated baseline in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by analyzing laboratory reagent blanks.

3.2 Interferences by phthalate esters can pose a major problem in Semi-Volatile analysis that lead to discrete artifacts and/or elevated baseline in the total ion current (TICs) profiles. These compounds generally appear in the chromatogram as large eluting peaks, common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operation. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent wetted surfaces are handled. Avoiding the use of plastics in the laboratory can best minimize interferences from phthalates. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination. High temperature baking of glassware is effective in insuring phthalate free glassware.

3.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. The extraction analyst should make note of any physical observations before, during and after

the extraction process that would indicate that additional clean-up techniques might be needed to minimize interfering substances in the final extract prior to analysis.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

4.1 Extraction and analysis holding Times are listed in Table 4-1 below:

Table 4-1
Extraction and Analysis Holding Times

Matrix	Extraction Holding Time	Analysis Holding Time
Aqueous	7 days from collection	40 days from extraction
Solids	14 days from collection	40 days from extraction
Waste	14 days from collection	40 days from extraction

4.2 All samples must be refrigerated at 4°C ± 2°C from the time of collection until extraction.

4.3 Store all extracts at 4°C ± 2°C in the dark in Teflon-sealed containers until all analyses are performed.

5.0 REAGENTS AND STANDARDS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Stock solutions - Stock solutions are prepared from pure standard materials or purchased as certified solutions as described in Table 5-7. Stock solutions are good for 6 months. Working and intermediate must not be assigned an expiration date past the expiration date of the stock solution.

5.3 1-Methylnaphthalene Stock Solution, 10000 mg/l. Prepare by dissolving 500 mg of the neat compound (Table 5-7 Ref No. 8) in 50 ml of methylene chloride.

5.4 1-Methylnaphthalene Second Source Stock Solution, 10000 mg/l. Prepare by dissolving 500 mg of the neat compound (Table 5-7 Ref No. 21) in 50 ml of methylene chloride.

5.5 Carbazole Stock Standard, 10000 mg/l. Prepare by dissolving 100 mg of the neat compound (Table 5-7 Ref No. 10) in 10 ml of methylene chloride.

5.6 Carbazole Second Source Stock Standard, 10000 mg/l. Prepare by dissolving 100 mg of the neat compound (Table 5-7 Ref No. 23) in 10 ml of methylene chloride.

5.7 Surrogate Spiking Solution, 50 mg/l B/N and 100 mg/l Acid fraction. Prepare by diluting 1

ml of each surrogate mix (Table 5-7 Ref Nos. 11 and 12) to 100 ml in methanol.

5.8 PAH SIM Surrogate Spiking Solution, 2.0 mg/l B/N. Prepare by diluting 40 µL of surrogate mix (Table 5-7 Ref No.11) to 100 ml in methanol.

5.9 Surrogate Spiking Solution for waste samples 250 mg/l B/N and 500 mg/l Acid fraction. Prepare by diluting 5 ml of each surrogate mix (Table 5-7 Ref Nos. 11 and 12) to 100 ml in methylene chloride.

5.10 Appendix II Intermediate Solutions for calibration curve, 200µg/ml: 1ml of each standard Table 5-7, Ref. Nos. 29, 30, 31 and 32 (2000 µg/ml), and 2 ml of 1000 µg/ml 7,12-Dimethylbenz[a]anthracene standard (Table 5-7 Ref. No.89) are diluted to 10ml with methylene chloride. This mixture is used to make appendix II calibration curve as described in Table 5-1.

5.11 Appendix II second source Intermediate Solutions for calibration curve, 200µg/ml: 1ml of each standard Table 5-7, Ref. Nos. 25, 26, 27 and 28 (2000 µg/ml), and 2 ml of 1000 µg/ml 7,12-Dimethylbenz[a]anthracene second source standard are diluted to 10ml with methylene chloride. This mixture can also be used to make appendix II calibration curve as described in Table 5-1.

7,12-Dimethylbenz[a]anthracene (second source) stock standard, 1000ug/ml, Prepare by dissolving 10mg of the neat standard (Table 5-7, Ref. No. 90 or 92) in 10ml flask with Methylene chloride.

5.12 SVOC Intermediate Solution for calibration curve, 200ug/ml: using following standards to make exactly 10ml solution with Methylene chloride.

1ml of each standard in Table 5-7, Ref. No. 1, 2, 3, 4, 5, 6, 7 (2000 ug/ml),
200ul of 10, 000 ug/ml Pyridine stock (see below to make this stock),
200ul of 10, 000 ug/ml 1-Methylnaphthalene stock (5.3),
200ul ug/ml of 10, 000 ug/ml Carbazole stock (5.5),
400ul of B/N surrogate (Table 5-7, Ref. No. 11),
200ul of Acid surrogate (Table 5-7, Ref. No. 12),
200ul of 10, 000 ug/ml 3-Methylphenol (see below to make this stock),
200ul of 10, 000 ug/ml Ethyl methanesulfonate stock (see blow to make this stock),
200ul of 10, 000ug/ml Methylmethanesulfonate stock (see below to make this stock), and 1ml of Custom Made Standard (2000ug/ml each, Table 5-7, Ref. No. 55).

This standard mixture is used to make 8270 calibration curve as described in Table 5-2.

Pyridine Stock Standard, 10, 000 mg/L, Prepare by dissolving 100mg of the neat standard (Table 5-7, Ref. No. 54) in 10ml flask with Methylene chloride.

3-Methylphenol Stock Standard, 10, 000 mg/L, Prepare by dissolving 100mg of the neat standard (Table 5-7, Ref. No. 91) in 10ml flask with Methylene chloride.

Ethyl methanesulfonate Stock Standard, 10, 000 mg/L, Prepare by dissolving 100mg of the neat standard (Table 5-7, Ref. No. 73) in 10ml flask with Methylene chloride.

Methylmethanesulfonate Stock Standard, 10, 000 mg/L, Prepare by dissolving 100mg of the neat standard (Table 5-7, Ref. No.74) in 10ml flask with Methylene chloride.

5.13 SVOC second source Intermediate Solution for calibration curve, 200ug/ml: using following standards to make exactly 10ml solution with Methylene chloride.

1ml of each standard in Table 5-7, Ref. No. 13, 14, 15, 16, 17, 18, 19 (2000ug/ml),
200ul of 10, 000 ug/ml 7 compounds mixture stock (see below to make this stock),
400ul of B/N surrogate (Table 5-7, Ref. No. 11),
200ul of Acid surrogate (Table 5-7, Ref. No. 12), and
400ul of 3-Methylphenol (Table 5-7, Ref. No. 56).

This standard mixture can also be used to make 8270 calibration curve as described in Table 5-2.

BNA 7 compound mixture Stock Standard, 10, 000 mg/L, Prepare by dissolving 100mg of each neat standard (Table 5-7, Ref. No. 57, 58, 59, 60, 62, 63, and 64) in 10ml flask with Methylene chloride.

5.14 Internal Standard Solutions, purchased directly from Restek. The internal standards are 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, and Perylene-d_{12}. The internal standards are added directly to each mixture as indicated in the subsequent reagent preparatory procedures.

5.15 Calibration standards - Prepare calibration standards at a minimum of five concentration levels for each analyte of interest. The lowest standard should be at a concentration equal to the LIMS reporting limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples but should not exceed the working range of the GC/MS system. Prepare initial calibration standards at concentration levels shown on the following table.

Table 5-1
Appendix 2 Calibration Standards

Volume of App 2 Intermediate Std. Soln (µl)	Volume of Internal Std. Spike Soln. (Ref. No 22) (µl)	Volume of $MeCl_2$ (µl)	Final Std. Conc. (ng/µl)
800	10	200	160
600	10	400	120
400	10	600	80
300	10	700	60
200	10	800	40
100	10	900	20
50	10	950	10

Table 5-2
Calibration Standards for 8270D Semivolatile Analysis

Intermediate Solution Conc. (ng/µl)	Volume of Intermediate Solution (µl)	Volume of Internal Std. Spike Soln. (Ref. No 22) (µl)	Volume of $MeCl_2$ (µl)	Final Volume (µl)	Final Std. Conc. (ng/µl)
200	800	10	200	1000	160
200	600	10	400	1000	120
200	400	10	600	1000	80
200	300	10	700	1000	60
200	200	10	800	1000	40
200	100	10	900	1000	20
200	50	10	950	1000	10

Table 5-3
Calibration Standards for 8270D PAH SIM Semivolatile Analysis

Intermediate Solution Conc. (ng/µL)	Volume of Intermediate Solution (µL)	Volume of Internal Std. Spike Soln. (Conc=100 ng/µL) (µL)	Volume of $MeCl_2$ (µL)	Final Volume (µL)	Final Std. Conc. (ng/ µL)
10	1000	10	0	1000	10.0
10	750	10	250	1000	7.5
10	500	10	500	1000	5.0
10	350	10	650	1000	3.5
10	200	10	800	1000	2.0
10	100	10	900	1000	1.0
10	10	10	990	1000	0.1
10	5	10	995	1000	0.05

5.16 DFTPP standard, 50 ug/ml solution. Prepared by diluting 0.5ml of Table 5-7 Ref. No 46 to 10ml in Methylene Chloride.

5.17 Special Pesticide Stock Compounds. Purchased separately as neat solutions. See Table 5-7.

5.18 Special Pesticide Stock Solution. Prepare by accurately weighing 10 mg each of Table 5-7 Ref. Nos. 33,34,35,36,37,38,39,40,41,42,43,44 and 45 and diluting to 50 ml with methylene chloride. The final concentration will be 200 mg/l of each pesticide. The pesticides in the mix are as follows:

Allethrin	MGK-264	Fenvalerate
Tetramethrin	Sumithrin	Resmethrin
Pyrethrins	Carbaryl	Dursban
Diazinon	Piperonyl butoxide	Baygon
Dazomet		

5.19 Special Pesticide Calibration standards - Prepare calibration standards at a minimum of five concentration levels for each analyte of interest as indicated in Table 5-3 below.

Special Pesticide Calibration Standards for 8270D Semivolatile Analysis

Volume of Special Pesticide Stock Soln. (5.13) (µl)	Volume of $MeCl_2$ (µl)	Final Volume (µl)	Final Std. Conc. (ng/µl)
1000	0	1000	200
750	250	1000	150
500	500	1000	100
250	750	1000	50
125	875	1000	25
50	950	1000	10

5.20 Matrix spiking/LCS standards

5.20.1 The matrix spike/LCS solutions are purchased from Restek as Base-Neutrals and Acid fractions. They are prepared for use by the dilution of 2.0 ml of the base neutral mixture (Table 5-7 Ref. No. 53) and/or 1 ml of the acid mixture (Table 5-7 Ref. No. 51) and diluting to 100 ml in methanol. The final concentrations and compounds in the mixes are indicated in Table 5-5 below.

Table 5-5
Matrix Spiking Standards

Base/Neutrals (100 µg/ml)	**Acids (100 µg/ml)**
1,2,4-trichlorobenzene	pentachlorophenol
acenaphthene	phenol
2,4-dinitrotoluene	2-chlorophenol
pyrene	4-chloro-3-methylphenol
N-nitroso-di-n-propylamine	4-nitrophenol
1,4-dichlorobenzene	

5.20.2 BNA LCS Extended Spiking Solution (Labeled as LCSSC in LIMS), Compounds listed in APPENDIX I. This spike is to be used for all SC samples. Spiking solution is prepared by making two separate 5 mL stock that will be added together during the extraction, Mix A and B.

5.20.2.1 Mix A. Prepared by adding 125uL of the Calibration Check (#95), 1mL of each of Base/Neutral Extractables Mixes 1 and 2 (#96 and #97), and 50uL of SVOC GC/MS Mix 4B (#98) to 5mL volumetric flask and dilute with Methanol. Final concentrations will vary but consist of 50, 100, and 150ug/mL.

5.20.2.2 Mix B. Prepared by adding 500uL of each individual analyte, #99 through #108, to a 5 mL volumetric flask and dilute with Methanol. Final concentration of each analyte is 100ug/mL.

5.21 PAH Spike Solution, 50 mg/l. Prepare by diluting 2.5 ml of PAH Stock solution (Table 5-7 Ref. No. 93) to 100 ml in Methanol.

5.22 PAH SIM Spike Solution, 10 mg/l. Prepare by diluting 500 µL of PAH Stock solution (Table 5-7 Ref. No. 93) to 100 ml in Methanol.

5.23 Methylene chloride, CH_2Cl_2. Pesticide quality or equivalent. Store apart from other solvents.

5.24 Methanol, CH_3OH. Pesticide quality or equivalent. Store apart from other solvents.

5.25 Sodium Sulfate, granular, anhydrous. Purify by heating to 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

5.26 Famphur Stock Standard, 10,000 mg/L, Prepare by dissolving 100mg of the neat standard (Table 5-7, Ref. No. 93) to make exactly 10ml solution in a 10ml flask with Methylene chloride.

5.27 Kepone Stock Standard, 10,000 mg/L, Prepare by dissolving 100mg of the neat standard
(Table 5-7, Ref. No. 94) to make exactly 10ml solution in a 10ml flask with Methylene chloride.

5.28 Kepone and Famphur mixture stock standard, 200 mg/L, 0.2ml of above Famphur (5.23) and Kepone (5.24) stock standards to make 10ml solution in a 10ml flask with Methylene chloride. This mixture is used to make Kepone-Famphur calibration curve as described in Table 5-5.

Table 5-6
Kepone-Famphur Calibration Curve Standards

Concentration Level (ng/µl)	**200mg/L stock used (µl)**	**$MeCl_2$ (µl)**	**Internal Standard used (µl)**
10	50	950	10
25	125	875	10
50	250	750	10
100	500	500	10
150	750	250	10
200	1000	0	10

5.29 Kepone and Famphur ICV standards, Prepare three levels as described in Table 5-6 using Ultra Semi-Volatile Mixture #7 standard (Table 5-7, Ref. No. 25).

Table 5-7
Kepone-Famphur ICV Standards

Concentration Level (ng/µl)	SV mixture 7 used (µl)	$MeCl_2$ (µl)	Internal Standard used (µl)
50	25	975	10
100	50	950	10
150	75	925	10

5.30 Vendor List. The standards used in this test are purchased using the vendors and catalog numbers in Table 5-7.

Table 5-8
8270D Standards and Chemicals

Reference Number	Standard Name	Vendor Name	Concentration	Catalog Number
1	Semi-Volatile Mixture #1	Ultra Scientific	2000 µg/ml	SVM-120A-1
2	Semi-Volatile Mixture #2	Ultra Scientific	2000 µg/ml	SVM-121-1
3	Semi-Volatile Mixture #3	Ultra Scientific	2000 µg/ml	SVM-122-1
4	Semi-Volatile Mixture #4	Ultra Scientific	2000 µg/ml	SVM-123-1
5	Semi-Volatile Mixture #5	Ultra Scientific	2000 µg/ml	SVM-124-1
6	Semi-Volatile Mixture #6	Ultra Scientific	2000 ug/ml	SVM-125-1
7	Semi-Volatile Mixture #9	Ultra Scientific	2000 µg/ml	SVM-128-1
8	1-Methylnaphthalene	Ultra Scientific	Neat (500mg)	RAH-044
9	Pyridine	Restek	2000 µg/ml	30409
10	Carbazole	Ultra Scientific	Neat (100mg)	HAH-022
11	B/N Surrogate	Restek	5000 µg/ml	31062
12	Acid Surrogate	Restek	10000 µg/ml	31063
13	Semi-Volatile Standard	AccuStandard	2000 µg/ml	M-8270-01
14	Semi-Volatile Standard	AccuStandard	2000 µg/ml	M-8270-02
15	Semi-Volatile Standard	AccuStandard	2000 µg/ml	M-8270-03
16	Semi-Volatile Standard	AccuStandard	2000 µg/ml	M-8270-04A
17	Semi-Volatile Standard	AccuStandard	2000 µg/ml	M-8270-04B
18	Semi-Volatile Standard	AccuStandard	2000 µg/ml	M-8270-05
19	Semi-Volatile Standard	AccuStandard	2000 µg/ml	M-8270-06
20	Pyridine	AccuStandard	2000 µg/ml	APP-9-186-20X
21	1-Methylnaphthalene	AccuStandard	2000 µg/ml	H-001S-D-40X
22	SV internal standard	Restek	4000 µg/ml	31006-510
23	Carbazole	Aldrich	Neat (1 g)	C310-3
24	8270 Calibration Mix # 5	Restek	2000 µg/ml	31622
25	Semi-Volatile Mixture #7	Ultra Scientific	2000 µg/ml	SVM-126-1
26	Semi-Volatile Mixture #8	Ultra Scientific	2000 µg/ml	SVM-127-1
27	Semi-Volatile Mixture #10	Ultra Scientific	2000 µg/ml	SVM-129-1
28	Semi-Volatile Mixture #11	Ultra Scientific	2000 µg/ml	SVM-131-1
29	AppIX Semi-Volatile Standard	AccuStandard	2000 µg/ml	M8270-07
30	AppIX Semi-Volatile Standard	AccuStandard	2000 µg/ml	M8270-08
31	AppIX Semi-Volatile Standard	AccuStandard	2000 µg/ml	M8270-09
32	AppIX Semi-Volatile Standard	AccuStandard	2000 µg/ml	M8270-10
33	Allethrin	AccuStandard	Neat	P-267N
34	Baygon	AccuStandard	Neat	P-009N

Reference Number	Standard Name	Vendor Name	Concentration	Catalog Number
35	Carbaryl	AccuStandard	Neat	P-083N
36	Diazinon	AccuStandard	Neat	P-033N
37	Dusban	AccuStandard	Neat	P-094N
38	Fenvalerate	AccuStandard	Neat	P-194N
39	MGK-264	AccuStandard	Neat	P-082N
40	Piperonyl butoxide	AccuStandard	Neat	P-348N
41	Pyrethrins	AccuStandard	Neat	P-187N
42	Resmethrin	AccuStandard	Neat	P-325N
43	Sumithrin (Phenothrin)	AccuStandard	Neat	P-050N
44	Teramethrin (Phthalthrin)	AccuStandard	Neat	P-406N
45	Dazomet	AccuStandard	Neat	P-469N
46	GC/MS Tuning Standard	AccuStandard	1000 µg/ml	M625-TS-20X
47	Benzidine	Ultra Scientific	5000 µg/ml	EPA-107-1
48	1,2-Diphenylhydrazine	Restek	1000 µg/ml	31497-510
49	8270Calibration Mix #5 (19PAHs)	Restek	2000 µg/ml	31622
50	Acid Matrix Spike Mix (1ml)	Restek	10000 µg/ml	31061
51	Acid Matrix Spike Mix (5ml)	Restek	10000 µg/ml	31071
52	B/N Matrix Spike Mix (1 ml)	Restek	5000 µg/ml	31074
53	B/N Matrix Spike Mix (5 ml)	Restek	5000 µg/ml	31084
54	Pyridine	Aldrich	Neat	27040-7
55	Custom Made 8270 Std	Accstandard	2000 µg/ml	S-10776B
56	3- Methylphenol	Supelco	5000 µg/ml	40251-U
57	1-Methylnaphthalene	Chem Service	Neat	0-787
58	Carbazole	Chem Service	Neat	F-2001
59	Benzaldehyde	Chem Service	Neat	F2323
60	Pyridine	Chem Service	Neat	F1084
61	Acetophone	Chem Service	Neat	F985
62	Atrazine	Chem Service	Neat	F2208
63	Biphenyl	Chem Service	Neat	F1062
64	Caprolactam	Chem Service	Neat	0-828
65	Custom Made Pesticide Std	Accustandard	1000 µg/ml	S-8636-R1
66	605 Benzidine Mix	Restex	2000 µg/ml	31030
67	Famphur	Ultra	99% Neat	PST-1430
68	Kepone	Ultra	99% Neat	PST-620
69	Perylene	Ultra	99% Neat	RAH-050
70	Benzoic Acid	Ultra	99.9% Neat	RCC-143
71	Perylene	Chem Service	99% Neat	F1047
72	Benzoic Acid	Accustandard	2mg/ml	Z-0140-1
73	Ethyl Methane Sulfate	Ultra	99.8% Neat	RCC-182
74	Methyl Methane Sulfate	Ultra	99.9% Neat	RCC-185
75	m-Cresol (3-Methylphenol)	Ultra	99% Neat	RCC-156
76	2,3,5,6-Tetrachlorophenol	Ultra	96.7% Neat	RCP-018
77	2,3,4,5-Tetrachlorophenol	Ultra	99% Neat	RCP-016
78	2,3,5,6-Tetrachlorophenol	Supelco	2000 µg/ml	48152
79	2,3,4,5-Tetrachlorophenol	Supelco	2000 µg/ml	48153
80	3,4,5-Trichlorophenol	Ultra	99% Neat	RCP-015
81	3,5-Dichlorophenol	Ultra	99% Neat	RCP-009
82	3-Chlorophenol	Ultra	99% Neat	RCP-002
83	3,4,5-Trichlorophenol	Supelco	99.3% Neat	442373(1)
84	3,5-Dichlorophenol	Supelco	99.9% Neat	442378(1)
85	Pentachlorophenol	Ultra	5000 µg/ml	EPA-1152
86	1-Methylnaphthalene	Restek	1000 µg/ml	31283

Reference Number	Standard Name	Vendor Name	Concentration	Catalog Number
87	2-Methylnaphthalene	Restek	1000 µg/ml	31285
88	SV Calibration Mix	Restek	2000 µg/ml	31011
89	7,12-Dimethylbenz(a)anthracene	Ultra	1000 µg/ml	EPA-1110
90	7,12-Dimethylbenz(a)anthracene	Chem Service	99% Neat	F918
91	7,12-Dimethylbenz(a)anthracene	Ultra	98% Neat	RAH-025
92	Sodium Sulfate	VWR	Pure	3375-09
93	Custom PAH Standard	AccuStandard	2000 ug/ml	S-14764-R2-10X
94	Custom PAH Standrd	UltraScientific	2000 ug/ml	CUS-8533
95	Calibration Check Compounds B/N	AccuStandard	2000 ug/ml	CLP-011A
96	Base/Neutral Extractables Mix 1	AccuStandard	500 ug/ml	M-625-BN-1
97	Base/Neutral Extractables Mix 2	AccuStandard	500 ug/ml	M-625-BN-2
98	SVOC GC/MS Mix 4B	AccuStandard	2000 ug/ml	M-8270-04B
99	4-Chloro-3-cresol	AccuStandard	100 ug/ml	M-8040-01
100	2-Chlorophenol	AccuStandard	100 ug/ml	M-8040-02
101	p-Cresol	AccuStandard	100 ug/ml	M-8040-05
102	o-Cresol	AccuStandard	100 ug/ml	M-8040-04
103	2,4-Dichlorophenol	AccuStandard	100 ug/ml	M-8040-07
104	2,6-Dichlorophenol	AccuStandard	100 ug/ml	M-8040-08
105	2,4-Dimethylpehnol	AccuStandard	100 ug/ml	M-8040-09
106	2,3,4,6-Tetrachlorophenol	AccuStandard	100 ug/ml	M-8040-17
107	2,4,5-Trichlorophenol	AccuStandard	100 ug/ml	M-8040-18
108	2,3,6-Trichlorophenol	AccuStandard	100 ug/ml	M-8040-19

6.0 APPARATUS AND MATERIALS

6.1 Balance - Analytical, capable of accurately weighing 0.0001 g.

6.2 Microsyringes - 10 µl, 25 µl, 100 µl, 250 µl, 500 µl, and 1,000 µl.

6.3 Syringe - 1 ml, gas-tight.

6.4 2 ml crimp top vial.

6.5 Disposable pipettes.

6.6 Carrier Gas, Helium - 99.99% or higher purity.

6.7 Gas chromatograph/Mass spectrometer system

6.7.1 Gas chromatograph (GC) - The laboratory uses Hewlett-Packard gas chromatograph Model 5890 series II GC and Hewlett-Packard Automatic Sampler Model 7673 or equivalent.

6.7.2 Column DB-5MS, 30m x 0.25 mm (ID), 0.25 µm film bonded phase silicone coated fused silica or equivalent, such as RTX-5 sil MS 20m x 0.18mm(ID),0.18µm df.

6.7.3 Mass spectrometer - The laboratory uses Hewlett-Packard 5972 and 5973 Mass Spectrometer or equivalent.

6.7.4 Data system - The data systems used at the laboratory are HP/MS Chem Station with Enviroquant software.

6.8 10 ml and 20 ml Teflon-sealed screw-cap vials and caps.

6.9 1.5 ml target crimp top vials.

6.10 Kuderna-Danish (K-D) apparatus.

6.11 Beaker – 400-ml, thick-walled.

6.12 Funnel-75 mm, diameter.

6.13 Separatory funnel-500ml, with polytetrafluoroethylene (PTFE) stopcock.

6.14 Erlenmeyer flasks-250-ml and 500ml, with a ground-glass joint at the neck.

6.15 Pipette- Pasteur, glass, disposable, 9140mmx5mm ID.

6.16 Filter paper – 15 cm diameter (Whatman No. 1 or equivalent).

6.17 Glass wool – Pyrex, acid washed.

6.18 Boiling chips – solvent-extracted with methylene chloride.

6.19 Water bath – capable of temperature control (± 2°C). The bath should be used in a hood.

6.20 Balance – analytical, capable of accurately weighing to 0.0001 g.

6.21 pH paper – wide range.

6.22 SPE Apparatus:

6.22.1 SPE Extractor (SPE-DEX 4790)

6.22.2 SPE Controller (SPE-DEX Controller for 4790)

6.23 Ultrasonic preparation - A horn-type device equipped with a titanium tip, or a device that will give equivalent performance, shall be used.

6.23.1 Ultrasonic Disrupter - The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. A device designed to reduce the cavitation sound is recommended. Follow the manufacturer's instructions for preparing the disrupter for extraction of samples with low and medium/high concentration. Horn is retuned at least monthly per manufacturer recommendations.

6.23.2 Use a 3/4" horn for the low concentration method and a 1/8" tapered microtip attached to a 1/2" horn for the medium/high concentration method.

6.23.3 **The extraction horns used for the low concentration and high concentration samples are not interchangeable.** Results indicate that the use of the 3/4" horn is inappropriate for the high concentration method, particularly for extraction of very non-polar organic compounds such as PCBs, which are strongly adsorbed to the soil matrix.

6.24 Soxhlet extractor - 40 mm ID, with 500-ml round bottom flask.

6.25 If using the Turbo-Vap sample concentration system:

6.25.1 300 ml concentration vessels with a 0.5 ml or 1.0 ml sensor endpoint.

6.25.2 Centrifuge tubes, graduated (10-15 ml) with screw caps with Teflon-lined septa, and 25 ml graduated centrifuge tubes for the collection of 20 ml fractions.

6.26 300 ml Turbo-Vap concentration vessels with a 0.5 ml or 1.0 ml sensor endpoint.

6.27 Turbo-Vap concentrator system.

7.0 PROCEDURE

7.1 Preparation of extraction log form and extraction log in LIMS.

7.1.1 Each day the section supervisor prepares a work log. The log lists samples that are included in batches that have not been completed and closed in LIMS. IF A SAMPLE IS ON THIS LIST AND IT HAS BEEN EXTRACTED OR ANALYZED, CHECK LIMS TO VERIFY THAT THE BATCH HAS BEEN CLOSED. Samples are listed on the work log in the order of due date.

7.1.1.1 Any samples that are received in a “rush” status will have a chain of custody delivered to the extraction supervisor by the project manager.

7.1.1.2 Prepare a written extraction log using the BNA logbook that is kept in the extraction supervisor’s office. The following entries must be made in the log.

7.1.1.2.1 Date and time that the batch is opened or the date and time the extraction system is started.

7.1.1.2.2 All sample(s) included in the extraction batch.

7.1.1.2.3 Volume or weight of samples extracted.

7.1.1.2.4 Date and time that the extraction is completed.

7.1.1.2.5 Extraction procedure employed.

7.1.1.2.6 The initials of the extraction analysts.

7.1.1.2.7 Laboratory number of all reagents used including surrogate standard, spiking standard, methylene chloride, and sodium sulfate used.

7.1.1.2.8 Volume of all reagents used including surrogate standard, spiking standard, and methylene chloride.

7.1.1.2.9 Final volume of all concentrates.

7.1.1.2.10 Date and time the batch is closed.

7.1.1.2.11 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.1.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.1.1.3.1 Open Prep Batch in LIMS by double clicking the "Prep" box.

7.1.1.3.2 To create a new form, click the "add" box. The prep start date, start time, and batch number are automatically created by the LIMS.

7.1.1.3.3 Select the appropriate Prep Code from the pull down list. The LIMS will automatically assign an MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCSD and enter the information.

7.1.1.3.4 Enter the technician initials from the pull down menu.

7.1.1.3.5 Click the "Load Samps" tab to obtain a list of samples that need preparation by this preparation method. Note that the list contains all samples requiring extraction by the selected method, not just samples that have been logged into the LIMS analysis.

7.1.1.3.6 Select the samples to be included in the batch that are assigned the desired prep method. The samples can be individually selected by highlighting the sample and clicking the "$\rightarrow$" arrow or all samples can be selected by clicking the "$\Rightarrow$" arrow.

7.1.1.3.7 Samples that have be selected for re-analysis can be manually added to the sample list following the procedure outlined in section 7.1.1.3.3.

7.1.1.3.8 "Save" the batch by clicking a previous batch number on the list and then returning to the newly created batch.

7.1.2 Table 7-1 indicates the number and type of samples that comprise a preparatory batch. Note: NELAC requirements specify that the maximum number of client samples a preparatory batch can not exceed 20. Further, a batch can not be left "open" for a time period that exceeds 24 hours.

Table 7-1
Samples in a NELAC Batch

Method Blank (MB)
LCS (and LCSD if no MS/MSD)
Client Samples
MS and MSD (If sufficient volume)

7.2 Extraction of high concentration waste samples by Method 3580A. See TABLE 7-7 for procedure specific checklist.

7.2.1 Remove the samples to be extracted from refrigerator so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.2.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS.

7.2.2 Prepare the borosilicate culture tubes by rinsing them two times with methylene chloride.

7.2.3 Transfer approximately 1 g of waste sample to a borosilicate culture tubes (record weight to the nearest 0.1 g). Wipe the mouth of the vial with a tissue to remove any sample material.

7.2.4 Add 2.5-ml surrogate spiking solution to all samples and blanks. For the sample in each analytical batch selected for spiking, add 2.5 ml of the matrix-spiking standard. For SC samples, use BNA LCS/MS Extended Spiking Solution for 3580_BNA.

7.2.5 Immediately dilute to 10 ml with methylene chloride.

7.2.6 Add 2 g of anhydrous sodium sulfate to the sample.

7.2.7 Cap and shake the sample for 2 min.

7.2.8 Allow the extract to settle in the 10-ml vial with screw top.

7.2.9 Place 1 ml of sample into a GC autosampler vial, add 10µl of internal standard, and seal with a crimp top. Label the vial appropriately.

7.3 Extraction of aqueous samples using separatory funnels by Method 3510. This procedure is also applicable to TCLP extracts. Volumes of organic solvents and other reagents should be adjusted accordingly to account for the reduced volume of TCLP extracts. See TABLE 7-4 for procedure specific checklist.

7.3.1 Remove the samples to be extracted from designated storage location so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.3.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS. At times the wrong extraction procedure is logged into LIMS (e.g., Continuous Liquid/Liquid Extraction instead of Separatory Funnel Extraction). If this is the case, contact the section supervisor.

7.3.1.2 The section supervisor will contact the Project Manager to determine the appropriate actions to be followed.

7.3.2 Prepare a 2-liter separatory funnel and K-D flask with filter funnel for each sample in the analytical batch by rinsing two times with 30 ml of methylene chloride. Discard the solvent media after each rinse.

7.3.3 Place purified glass wool in each funnel and add approximately 10-20 grams of purified sodium sulfate to each funnel.

7.3.4 Table 7-1 indicates the number and type of samples that comprise a preparatory batch. Note: NELAC requirements specify that the maximum number of client samples in a preparatory batch can not exceed 20. Further, a batch can not be left "open" for a period that exceeds 24 hours.

7.3.5 Label each funnel and Erlenmeyer flask using the blue or orange label tape and a "Sharpie". Blue tape is used for 8270/625 BNA analysis. Orange tape is used for Base Neutral fraction only. The information included in the label is the sample name, batch number, test type, and sample matrix as indicated in the example below.

Example Label used in Extraction Laboratory

MB	#9999
Sample ID	
8270C_W	

7.3.6 Mark the sample volume on the sample bottle and transfer the sample to a 2-liter separatory funnel. After rinsing the bottle with methylene chloride, fill with water and measure the volume using a graduated cylinder.

7.3.7 Pre-clean a 1-ml syringe by rinsing at least 5 times with methylene chloride.

7.3.8 Using the syringe, add 1.0 ml of the Spike solution to the LCS, LCSD, MS, and MSD samples. Rinse the syringe with methylene chloride as before. For SC samples, use the BNA LCS/MS Extended Spiking Solution for 3510_BNA.

7.3.9 Add 1.0 ml of the surrogate spiking solutions to all of the samples.

7.3.10 Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to a pH of <2 for BNA using 1:1 (v/v) sulfuric acid or pH 5-9 for Base-Neutrals using 10 N sodium hydroxide. Lower concentrations of acid or base solution may be employed.

7.3.11 Add 60 ml of methylene chloride to the sample bottle and rinse both the bottle and the graduated cylinder. Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure.

7.3.12 Allow the organic layer to separate from the water phase for 2-3 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, and centrifugation.

7.3.13 Collect the methylene chloride extract in a K-D flask flask by pouring the extract through a filter funnel containing purified glass wool and sodium sulfate crystals.

7.3.14 Extract two more times with additional 60-ml portions of methylene chloride. Collect the extract as before. Remove any additional solvent layer. If base neutral extraction is not desired, proceed directly to section 7.7 for extract concentration and solvent exchange.

7.3.15 Adjust the pH of the sample to >11 for Base-Neutrals extraction using 10 N sodium hydroxide or other suitable concentration.

7.3.16 Extract three times with 60-ml aliquots of methylene chloride, as before. Combine the methylene chloride fractions with the methylene chloride from the acid fractions in the K-D flask.

7.3.17 After the third extraction, rinse the funnel containing the glass wool and sodium sulfate with additional methylene chloride. **Do not allow any water into the flask. This will compromise the results.**

7.3.18 Proceed to Section 7.7.1 for extract concentration and solvent exchange.

7.4 Extraction of aqueous samples by SPE. See TABLE 7-5

7.5 Extraction of soil samples using horn sonicator by Method 3550C. See TABLE 7-6 for procedure specific checklist.

Extractions are performed with the appropriate solvent, the extraction is performed in the specified pulse mode, and the horn tip is positioned just below the surface of the solvent yet above the sample.

Very active mixing of the sample and the solvent must occur when the ultrasonic pulse is activated. The analyst must observe such mixing at some point during the extraction process.

7.5.1 Remove the samples to be extracted from refrigerator so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information, follow the following steps.

7.5.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS.

7.5.2 Prepare a 400-ml jar and Zymark tube with filter funnel for each sample in the analytical batch by rinsing two times with 30 ml of methylene chloride. Discard the solvent media after each rinse. Assemble the apparatus.

7.5.3 Place a folded filter paper in each funnel and add approximately 10-20 grams of purified sodium sulfate to each funnel.

7.5.4 Table 7-1 indicates the number and type of samples that comprise a preparatory batch. Note: NELAC requirements specify that the maximum number of client samples in a preparatory batch can not exceed 20. Further, a batch can not be left "open" for a period that exceeds 24 hours.

7.5.5 Label jars and Erlenmeyer flask using the blue or orange label tape and a "Sharpie". Blue tape is used for 8270/625 BNA analysis. Orange tape is used for Base Neutral fraction only. The information included in the label is the sample name, batch number, test type, and sample matrix as indicated in the example below.

Example Label used in Extraction Laboratory

MB	#9999
Sample ID	
8270C_S	

7.5.6 Remove approximately one-half of the contents of the soil jar. In a clean container, mix the soil in an attempt to make it as homogenous as possible.

7.5.7 Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture **must be mixed** with 60 g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate may be added. After addition of sodium sulfate, the sample should be free flowing.

7.5.8 Accurately weigh approximately 30 grams of soil sample and place into the appropriate jar. For the sample chosen for MS/MSD, the initial weights for MS and MSD must be within 5% of each other.

7.5.9 Pre-clean a 1-ml syringe by rinsing at least 5 times with methylene chloride.

7.5.10 Using the syringe, add 1.0 ml of the Spike solution to the LCS, LCSD, MS, and MSD samples. Rinse the syringe with methylene chloride as before. For SC samples use the BNA LCS/MS Extended Spiking Solution for 3550_BNA.

7.5.11 Add 1.0 ml of the surrogate spiking solutions to all of the samples.

7.5.12 Immediately add 100 ml of methylene chloride to each jar.

7.5.13 Place the bottom surface of the tip of the #207 (or equivalent) 3/4 inch disrupter horn about 1/2 inch below the surface of the solvent, but above the sediment layer.

7.5.14 Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy). Percent-duty cycle knob must be set at 50% (energy on 50% of time and off 50% of time). **Do not use microtip probe. Do not sonicate with a power setting that is less than 7**.

7.5.15 Filter the extract into the Zymark tube through Whatman No. 41 filter paper with purified sodium sulfate. Rinse the sample vessel and filter media thoroughly into the Zymark tube.

7.5.16 Repeat the extraction twice with an additional 100 ml portion of solvent. Filter the extract into the Zymark tube through the filter paper and sodium sulfate. On the final ultrasonic extraction, pour the entire sample into the funnel and rinse with extraction solvent. Continue filtration until all visible solvent is removed from the funnel, but do not attempt to completely dry the sample.

7.5.17 Proceed to Section 7.7.2 for extract concentration and solvent exchange.

7.6 Extraction of soil samples using Soxhlet extraction apparatus by Method 3540.

7.6.1 Remove the samples to be extracted from designated storage location so they can warm to room temperature. If the samples cannot be found in the refrigerator, they may have been omitted from the chain of custody, incorrectly logged into LIMS, or their test status changed. To determine this information,

follow the following steps.

7.6.1.1 Check the sample extraction list (See section 7.1.1) for directions on the use of LIMS. At times the wrong extraction procedure is logged into LIMS (e.g., Continuous Liquid/Liquid Extraction instead of Separatory Funnel Extraction). If this is the case, contact the section supervisor.

7.6.1.2 The section supervisor will contact the Project Manager to determine the appropriate actions to be followed.

7.6.2 Prepare a Soxhlet extraction apparatus and Erlenmeyer flask with filter funnel for each sample in the analytical batch by rinsing two times with 30 ml of methylene chloride. Discard the solvent media after each rinse. Assemble the apparatus.

7.6.3 Place a folded filter paper in each funnel and add approximately 10-20 grams of sodium sulfate to each funnel.

7.6.4 Table 7-1 indicates the number and type of samples that comprise a preparatory batch. Note: NELAC requirements specify that the maximum number of client samples in a preparatory batch can not exceed 20. Further, a batch can not be left "open" for a period that exceeds 24 hours.

7.6.5 Label jars and Erlenmeyer flask using the blue or orange label tape and a "Sharpie". Blue tape is used for 8270/625 BNA analysis. Orange tape is used for Base Neutral fraction only. The information included in the label is the sample name, batch number, test type, and sample matrix as indicated in the example below.

Example Label used in Extraction Laboratory

MB	#9999
Sample ID	
8270C_S	

7.6.6 Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to a pH of 5-9 using 1:1 (v/v) sulfuric acid or 10 N sodium hydroxide.

7.6.7 Remove approximately one-half of the contents of the soil jar. In a clean container, mix the soil in an attempt to make it as homogenous as possible.

7.6.8 Blend 10 g of the solid sample with 10 g of anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble.

7.6.9 Pre-clean a 1-ml syringe by rinsing at least 5 times with methylene chloride.

7.6.10 Using the syringe, add 1.0 ml of the Spike solution to the LCS, LCSD, MS, and MSD samples. Rinse the syringe with methylene chloride as before.

7.6.11 Add 1.0 ml of the surrogate spiking solutions to all of the samples.

7.6.12 If EPA SW-846 Method 3640, Gel-Permeation Cleanup, is to be employed, add twice the volume of the surrogate spiking solution and the matrix spiking standard, since half of the extract is not recovered from the GPC apparatus. (Alternatively, use 1.0 ml of the spiking solutions and concentrate the final extract to half the normal volume, e.g., 0.5-ml instead of 1.0 ml).

7.6.13 Place approximately 300 ml of methylene chloride into a 500-ml round bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16 - 24 hours at 4 - 6 cycles/hour.

7.6.14 Allow the extract to cool after the extraction is complete. Record the extraction start time and completion time.

7.6.15 Quantitatively transfer the extract to the K-D concentrator or Turbo-Vap tube. Proceed to Section 7.7 for extract concentration and solvent exchange.

7.7 Extraction concentration.

7.7.1 Kuderna-Danish (K-D) Sample Concentration

7.7.1.1 Add one to two clean boiling chips to the evaporation flask and attach a three ball Snyder column to the flask. Pre wet the Snyder column by adding about 1 ml of methylene chloride through the top.

7.7.1.2 Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10 - 15 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

7.7.1.3 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1 - 2 ml of methylene chloride. Remove the bottom section of the K-D apparatus.

7.7.1.4 Attach a micro Snyder column to the concentrator tube and carefully concentrate to below 1 ml on the water bath**. DO NOT ALLOW THE TUBE TO GO DRY**.

7.7.1.5 Rinse the concentrator tube with a small amount of methylene

chloride and bring final volume to exactly 1.0 ml.

7.7.1.6 Transfer the concentrate to 2-ml GC autosampler vials or a screwtop vial for analysis.

7.7.2 Turbo-Vap Sample Concentration

7.7.2.1 Set the Turbo-Vap concentrator bath temperature to 39°C. Set the Turbo-Vap pressure to 20. Check the water level in the bath and ensure that it is half way up the lower set of perforations in the back of the chamber.

7.7.2.2 Program the Turbo-Vap to stop indicate concentration completion by an audible sound. Note that the sample could go dry because the system continues to operate after the system emits the sound.

7.7.2.3 Place the vessels in the Turbo-Vap and close the lid. Press the start button for the appropriate vessel. When the sample is complete, the sensor will sound intermittently until the vessel is removed.

7.7.2.4 Remove the vessel from the Turbo-Vap"' to prevent further evaporation. The outside of the condenser may be covered in water droplets. Be sure that they do not drip into the sample extract. If the sample evaporates to dryness, re-extract the entire sample.

7.7.2.5 Cool the sample, rinse down the sides of the Zymark tube and dilute to exactly 1.0 ml with methylene chloride. Transfer to a screw top vial for storage.

7.7.2.6 If the sample will not concentrate to 10 ml or less, is very viscous, or is very dark in color, make comments in the logbook and dilute to a higher volume.

7.8 Sample cleanup methods.

7.8.1 If a sample is of biological origin, or contains high molecular weight materials, the use of Method 3640 (GPC cleanup-pesticide option) is recommended. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel, or florisil) may be required following the GPC cleanup.

7.9 Gas chromatographic/Mass Spec conditions

7.9.1 Set up operating conditions for the GC/MS using the following guidelines. These conditions may be changed as necessary to improve or maintain analytical conditions.

7.9.2 These chromatographic conditions should be sufficient to achieve the detection limits in Section 5 of the QA Manual.

7.9.3 Preparation of Gas Chromatographic conditions using Chemstation/Enviroquant software and "Methods" function. Typically, the gas chromatographic conditions have been pre-set so as to conform to the method, however, due to unforeseen circumstances, it may be necessary to change these conditions. This task can be performed by following the subsequent instructions.

7.9.3.1 Open Enviroquant software.

7.9.3.2 In the pull-down menu, click "Method". Select the desired method.

7.9.3.3 Click "Edit Entire Method", then "OK". The software will prompt the user through the method for edit.

7.9.3.4 Current GC/MS conditions for GC-MS-9 Method 8RTX5M using electronic pressure control (EPC) are as follows:

Electron energy:	70 volts (nominal).
Mass range:	35-500 amu.
Scan time:	3.15 Scans / Sec.
Initial column temp. hold:	40°C for 0.50 minutes
Column temp. ramp 1:	40°C at 14°C/minute to 90°C
Column temp. ramp 2:	90°C at 22°C/minute to 325°C
Final column temp. hold:	325°C for a maximum of 1.5 minutes
Injector temperature:	270°C
Injector-Grob-type:	splitless
Sample injection volume:	0.5 µl
Constant Flow: off	
Carrier gas:	Helium at 1 ml/minute
Purge Time (on):	0.5 minute
Solvent delay:	0.9 minute
Pressure Pulse Start	6.7 psi
Pressure Pulse Ramp Up	99 psi/min to 40 psi, hold 0.5 min
Pressure Pulse Ramp Down	99 psi/min to 6.7 psi

7.9.3.5 Set the output destination to screen, printer, and file.

7.9.3.6 Set the method to "autointegrate", and not to generate a report during the run (wastes paper).

7.9.3.7 Set reference window to 10%, non-reference window to 5%, correlation window to 0.02 minutes, default multiplier to 1.00, and default sample concentration to 0.00.

7.9.4 Preparation of 'Run Log' using Chemstation/Enviroquant software.

7.9.4.1 Open Enviroquant software by "clicking" on Icon.

7.9.4.2 Select "Sequence", then "Edit sample log table".

7.9.4.3 Select sample type for each sample.

7.9.4.4 Number all of the samples.

7.9.4.5 Change data file using the following format:

41030501,

where:

4 = GC number
Number 1= Year: '01'
0305 = Date in MM DD format
01 = sequential file number

7.9.4.6 Click "OK" when completed.

7.9.4.7 On the pull-down menu, select "Sequence", then "Save".

7.9.4.8 When the following prompt appears, change the last part to MM DD format indicating the current day.

C:\HPCHEM\4\Data\410305,

where:

410305 = Instrument 4, month "03", day "05"

7.9.4.9 When complete, click "OK".

7.9.4.10 On the pull-down menu, select "Sequence", then "Run" to start the instrument run.

7.9.5 Editing Sequences

7.9.5.1 Click "File", then "Edit".

7.9.5.2 Sample information can be directly entered into the pop up box that appears.

7.10 Initial calibration of the GC/MS System

7.10.1 Prior to analysis of any tunes, standards or samples, several MS and GC parameters should be checked and recorded in order to ensure that operating conditions have not changed significantly from those of prior analyses. The routine system checks are as follows:

7.10.1.1 The MS vacuum system source pressure should be checked and should be stable. (Acceptable pressure is approximately 50 mtorr or lower when the interface temperature is 280°C.).

7.10.1.2 The auto-sampler syringe rinse solution should be filled with methylene chloride and waste bottles should be emptied.

7.10.1.3 The MS system should be checked for leaks by monitoring for selected ions that indicate the presence of air and water (m/z ratio at 18, 28, 32, 44).

7.10.1.4 A manual tune should be performed, recording the ion abundances and relative percentages characteristic for the manual tuning compound PFTBA.

7.10.2 Instrument conditions and hardware tune must be evaluated and meeting all ion abundance criteria per Section 7.12.1 prior to analysis of any standards or samples.

7.11 Initial GC/MS calibration curve

7.11.1 The internal standard method is used for initial calibration. Analysis of standards containing the target compounds as described in Tables 5-1, 5-2 and 5-3 at a minimum of five different concentrations covering the working range of the instrument. The lowest standard must be at the concentration equal to the LIMS reporting limit while the highest standard should be at the LIMS UQL.

7.11.2 *Calibration using average response with internal standards*. Calculate response factors (RFs) for each target analyte relative to one of the internal standards as follows:

$$RF = \frac{A_s \ X \ C_{is}}{A_{is} \ X \ C_s}$$

where:

A_s = Peak area or height of the analyte or surrogate
A_{is} = Peas area or height of the internal standard
C_{is} = Concentration of the analyte or surrogate, in ug/L
C_{is} = Concentration of the internal standard, in ug/L

7.11.2.1 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum response factor for the most common target analytes, as noted in Table 1-1,

be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet this criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. The analyst should also strive to place more emphasis on meeting calibration criteria for those compounds that are critical project compounds, rather than meeting the criteria for those less important compounds.

7.11.2.2 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, then the chromatographic system is considered too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with 7.11.

7.11.2.3 Evaluation of retention times. The relative retention time (RRT) of each target analyte calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

$$RRT = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

7.11.2.4 Linearity of target analytes – If the RSD of any target analyte is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

7.11.3 SW-846 methods allow the use of both linear and non-linear models for the calibration data. It is AES' policy that non-linear models are not used in any case. In all methods employed by the laboratory, the correlation between response and concentration can be described in a linear method, either average response or linear regression.

7.11.4 *Calibration using linear regressions*. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

where:

y = Instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = The intercept

When internal standards are used, the equation becomes:

$$\frac{As\ Cis}{Ais} = aCs + b$$

where:

A = Area (or height) of the peak for the target analyte in the sample s
A = Area (or height) of the peak for the internal standard is
C = Concentration of the target analyte in the calibration standard s
C = Concentration of the internal standard is
a = Slope of the line (also called the coefficient of C) s
b = The intercept

7.11.4.1 The analyst should not force the line through the origin, but have the intercept calculated from the five data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0,0) as a sixth calibration point.

7.11.4.2 The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit.

In

order to be used for quantitative purposes, per AES' policy, r must be greater than or equal to 0.995.

7.11.4.3 The Enviroquant software will automatically calculate the linear regression for any target analyte by selecting "linear regression" in the calculation mode. When in this mode, the software plots the analyte response ratio against the analyte ratio.

7.11.4.3.1 The response ratio is defined as the area of the analyte divided

by the area of the internal standard.

7.11.4.3.2 The analyte ratio is defined as the concentration of the analyte

divided by the concentration of the internal standard.

7.11.5 The initial calibration curves must be verified using a second source standard (ICV) containing all target analytes. The value determined from the ICV should be within 30% of the expected concentration for each analyte.

7.11.6 The calibration curve review checklist is completed for each curve.

7.12 Daily GC/MS calibration verification

7.12.1 DFTPP Tune Evaluation. Before analysis and every 12 hours of operation, the hardware tuning for each GC/MS system must be verified as follows:

7.12.1.1 Inject 1ul (0.5ul for microbore columns) of DFTPP standard and acquire data.

7.12.1.2 Spectral information must be obtained using one of the following options:

1. The average of the apex scan plus one scan to the left of the apex and one scan to the right of the apex with background subtraction using a single scan no more than 20 scans from the apex and completely off the DFTPP peak. Do not subtract part of the DFTPP peak.

2. A single scan within ±3 scans of the apex with background subtraction using a single scan no more than 20 scans from the apex and completely off the DFTPP peak. Do not subtract part of the DFTPP peak.

3. The average scan across the entire chromatographic peak with background subtraction using a single scan no more than 20 scans from the apex and completely off the DFTPP peak. Do not subtract part of the DFTPP peak.

7.12.1.3 Ion abundances for the DFTPP resulting from evaluation per 7.12.1.2 must meet the criteria specified in Table 7-2 below before analysis of samples and/or standards may begin.

7.12.1.4 If ion abundance criteria are not met, proceed with one or both of the following options:

7.12.1.4.1 Make a fresh DFTPP standard and reinject. If Table 7-2 criteria are now met using evaluation per 7.12.1.2, analysis may continue.

7.12.1.4.2 If Table 7-2 criteria are not met, instrument maintenance must be performed to restore ion abundance ratios to meet Table 7-2 criteria and/or the instrument must be retuned to restore ion abundance ratios to meet Table 7-2 criteria.

Table 7-2
DFTPP KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

7.12.2 At the beginning of each day and every 12 hours thereafter that the base/neutral fraction is to be analyzed, the benzidine and pentachlorophenol tailing must be calculated. Inject 50 ng of these compounds as a part of standard mixture that may contain DFTPP. The benzidine tailing factor and pentachlorophenol tailing factor must be $\leq$2.0. Degradation of DDT to DDE and DDD must be calculated and should not exceed 20%.

7.12.3 Initial calibrations must be verified at the beginning of each 12 hour shift by using a CCV standard.

7.12.4 The CCV should be a standard containing all the compounds of interest for quantitation at a concentration either near the midpoint concentration for the calibration range of the GC/MS or near the action level for the project.

7.12.4.1 Each of the most common target analytes in the CCV should meet the minimum response factors as noted in TABLE 1-1. This criteria is particularly important when the common target analytes are also critical project-required compounds. This is the same check that is applied during initial calibration.

7.12.4.2 If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.12.4.3 All target compounds of interest must be evaluated using a 20% criterion. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.

7.12.4.4 If the percent difference or percent drift for a compound is $\leq$20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. If the criterion is not met (i.e., >20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be determined that there was adequate sensitivity to detect the compound at the applicable quantitation limit, $\geq$50%..

7.12.4.5 Problems similar to those listed under initial calibration could affect the ability to pass the calibration verification standard analysis. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The calibration verification criteria must be met before sample analysis begins.

7.12.4.6 The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within $\pm$30% of the standard's true concentration. Other recovery criteria may be applicable depending on the project's data quality objectives and for those situations the minimum quanititation check criteria should be outlined in a laboratory SOP or project-specific QAPP. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

7.12.5.7 Corrective actions for failure to meet any of the above criteria may include, but are not limited to, reinjection of freshly prepared standard, inlet maintenance, column maintenance, etc. If corrective actions do not result in all evaluation criteria being met, recalibration is required

prior to further analysis.

7.12.5 Evaluation of retention times. If the retention time for any internal standard in any ICV or CCV changes by more than 30 seconds from that in the mid-point level of the most recent initial calibration, the chromatographic system must be inspected for malfunctions and corrections made as required.

7.12.6 The internal standard responses in the check calibration standard must be evaluated immediately after or during data acquisition. **If the peak area for any of the internal standards changes by a factor of two or more (- 50% to + 100%) from that in the mid-point standard level of the calibration curve being used, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate**. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

7.12.7 TABLE 7-3 lists the internal standards with their corresponding analytes that are used for GC/MS semivolatile analysis.

TABLE 7-3
Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	**Chrysene-d_{12}**	**Perylene-d_{12}**
Aniline	Benzidine	Benzo(b)fluoroanthene
Benzyl alcohol	Benzo(a)anthracene	Benzo(k)fluoroanthene
Bis(2-chloroethyl) ether	Bis(2-ethylhexyl)phthalate	Benzo(g,h,i)perylene
Bis(2-chloroisopropyl) ether	Butyl benzyl phthalate	Benzo(a)pyrene
2-Chlorophenol	Chrysene	Dibenz(a,j)acridine
1,3-Dichlorobenzene	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
1,4-Dichlorobenzene	p-Dimethylaminoazobenzene	Fenvalerate
1,2-Dichlorobenzene	Pyrene	
Ethyl methanesulfonate	Terphenyl-d_{14} (surr.)	
2-Fluorophenol (surr.)	7,12-Dimethylbenz- (a)anthracene	
Hexachloroethane	Di-n-octyl phthalate	
Methyl methanesulfonate	Indeno(1,2,3-cd)pyrene	
2-Methylphenol	3-Methylcholanthrene	
4-Methylphenol	Phenothrin	
N-nitrosodimethylamine		
N-nitroso-di-n-propylamine		
Phenol		
Phenol-d_5(surr.)		
2-Picoline		
Benzaldehyde		
N-Nitrosomorpholine		
N-Nitrosomethylethylamine		
N-Nitrosodiethylamine		
N-Nitrosopyrrolidine		
o-Toluidine		

Phenanthrene-d_{10}	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
4-Aminobiphenyl	Acetophenone	Acenaphthene
Anthracene	Benzoic acid	Acenaphthlene
4-Bromophenyl phenyl ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Di-n-butyl phthalate	4-Chloroaniline	2-Chloronaphthalene
4,6-Dinitro-2-methylphenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
Diphenylamine	2,4-Dichlorophenol	Dibenzofuran
Fluoranthene	2,6-Dichlorophenol	Diethyl phthalate
Hexachlorobenzene	α,α-Dimethylphenethylamine	Dimethyl phthalate
N-Nitrosodiphenylamine	2,4-Dimethylphenol	2,4-Dinitrophenol
Pentachlorophenol	Hexachlorobutadiene	2,4-Dinitrotoluene
Pentachloronitrobenzene	Isophorone	2,6-Dinitrotoluene
Phenacetin	2-Methylnaphthalene	Fluorene
Phenanthrene	Naphthalene	2-Fluorobiphenyl (surr.)
Pronamide	Nitrobenzene	Hexachlorocyclopentadiene
Atrazine	Nitrobenzene-d_5 (surr.)	1-Naphthylamine
Azobenzene	2-Nitrophenol	2-Naphthylamine
1,2-Diphenylhydrazine	N-Nitrosodibutylamine	2-Nitroaniline
Carbazole	N-Nitrosopiperidine	3-Nitroaniline
Disulfoton	1,2,4-Trichlorobenzene	4-Nitroaniline
Methyl Parathion	Caprolactam	4-Nitrophenol
Aramite	1-methylnapthalene	Pentachlorobenzene
Parathion	p-Phenylenediamine	1,2,4,5-Tetrachlorobenzene
4-Nitroquinoline-1-oxide	Hexachloropene	2,3,4,6-Tetrachlorophenol
Methapyrilene	Safrole	2,4,6-Tribromophenol
Isodrin	o,o,o-Triethylphosporothioa	2,4,6-Trichlorophenol
2-Acetylaminofluorene	Isafrole	2,4,5-Trichlorophenol
Chlorobenzene	m-Dinitrobenzene	1,1-Biphenyl
3,3’ Dimethylbenzidine	1,4-Napthoquinone	Baygon
Famphur		Diazinon
Kepone		5-Nitro-o-toluidine
Carbaryl		Thionazin
Chloropyrifos		Sulfotep
MGK		Diallate (Cis,trans)
Allethrin		1,3,5-Trinitrobenzene
Pyrethrin		Dimethoate
Piperonyl Butoxide		
Resmethrin		
Tetramethrin		

7.13 Sample Analysis

7.13.1 Add 10 µl of 4000 ng/µl internal standard solution with a 10 µl microsyringe to each accurately measured 1-ml final sample extract.

7.13.2 Inject 1 µl of final sample extract to the GC/MS system to RTX-5 sil MS column, inject 0.5 µl of final sample extract to the GC/MS system. Record the injection in the GC/MS instrument run logbook

7.13.3 If the initial analysis or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Additional internal standard must be added to the diluted extract to maintain the required 40 ng/µl of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

7.13.4 Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, the analyst must either:

7.13.4.1 Analyze a solvent blank immediately after the contaminated sample. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences. This procedure may only be performed when operating in a manual mode.

7.13.5 If an auto sampler is used for a analytical sequence run, the next sample must not contain a concentration above the reporting limit (< RL) of any analyte that exceeded the calibration range in the contaminated sample. If these analytes are present in the sample, the system must be decontaminated and the subsequent samples reanalyzed.

7.13.6 Before processing any samples, the analyst must analyze a method blank or CH_2Cl_2 to demonstrate that interferences from the analytical system and glassware are under control. The concentration of analytes in the reagent blank must be less than the reporting limit. See Section 8 of this SOP and Section 5 of the QA Manual for additional information.

7.13.7 Analyze all blank, LCS, and matrix spike samples in the same manner as the client samples

7.14 Qualitative data interpretation

7.14.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum.

7.14.1.1 The reference mass spectrum must be generated by the laboratory using the conditions of this method.

7.14.1.2 The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

7.14.1.3 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.14.1.4 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.14.1.5 The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.).

7.14.1.6 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.14.2 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles (EICP) of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds.

7.14.3 When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.14.4 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification (Tentatively Identified Compounds, TIC). The necessity to perform this type of identification will be determined by the type of analyses being conducted.

7.14.5 Guidelines for making tentative identification are as follows.

7.14.5.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

7.14.5.2 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

7.14.5.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

7.14.5.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

7.14.5.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system, library reduction programs can sometimes create these discrepancies.

7.14.5.6 Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

7.15 Quantitative data interpretation

7.15.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.15.2 When linearity exists, calculate the concentration of each identified analyte in the sample as follows:

Water

$$Concentration(\mu g / L) = \frac{A_x * Q_{is} * V_{ex}}{A_{is} * RF_{avg} * V_o}$$

where:
A_x = Area of characteristic ion for compound being measured.
Q_{is} = Amount of internal standard injected (ng/µl).
A_{Is} = Area of characteristic ion for the internal standard.
RF_{avg} = Mean relative response factor for compound being measured.
V_o = Volume of liquid extracted, in L.
V_{ex} = Final extract volume, in µl

Sediment/Soil Sludge and Waste (on a wet weight basis)

$$Concentration(\mu g / Kg) = \frac{A_x * Q_{is} * V_{ex}}{A_{is} * RF_{avg} * W_s}$$

where:
A_x, Q_{IS}, A_{IS}, RF_{avg} = Same as for water.
V_{ex} = Final volume of extract (µL).
W_s = Weight of sample extracted (Kg).

Muti-phase Samples:

If the individual phase are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$FinalConcentration = \frac{(V_1 * C_1) + (V_2 * C_2)}{V_1 + V_2}$$

where:
V_1 = The volume of the first phase.
C_1 = the concentration of the analyte of concern in the first phase.
V_2 = The column of the second phase.
C_2 = The concentration of the analyte of concern in the second phase.

7.15.3 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulae given above should be used with the following modifications: The areas A_x and A_{IS} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.15.4 The concentration obtained should be reported using the following criteria.

7.15.4.1 The determined value is an estimate.

7.15.4.2 Report which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.16 Sequence Set Up

7.16.1 Open Enviroquant software.

7.16.2 Develop Log Table.

7.16.2.1 Select “Sequence”, then “Edit sample log table”.

7.16.2.2 Select sample type for each sample.

7.16.2.3 Number all of the samples.

7.16.2.4 Change data file using the following format:

SV7003516,

where:

SV = Semi Volatiles
Number 7 = instrument number
003516 = sequential file number

7.16.2.5 Enter the sample information using the following format

Data file SVSTD 050W,

where:

SV = Semi Volatiles
STD8116 = standard (8116 sequential number from LIMS)
MB8116 = method blank (8116 sequential number from LIMS)
DFTPP = DFTPP tune
LCS8116 = LCS (8116 sequential number from LIMS)
050 = sample concentration
W = sample matrix (water)
Various samples for analysis
Sample weight in grams

7.16.2.6 Enter the method (8270).

7.16.2.7 Save the data in the following directory:

D:\MSDChem\1\Data\Nov02,

where:

Nov02 is the date the file is created.

7.16.2.8 Click "Run", then enter the date of the run. Note that if this is not done, the new analytical batch will overwrite the old batch.

7.16.2.9 Click "Sequence, Position and Run".

7.16.2.10 Click "Sample", then click "OK".

7.16.2.11 Click "Run Sequence".

7.16.2.12 If the prompt "Process Keywords Before Processing Sequence" appears (it will after modifications are made to existing sequences), click "Yes".

7.17 Editing Sequences

7.17.1 Click "File", then "Edit".

7.17.2 Sample information can be directly entered into the pop up box that appears.

7.18 Selected Ion Monitoring (SIM) Analysis

7.18.1 If SIM analysis is requested for a sample, a full scan analysis at the regular concentration levels is performed on that sample prior to the SIM analysis (Exhibit D Semivolatiles, Section 10.1, SOP01.1, 5/2005).

7.18.2 The use of Selected Ion Monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Using the primary ion for quantitation and the secondary ions for confirmation, set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table.

The dwell time may be automatically calculated by the laboratory's GC/MS software or manually calculated using the following formula. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak. The start and stop time for the SIM groups are determined from the full scan analysis using the formula below:

$$\text{Dwell Time for the Group} = \frac{\text{Scan Time (msec)}}{\text{Total Ions in the Group}}$$

7.18.3 The following instrument parameters are used for SIM analysis:

Electron energy:	70 volts (nominal).
Mass range:	35-500 amu.
Scan time:	See individual group
Initial column temp. hold:	40°C for 0.50 minutes
Column temp. ramp 1:	40°C at 14°C/minute to 90°C
Column temp. ramp 2:	90°C at 22°C/minute to 325°C
Final column temp. hold:	325°C for a maximum of 1.5 minutes
Injector temperature:	270°C
Injector-Grob-type:	splitless
Sample injection volume:	0.5 µl
Constant Flow:	off
Carrier gas:	Helium at 1.2 ml/minute
Purge Time (on):	0.5 minute
Solvent delay:	0.9 minute
Pressure Pulse Start	6.7 psi
Pressure Pulse Ramp Up	99 psi/min to 40 psi, hold 0.5 min
Pressure Pulse Ramp Down	99 psi/min to 6.7 psi

SIM Parameters

Group	Start Time	# of Ions
1	0.8	4
2	4.7	4
3	5.53	6
4	6.4	3
5	7.06	3
6	7.52	8
7	8.3	4
8	9.17	7
9	10.44	4
10	11.09	4
11	11.32	4
12	12.07	6
13	13.22	4
14	13.87	7
15	14.58	8

*Note--Start time will change depending on instrument conditions.

Group	1	2	3	4	5	6	7	8
Cycles/sec	5.05	5.05	4.01	5.81	5.8	5.09	5.04	4.63
Resolution	low	low	low	low	low	low	low	low
m/z	74.1	54	68	74.1	74.1	74.1	74.1	74.1
Dewll (msec)	100	25	25	100	100	100	100	100
m/z	74.1 (100)	54 (25)	68 (25)	74.1 (100)	74.1 (100)	74.1 (100)	74.1 (100)	74.1 (100)
	115 (25)	74 (100)	74.1 (100)	141.05 (25)	171 (25)	151 (10)	165 (25)	80 (15)
	150 (25)	82 (25)	127 (25)	142.05 (25)	172 (25)	152 (10)	166 (25)	94 (15)
	152 (25)	128 (25)	128 (25)			153 (10)	167 (25)	176 (15)
			129 (25)			154 (10)		178 (15)
			136 (25)			160 (10)		179 (15)
						162 (10)		188 (15)
						164 (10)		

Group	9	10	11	12	13	14	15
Cycles/sec	5.04	5.04	5.03	5.7	5.92	3.62	3.31
Resolution	low	low	low	low	low	low	low
m/z	74.1	74.1	74.1	74.1	74.1	74.1	74.1
Dewll (msec)	100	100	100	100	100	100	100
m/z	74.1 (100)	74.1 (100)	74.1 (100)	74.1 (100)	74.1 (100)	74.1 (100)	74.1 (100)
	101 (25)	200 (25)	122 (25)	120 (10)	125 (15)	125 (25)	138 (25)
	202 (25)	202 (25)	212 (25)	226 (10)	252 (15)	252 (25)	139 (25)
	203 (25)	203 (25)	244 (25)	228 (10)	253 (15)	253 (25)	227 (25)
				229 (10)		260 (25)	276 (25)
				240 (10)		264 (25)	277 (25)
						265 (25)	278 (25)
							279 (25)

**Note--The last m/z in the table represents the different ions in those groups and the number in paranthesis are for the dwell times in msec.

TABLE 7-4
Checklists for Extraction Procedures
3510_BNA

___ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(3510_BNA AND 8270_TCL_W/8270_TCL_W_CLP/8270_A1_W/8270_A2_W/8270_A9_W/ 8270C_W/1311_B)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (SEPARATORY FUNNELS, K-D FLASKS AND TIPS, FUNNELS AND SYRINGE)

____ : USE BLUE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : TRANSFER 1000 ml SAMPLE INTO SEPARATORY FUNNEL. ADD 1000 ml DI WATER FOR MB AND LCS (AND LCSD IF THE SAMPLE AMOUNT IS INSUFFICIENT TO PREP MS/MSD) (IF ONLY 2 BOTTLES HAVE BEEN PROVIDED BY THE CLIENT, USE 500 ml FOR SAMPLE, MS AND MSD FOR THE QC SAMPLE)
(USE 100 ml SAMPLE AND DI WATER FOR TCLP/1311_B SAMPLES)

____ : CHECK pH OF THE SAMPLES, pH SHOULD BE LESS THAN 2

____ : ADD 5 ml SULFURIC ACID TO SAMPLES TO ADJUST THE pH TO LESS THAN 2 IF NECESSARY. CHECK THE pH OF THE SAMPLES, ADD MORE ACID IF NECESSARY.

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml BNA SPIKE AND ADD 1.0 ml BNA SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE. FOR SC SAMPLES USE BNA LCS/MS EXTENDED SPIKING SOLUTION, LCSSC. (SPIKE LCS AND MS WITH BNA TCLP SPIKE FOR TCLP BATCHES, DO NOT PREP MSD)

____ : ADD 60 ml METHYLENE CHLORIDE TO THE SAMPLE, CAP THE SEPARATORY FUNNEL AND SHAKE FOR ABOUT 2 MINUTES

____ : LET THE METHYLENE CHLORIDE SETTLE FOR ABOUT 2-3 MINUTES AND FILTER THE BOTTOM METHYLENE CHLORIDE PORTION THROUGH GLASS WOOL WITH SODIUM SULFATE INTO THE K-D FLASK

____ : REPEAT THE EXTRACTION 2 MORE TIMES WITH 60 ml METHYLENE CHLORIDE EACH

FOR 8270_A1_W / 8270_A2_W / 8270_A9_W / 8270C_W / 1311_B SAMPLES:

____ : ADD 10 ml NaOH SOLUTION TO THE SAMPLES (460 g NaOH / 1 L DI WATER) TO ADJUST THE pH TO HIGHER THAN 11

____ : CHECK THE pH OF THE SAMPLES, IF NECESSARY ADD MORE NaOH SOLUTION

____ : EXTRACT 3 MORE TIMES WITH 60 ml METHYLENE CHLORIDE AND FILTER THE BASE EXTRACTS TO THE SAME K-D AS ACID EXTRACTS AND COMBINE THEM

____ : ADD COUPLE OF BOILING CHIPS AND CONNECT A SNYDER COLUMN TO THE K-D FLASK

____ : CONCENTRATE THE SAMPLES IN WATERBATH AT TEMPERATURE 60-65°C. (MAKE SURE THE TIP IS TIGHTL Y CONNECTED TO THE K-D FLASK BEFORE PUTTING IN THE WATERBATH)

____ : CONCENTRATE THE SAMPLES DOWN TO AROUND 2.0-3.0 ml. BE CAREFUL TO TAKE THE K-D OUT BEFORE THE SAMPLE IS DRY. LET IT COOL FOR ABOUT 5 MINUTES BEFORE SEPARATING THE TIP

____ : CONCENTRATE TO ABOUT 1.0 ml AGAIN BY USING A MICRO-SNYDER COLUMN ON THE TIP (ADD ONE MORE BOILING CHIP TO THE TIP BEFORE STARTING CONCENTRATION)

____ : RINSE THE TIP WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE TIP AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

TABLE 7-5
Checklist for 3535_PAH (SPE)

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE(Prep Code AND 8270_PAH_W/8270_SPEST_W)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : REMOVE THE SAMPLE BOTTLES FROM THE FRIDGE AND LET THEM REACH THE ROOM TEMPERATURE. CHECK PREP AND TEST INFO

____ : USE ORANGE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE BOTTLES

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : IF THERE ARE ANY SAMPLES THAT HAVE SEDIMENT ON THE BOTTOM, TRANSFER THE LIQUID PORTION INTO A CLEAN 1L AMBER GLASS BOTTLE AND LABEL IT.

____ : PUT 1000 ml DI WATER INTO 1L AMBER GLASS BOTTLE FOR MB AND LCS (AND LCSD IF THE SAMPLE AMOUNT IS INSUFFICIENT TO PREP MS/MSD). (IF ONLY 2 BOTTLES HAVE BEEN PROVIDED BY THE CLIENT, USE 500 ml FOR SAMPLE, MS AND MSD FOR THE QC SAMPLE)

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml OF 50 ppm PAH SPIKE AND ADD 1.0 ml OF 50 ppm PAH SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE) (USE 0.5 ml SPIKE AND SURROGATE IF YOU START WITH 500 ml SAMPLE)

____ : ADJUST THE PH OF EACH SAMPLE AND QC SAMPLE TO LESS THAN 2 BY ADDING 2-3 ml OF 1:1 HCl. CAP THE BOTTLES AND MIX WELL.

____ : LOAD THE SPEEDISK H_20-PHOBIC DVB (JT BAKER 8068-06) FILTER ONTO THE INSTRUMENT.

____ : REMOVE CAP FROM THE SAMPLE BOTTLES, PLACE A SMALL PIECE OF ALUMINUM FOIL OVER THE OPENING AND SCREW ON THE BOTTLE CAP ADAPTER.

____ : PLACE A CLEAN 40 ml VOA VIAL ONTO THE INSTRUMENT.

____ : LOAD THE 1L SAMPLE BOTTLES ONTO THE INSTRUMENT.

____ : LOAD METHOD 8270.1 AND PROCESS THE SAMPLES.

____ : REMOVE THE VOA VIAL WITH THE EXTRACT FROM THE INSTRUMENT.

____ : POUR EXTRACT INTO THE DRYDISK RESERVOIR AND OPEN THE STOPCOCK. ALLOW TO DRAIN ABOUT 30 SECONDS.

____ : CONCENTRATE THE SAMPLES IN THE TURBOVAP WITH SETTINGS AT 39°C AND 20 PSI

____ : CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP BEEPS

____ : RINSE THE ZYMARK TUBE WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO ANAUTOSAMPLER VIAL. RINSE THE ZYMARK TUBE AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

TABLE 7-6
Checklists for Extraction Procedures
3550_BNA / 3550_BNA_CLP

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3550_BNA / 3550_BNA_CLP AND 8270_TCL_S / 8270_TCL_S_CLP / 8270_PP_S)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (GLASS JARS, ZYMARK TUBES, FUNNELS AND SYRINGE)

____ : USE BLUE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : WEIGH 30 g OF SAMPLE INTO GLASS JARS. USE 30 g OF SODIUM SULFATE FOR MB AND LCS

____ : DRY THE SAMPLES WITH SODIUM SULFATE. SAMPLES SHOULD BE FREE FLOWING. TRY TO MINIMIZE THE PARTICLE SIZE AS MUCH AS POSSIBLE.

____ : ADD 100 ml METHYLENE CHLORIDE TO THE SAMPLES

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml BNA SPIKE AND ADD 1.0 ml BNA SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE) FOR SC SAMPLES USE BNA LCS/MS EXTENDED SPIKING SOLUTION, LCSSC

____ : SONICATE SAMPLES FOR 3 MINUTES WITH PULSE SETTING AT 0.5 SEC AND OUTPUT CONTROL AT 10 (FULL POWER). REPEAT SONICATION STEP 2 MORE TIMES USING 100 ML SOLVENT EACH TIME.

____ : FILTER EXTRACTIONS INTO ZYMARK TUBES THROUGH FILTER PAPER WITH SODIUM SULFATE. RINSE THE SAMPLES WELL WITH METHYLENE CHLORIDE AFTER THE SONICATIONS AND FILTER THAT TO THE ZYMARK TUBE ALSO.

____ : CONCENTRATE THE SAMPLES IN THE TURBOVAP WITH SETTINGS AT 39°C AND 20 PSI

____ : CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP BEEPS

____ : RINSE THE ZYMARK TUBE WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE ZYMARK TUBE AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

TABLE 7-7
Checklists for Extraction Procedures
3580A_BNA

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3580A_BNA AND 8270_TCL_X)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : USE BLUE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : WEIGH 1 g OF SAMPLE INTO LABELED BOROSILICATE CULTURE TUBES

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml BNA WASTE SPIKE AND ADD 1.0 ml BNA WASTE SURROGATE TO EACH SAMPLE AND QC SAMPLE. FOR SC SAMPLES USE BNA LCS/MS EXTENDED SPIKING SOLUTION, LCSSC.

____ : DILUTE THE SAMPLES TO 10.0 ml WITH METHYLENE CHLORIDE

____ : SHAKE OR PIPET STIR FOR 2 MINUTES AND ALLOW IT TO SETTLE. (IF THE SAMPLE HAS A BAD AND/OR NOT SEPARATE FROM THE SOLVENT FILTER IT THORUGH A FILTER PAPER WITH SODIUM SULFATE IN IT)

____ : TRANSFER 1.0 ml SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL AND CAP THE VIAL TIGHTLY. TRANSFER THE REST OF THE SAMPLE INTO A 10.0 ml VIAL FOR STORAGE

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

TABLE 7-8
3510_PAH

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE(3510_PAH AND 8270_PAH_W)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (SEPARATORY FUNNELS, K-D FLASKS AND TIPS, FUNNELS AND SYRINGE)

____ : USE ORANGE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : TRANSFER 1000 ml SAMPLE INTO SEPARATORY FUNNEL. ADD 1000 ml DI WATER FOR MB AND LCS (AND LCSD IF THE SAMPLE AMOUNT IS INSUFFICIENT TO PREP MS/MSD) (IF ONLY 2 BOTTLES HAVE BEEN PROVIDED BY THE CLIENT, USE 500 ml FOR SAMPLE, MS AND MSD FOR THE QC SAMPLE)

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml PAH SPIKE AND ADD 1.0 ml PAH SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE)

____ : ADD 60 ml METHYLENE CHLORIDE TO THE SAMPLE, CAP THE SEPARATORY FUNNEL AND SHAKE FOR ABOUT 2 MINUTES

____ : LET THE METHYLENE CHLORIDE SETTLE FOR ABOUT 2-3 MINUTES AND FILTER THE BOTTOM METHYLENE CHLORIDE PORTION THROUGH GLASS WOOL WITH SODIUM SULFATE INTO THE K-D FLASK

____ : REPEAT THE EXTRACTION 2 MORE TIMES WITH 60 ml METHYLENE CHLORIDE EACH

____ : ADD COUPLE OF BOILING CHIPS AND CONNECT A SNYDER COLUMN TO THE K-D FLASK

____ : CONCENTRATE THE SAMPLES IN WATERBATH AT TEMPERATURE 60-65°C. (MAKE SURE THE TIP IS TIGHTL Y CONNECTED TO THE K-D FLASK BEFORE PUTTING IN THE WATERBATH)

____ : CONCENTRATE THE SAMPLES DOWN TO AROUND 2.0-3.0 ml. BE CAREFUL TO TAKE THE K-D OUT BEFORE THE SAMPLE IS DRY. LET IT COOL FOR ABOUT 5 MINUTES BEFORE SEPARATING THE TIP

____ : CONCENTRATE TO ABOUT 1.0 ml AGAIN BY USING A MICRO-SNYDER COLUMN ON THE TIP (ADD ONE MORE BOILING CHIP TO THE TIP BEFORE STARTING CONCENTRATION)

____ : RINSE THE TIP WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE TIP AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

TABLE 7-9
3550_PAH / 3550_PAH_CLP (K-D)

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3550_PAH / 3550_PAH_CLP AND 8270_PAH_S / 8270_PAH_S_CLP)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (GLASS JARS, K-D FLASKS AND TIPS, FUNNELS AND SYRINGE)

____ : USE ORANGE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : WEIGH 30 g OF SAMPLE INTO GLASS JARS. USE 30 g OF SODIUM SULFATE FOR MB AND LCS

____ : DRY THE SAMPLES WITH SODIUM SULFATE. SAMPLES SHOULD BE FREE FLOWING. TRY TO MINIMIZE THE PARTICLE SIZE AS MUCH AS POSSIBLE.

____ : ADD 100 ml METHYLENE CHLORIDE TO THE SAMPLES

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml PAH SPIKE AND ADD 1.0 ml PAH SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE)

____ : SONICATE SAMPLES FOR 3 MINUTES WITH PULSE SETTING AT 0.5 SEC AND OUTPUT CONTROL AT 10 (FULL POWER). REPEAT SONICATION STEP 2 MORE TIMES USING 100 ML SOLVENT EACH TIME

____ : FILTER EXTRACTIONS INTO K-D FLASKS THROUGH FILTER PAPER WITH SODIUM SULFATE. RINSE THE SAMPLES WELL WITH METHYLENE CHLORIDE AFTER THE SONICATIONS AND FILTER THAT TO THE K-D FLASKS ALSO.

____ : CONCENTRATE THE SAMPLES IN WATERBATH AT TEMPERATURE 60-65°C. (MAKE SURE THE TIP IS TIGHTL Y CONNECTED TO THE K-D FLASK BEFORE PUTTING IN THE WATERBATH)

____ : CONCENTRATE THE SAMPLES DOWN TO AROUND 2.0-3.0 ml. BE CAREFUL TO TAKE THE K-D OUT BEFORE THE SAMPLE IS DRY. LET IT COOL FOR ABOUT 5 MINUTES BEFORE SEPARATING THE TIP

____ : CONCENTRATE TO ABOUT 1.0 ml AGAIN BY USING A MICRO-SNYDER COLUMN ON THE TIP (ADD ONE MORE BOILING CHIP TO THE TIP BEFORE STARTING CONCENTRATION)

____ : RINSE THE TIP WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE TIP AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-10
3510_PAH_SIM

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(3510_PAH_SIM AND 8270_SIM_PAH_W)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (SEPARATORY FUNNELS, K-D FLASKS AND TIPS, FUNNELS AND SYRINGE)

____ : USE ORANGE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : TRANSFER 1000 ml SAMPLE INTO SEPARATORY FUNNEL. ADD 1000 ml DI WATER FOR MB AND LCS (AND LCSD IF THE SAMPLE AMOUNT IS INSUFFICIENT TO PREP MS/MSD) (IF ONLY 2 BOTTLES HAVE BEEN PROVIDED BY THE CLIENT, USE 500 ml FOR SAMPLE, MS AND MSD FOR THE QC SAMPLE)

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml of 2 mg/L PAH SPIKE AND ADD 1.0 ml of 2 mg/L PAH SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE)

____ : ADD 60 ml METHYLENE CHLORIDE TO THE SAMPLE, CAP THE SEPARATORY FUNNEL AND SHAKE FOR ABOUT 2 MINUTES

____ : LET THE METHYLENE CHLORIDE SETTLE FOR ABOUT 2-3 MINUTES AND FILTER THE BOTTOM METHYLENE CHLORIDE PORTION THROUGH GLASS WOOL WITH SODIUM SULFATE INTO THE K-D FLASK

____ : REPEAT THE EXTRACTION 2 MORE TIMES WITH 60 ml METHYLENE CHLORIDE EACH

____ : ADD COUPLE OF BOILING CHIPS AND CONNECT A SNYDER COLUMN TO THE K-D FLASK

____ : CONCENTRATE THE SAMPLES IN WATERBATH AT TEMPERATURE 60-65°C. (MAKE SURE THE TIP IS TIGHTL Y CONNECTED TO THE K-D FLASK BEFORE PUTTING IN THE WATERBATH)

____ : CONCENTRATE THE SAMPLES DOWN TO AROUND 2.0-3.0 ml. BE CAREFUL TO TAKE THE K-D OUT BEFORE THE SAMPLE IS DRY. LET IT COOL FOR ABOUT 5 MINUTES BEFORE SEPARATING THE TIP

____ : CONCENTRATE TO ABOUT 1.0 ml AGAIN BY USING A MICRO-SNYDER COLUMN ON THE TIP (ADD ONE MORE BOILING CHIP TO THE TIP BEFORE STARTING CONCENTRATION)

____ : RINSE THE TIP WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE TIP AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-11
3520_PAH_SIM

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(3520_PAH_SIM AND 8270_PAH_SIM_W)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (LIQUID-LIQUID EXTRACTOR, ROUND FLASKS, K-D FLASKS AND TIPS, FUNNELS AND SYRINGE)

____ : USE ORANGE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : ADD 50 ml METHYLENE CHLORIDE TO THE SMALL ROUND FLASKS (100 ml FOR BIG FLASKS) AND ADD BOILING CHIPS TO THE FLASKS

____ : ADD 100 ml METHYLENE CHLORIDE TO THE LIQUID-LIQUID EXTRACTOR STEM (200 ml FOR THE STEMS THAT HAS THE CIRCULATION TUBE CONNECTED FROM THE SIDE) AND CONNECT THE ROUND FLASKS TO THE STEMS

____ : TRANSFER 1000 ml SAMPLE INTO LIQUID-LIQUID EXTRACTOR STEM. ADD 1000 ml DI WATER FOR MB AND LCS (AND LCSD IF THE SAMPLE AMOUNT IS INSUFFICIENT TO PREP MS/MSD) (IF ONLY 2 BOTTLES HAVE BEEN PROVIDED BY THE CLIENT, USE 500 ml FOR SAMPLE, MS AND MSD FOR THE QC SAMPLE)

____ : ADD DI WATER TO THE SAMPLES SO THAT THE LIQUID-LIQUID EXTRACTOR OPERATES (ADD DI WATER TILL YOU SEE SOLVENT CIRCULATING FROM THE STEM TO THE FLASK)

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml of 2 mg/L PAH SPIKE AND ADD 1.0 ml of 2 mg/L PAH SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE)

____ : EXTRACT FOR 18 HOURS (± 2 HOURS) (CHECK FOR SOLVENT LEVEL IN THE FLASKS, ADD MORE FROM THE TOP IF NECESSARY TO PREVENT THEM FROM DRYING OUT) (CHECK THE APPARATUS AFTER 1 HOUR TO MAKE SURE METHYLENE CHLORIDE IS CIRCULATING. YOU SHOULD SEE AT LEAST 2 DROPS PER SECOND)

____ : FILTER THE EXTRACTS IN THE ROUND FLASKS INTO K-D FLASKS THROUGH GLASS WOOL WITH SODIUM SULFATE. RINSE THE ROUND FLASKS AT LEAST 2 TIMES WITH METHYLENE CHLORIDE AND FILTER THEM TO K-D ALSO.

____ : ADD COUPLE OF BOILING CHIPS AND CONNECT A SNYDER COLUMN TO THE K-D FLASK

____ : CONCENTRATE THE SAMPLES IN WATERBATH AT TEMPERATURE 60-65°C. (MAKE SURE THE TIP IS TIGHTL Y CONNECTED TO THE K-D FLASK BEFORE PUTTING IN THE WATERBATH)

____ : CONCENTRATE THE SAMPLES DOWN TO AROUND 2.0-3.0 ml. BE CAREFUL TO TAKE THE K-D OUT BEFORE THE SAMPLE IS DRY. LET IT COOL FOR ABOUT 5 MINUTES BEFORE SEPARATING THE TIP

____ : CONCENTRATE TO ABOUT 1.0 ml AGAIN BY USING A MICRO-SNYDER COLUMN ON THE TIP (ADD ONE MORE BOILING CHIP TO THE TIP BEFORE STARTING CONCENTRATION)

____ : RINSE THE TIP WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE TIP AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

Table 7-12
3550_PAH _SIM

____ : PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (3550_PAH_SIM AND 8270_PAH_SIM_S)

____ : BATCH SAMPLES IN THE LIMS AND PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : ASSEMBLE THE PROPER GLASSWARE AND RINSE THEM WITH METHYLENE CHLORIDE (GLASS JARS, ZYMARK TUBES, FUNNELS AND SYRINGE)

____ : USE ORANGE LABELS TO WRITE THE SAMPLE INFO AND LABEL THE GLASSWARE

____ : PICK A SAMPLE FOR QC (MS AND MSD) AND HOMOGENIZE SAMPLE

____ : WEIGH 30 g OF SAMPLE INTO GLASS JARS. USE 30 g OF SODIUM SULFATE FOR MB AND LCS

____ : DRY THE SAMPLES WITH SODIUM SULFATE. SAMPLES SHOULD BE FREE FLOWING. TRY TO MINIMIZE THE PARTICLE SIZE AS MUCH AS POSSIBLE.

____ : ADD 100 ml METHYLENE CHLORIDE TO THE SAMPLES

____ : SPIKE LCS, MS AND MSD WITH 1.0 ml of 2 mg/L PAH SPIKE AND ADD 1.0 ml of 2 mg/L PAH SURROGATE TO EACH SAMPLE AND QC SAMPLE (USE GLASS SYRINGE)

____ : SONICATE SAMPLES 3 TIMES AT 3 MINUTE INTERVALS WITH PULSE SETTING AT 0.5 SEC. ADD 100 ml METHYLENE CHLORIDE BEFORE SECOND AND THIRD SONICATION

____ : FILTER EXTRACTIONS INTO ZYMARK TUBES THROUGH FILTER PAPER WITH SODIUM SULFATE. RINSE THE SAMPLES WELL WITH METHYLENE CHLORIDE AFTER THE SONICATIONS AND FILTER THAT TO THE ZYMARK TUBE ALSO.

____ : CONCENTRATE THE SAMPLES IN THE TURBOVAP WITH SETTINGS AT 39°C AND 20 PSI

____ : CONCENTRATE THE SAMPLE DOWN TO AROUND 1.0 ml, TURBOVAP BEEPS WHEN THE SAMPLE IS JUST A LITTLE LESS THAN 1.0 ml. REMOVE THE SAMPLE WHEN TURBOVAP BEEPS

____ : RINSE THE ZYMARK TUBE WITH SAMPLE AND TRANSFER THE SAMPLE ALIQUOT INTO AN AUTOSAMPLER VIAL. RINSE THE ZYMARK TUBE AND BRING THE FINAL VOLUME TO 1.0 ml WITH METHYLENE CHLORIDE AND CAP THE VIAL TIGHTLY

____ : LABEL THE VIALS WITH APPROPRIATE SAMPLE INFO

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP# QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability. Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Calibration of the GC/MS. This is accomplished through a 5-point calibration curve. Calibration criteria and calibration verification is discussed in detail in sections 7.10, 7.11, and 7.12.

8.1.3 Retention time window. The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

8.1.4 Method Detection Limit Study. The method detection limit is calculated by analyzing at minimum seven replicates prepared in blank water at 1 to 5 times higher than the estimated detection limit. Quantitation limits are laboratory derived from the MDL study data set. MDL's must be determined initially for each instrument and updated whenever instrument changes have been made that will affect the established MDLs. Actual MDLs are listed in Tables 5-7 and 5-8 in the AES QA Manual SOP QA-010000.

8.1.5 Method blank. Reagent blank analysis must be performed at the following frequency: Every twenty (20) samples of similar concentration and/or sample matrix or whenever samples are extracted and analyzed by the same procedure at same time, whichever is more frequent. The concentration of the method blank of any analyte of interest should not be exceeding the laboratory established practical quantitation limit (PQL).

8.1.6 Surrogate Recovery. All samples, blanks and QC samples are fortified with surrogate spiking compound before extraction and injection in order to monitor sample extraction efficiency. The recovery of the surrogate compound must be within the recovery limits established by the laboratory.

8.1.7 Laboratory Control Sample (LCS). The Laboratory Control Sample (LCS) is used to monitor, assess and document laboratory method performance and is performed on every batch or twenty samples, whichever occurs more frequently. The recovery of the analytes must meet established laboratory guidelines. For SC samples using BNA LCS Extended Spiking Solution, LCSSC %Recovery criteria, see Appendix I.

8.1.8 Sample spike and duplicate spike. Matrix spikes and matrix spike duplicates are used to determine the effect of the sample matrix on the recovery of analytes, and are analyzed for each analytical batch up to 20 samples. The recovery of the analytes must meet established laboratory guidelines.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are in SOP #QA-01000, "Quality Assurance Manual," in Section 5.8, "Procedures For Assessing Out-Of-Control Situations," and in Table 5-6, "Summary of Calibration and Quality Control Procedures for Various Test Types". These tables include corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance and any procedure deviations in appropriate logbooks.

9.0 HEALTH, SAFETY REQUIREMENTS AND SAMPLE DISPOSAL

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available (i.e., gloves, lab coats, goggles, and dust masks). Reference files of OSHA regulations and MSDS's are available to all personnel involved in the chemical analysis. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

9.2 The analyst shall observe all safety precautions provided in the laboratory safety program.

9.3 Work with any of these compounds in high concentrations should be performed in a hood. An NIOSH approved toxic gas respirator should be worn when the analyst handles very high concentrations of these toxic compounds.

9.4 Charcoal traps should be installed on the GC split vents of the instruments with split/splitless injectors to prevent airborne contamination of the work area. All vacuum pumps (Edwards) should contain vapor traps on the outlet pump manifold.

9.5 Sample extracts should be stored for 6 months after GC/MS analysis. Disposal of extracts should follow the laboratory waste disposal program outlined in the QA Manual.

10.0 DATA REPORTING

10.1 The LIMS system automatically rounds the data based upon factors that are set up for each test category. Typically, the LIMS reports to two significant figures.

10.2 The reporting limits can be changed in LIMS. Various laboratory personnel including project managers have the rights to change the limits per the requirements of the clients.

10.3 Calculation of results using Enviroquant software.

10.3.1 Open Enviroquant software by "clicking" on Icon.

10.3.2 On the pull-down menu, click on "Data Analysis".

10.3.3 On the pull-down menu, click on "Load", then "Data file". The path C:\hpchem\2\data should appear.

10.3.4 Using the "arrow" key, scroll down to the desired sequence number. The sequence number represents the GC number, year, month and day in a format 410321. Note that the left side of the box contains all of the samples in the run. Each sample can be selected by positioning the mouse pointer over the sample and double clicking it.

10.3.5 Once a sample has been selected, On the pull-down menu, click on "Load", then "Method". Select the method BMA15.01 where MA = the month, March, 15 = the date, and 01 = the year. From this point, the method will remain the same for each sample selection.

10.3.6 To calculate a result, on the pull-down menu, click on "Quant", then "Calculate and generate report". An alternative method is to click on "Quant", then "Int" and "Integrate".

10.3.7 Review the Enviroquant calculated result by clicking on "Quant", then "Q edit". To enlarge the various areas of the chromatogram, place the pointer on the chromatogram and right click the mouse. Drag the mouse over the area to enlarge. Double click the chromatogram to return it to its original size.

10.3.8 Once the chromatogram has been enlarged, the baselines can be redrawn by placing the mouse at one end of the peak and moving it across the bottom while holding down the right mouse. Release the mouse when the line is the correct length.

10.3.9 A general rule for drawing the baseline is that it should be started from the lowest side of the peak so that the baseline makes a "right angle" at the raised side of the peak..

10.3.10 Perform the same procedure on the rest of the chromatograms.

11.0 FILE MAINTENANCE

11.1 Data is stored in file boxes until the batch is complete. Once the batch is complete, all data is submitted to the V. P. of Technical Services for review. The data contained in the folders consists of the following:

DFTPP tune(s)
MB (Extracted)
CCV sample chromatograms and calculated results.
LCD/LCSD sample chromatograms and calculated results.
MS/MSD sample chromatograms and calculated results.
Actual sample chromatograms and calculated results.
Daily run log.

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto portable hard drives. Two copies are made. One copy is stored on the laboratory premises, the company President takes the other copy.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks must be completed each time that any maintenance is performed upon the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as replacement of motors in tower autosamplers.

12.2 Each instrument logbook must have a cover page that includes the following information.

Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.3 Routine maintenance: Typical routine maintenance consists of keeping the system clean and insuring that chromatography remains acceptable. Examples would be peak tailing and degradation of DDT and Dieldrin in pesticide analyses.

The table below indicates the frequency of routine maintenance for various instrument types within the laboratory.

Maintenance Action	Recommended Frequency
Changing injection port liners	Weekly or when chromatography is affected
Trimming column	Weekly or when chromatography is affected
Cleaning GC/MS source	Semi-annually or when chromatography is affected
Changing GC Column	Annually or when other attempts to resolve chromatography fail

12.4 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as autosampler that will not function. If this type of problem occurs, contact the department manager for assistance.

13.0 METHOD PERFORMANCE

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The reporting limit RL is defined as the concentration of a substance that is above the level of uncertainty. The concentration listed in the table in Section VIII was obtained using reagent water. Similar results can be achieved using representative wastewater. The MDL actually achieved is a given analysis will vary depending on instrument sensitivity and matrix effects.

13.2 This method is recommended for use in the concentration range from the MDL to 1000 x MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000 x MDL.

13.3 20 laboratories using reagent water, drinking water, surface water tested this method and three industrial wastewater spiked at six concentrations over the range 8.0 to 500 μg/l. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.1 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.2 Wastes with other pH levels may be directly discharged into the sinks.

14.3.3 Further information related to the disposal of wastes is contained in the QA Manual.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 GC/MS – Gas chromatography / mass spectrometer

15.2 MDL – Method detection limit

15.3 PCB – Polychlorinated biphenyls

15.4 DFTPP – Standard solution containing 50 µg / ml solution of DFTPP in methylene Chloride

15.5 Matrix spiking standard – matrix spiking standards containing 100 µg/ml of each acid analyte and 50 µg/ml of each basic analyte in acetone

15.6 RF – response factor

15.7 RSD – Relative standard deviation

15.8 LCS – Laboratory Control Sample. A known sought for analyte is added to distilled water on clean soil and the concentration is measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits. An LCSD may be analyzed in the absence of MS/MSD.

15.9 MS - Matrix Spike procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 %.

15.10 Tuning a GC/MS – The physical procedure in which instrument conditions are varied so that the DFTPP injection will pass the method criteria.

15.11 EICP – Extracted Ion Current Profile

16.0 REFERENCES

16.1 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 8270D, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)", Revision 4, February 2007.

16.2 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 8000B, "Determinative Chromatographic Separations", Revision 2, December 1996.

16.3 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3500C, "Sample Preparation for Organic Semi-Volatile Compounds", Revision 3, February 2007.

16.4 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction", Revision 3, December 1996.

16.5 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3520C, "Continuous Liquid-Liquid Extraction", Revision 3, December 1996.

16.6 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3550C, "Ultrasonic Extraction", Revision 3, February 2007.

16.7 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3540C, "Soxhlet Extraction", Revision 3, December 1996.

16.8 SW846 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3580A, "Waste Dilution", Revision 1, July 1992.

16.9 USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organics Analysis, Exhibit D, Analytical Method for the Analysis of Semivolatile Organic Compounds, SOM01.1, 8/2008.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://portal/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://portal/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

18.0 SOP REVISION HISTORY

Revision Date	Revision #	Summary of and Reason for Changes/Updates	Responsible for Revision
10/9/2003	4	Update	Greg Jones
12/13/2005	5	Update	Greg Jones
12/14/2006	6	Update	Greg Jones
3/9/2009	7	SC/MUR Update	Dana Till
11/23/2010	8	Updates to Sections 5.8, Table 5-3, 5.21, 5.22, Table 5-8, 7.1.8, Table 7-10, Table 7-11, and Table 7-12. SC SIM PAH update.	Dana Till
2/13/2012	9	SC Audit Response, Update to Sections 5.20.2, 7.2.4, 7.3.8, 7.5.10, 7.18.1, 7.12.4.4, 8.1.7, and Tables 5-8, 7-4, 7-6, and 7-7. Added Sections 18.0 and APPENDIX I.	Dana Till

APPENDIX I
% Recovery Criteria for LCSSC, AQUEOUS

Spike Compound	%Recovery Criteria
1,2-Dichlorobenzene*	50 – 130%
1,2-Diphenylhydrazine	70 – 130%
1,3-Dichlorobenzene	70 – 130%
1,4-Dichlorobenzene	70 – 130%
2,4,5-Trichlorophenol	70 – 130%
2,4,6-Trichlorophenol	70 – 130%
2,4-Dichlorophenol	70 – 130%
2,4-Dimethylphenol	70 – 130%
2,4-Dinitrotoluene	70 – 130%
2,6-Dichlorophenol	70 – 130%
2,6-Dinitrotoluene	70 – 130%
2-Chlorophenol	70 – 130%
2-Methylphenol	70 – 130%
3,3'-Dichlorobenzidine	70 – 130%
4-Bromophenyl ether	70 – 130%
4-Chloro-3-methylphenol	70 – 130%
4-Methylphenol	70 – 130%
Acenaphthene	70 – 130%
Acenapthylene	70 – 130%
Anthracene	70 – 130%
Benzo(a)anthracene	70 – 130%
Benzo(a)pyrene	70 – 130%
Benzo(b)fluoranthene	70 – 130%
Bis(2-chloroethoxy)methane	70 – 130%
Bis(2-cloroethyl)ether	70 – 130%
Bis(2-chloroisopropyl)ether	70 – 130%
Bis(2-ethylhexyl)phthalate	70 – 130%
Chrysene	70 – 130%
Di-n-butyl phthalate	70 – 130%
Di-n-octyl phthalate	70 – 130%
Dibenzo(a,h)anthracene	70 – 130%
Diethyl phthalate	70 – 130%
Dimethyl phthalate	70 – 130%
Diphenylamine	70 – 130%
Fluoranthene	70 – 130%
Fluorene	70 – 130%
Hexachlorobenzene	70 – 130%
Hexachlorobutadiene	70 – 130%
N-Nitrosodiphenylamine	40 – 130%
Naphthalene	70 – 130%
Nitrobenzene	70 – 130%
Pentachloronitrobenzene	70 – 130%
Phenacetin	70 – 130%
Pronamide	70 – 130%
Pyrene	70 – 130%

*Compound has interim limits until historical data may be generated.

% Recovery Criteria for LCSSC, SOIL

Spike Compound	%Recovery Criteria
1,2-Dichlorobenzene*	50 – 130%
1,2-Diphenylhydrazine	70 – 130%
1,3-Dichlorobenzene*	50 – 130%
1,4-Dichlorobenzene*	50 – 130%
2,4,5-Trichlorophenol	70 – 130%
2,4,6-Trichlorophenol	70 – 130%
2,4-Dichlorophenol	70 – 130%
2,4-Dimethylphenol	70 – 130%
2,4-Dinitrotoluene	70 – 130%
2,6-Dichlorophenol	70 – 130%
2,6-Dinitrotoluene	70 – 130%
2-Chlorophenol	70 – 130%
2-Methylphenol	70 – 130%
3,3'-Dichlorobenzidine*	40 – 130%
4-Bromophenyl ether	70 – 130%
4-Chloro-3-methylphenol*	50 – 130%
4-Methylphenol	70 – 130%
Acenaphthene	70 – 130%
Acenapthylene	70 – 130%
Anthracene	70 – 130%
Benzo(a)anthracene	70 – 130%
Benzo(a)pyrene	70 – 130%
Benzo(b)fluoranthene	70 – 130%
Bis(2-chloroethoxy)methane	70 – 130%
Bis(2-cloroethyl)ether	70 – 130%
Bis(2-chloroisopropyl)ether	70 – 130%
Bis(2-ethylhexyl)phthalate	70 – 130%
Chrysene	70 – 130%
Di-n-butyl phthalate	70 – 130%
Di-n-octyl phthalate	70 – 130%
Dibenzo(a,h)anthracene	70 – 130%
Diethyl phthalate	70 – 130%
Dimethyl phthalate	70 – 130%
Diphenylamine*	50 – 130%
Fluoranthene	70 – 130%
Fluorene	70 – 130%
Hexachlorobenzene	70 – 130%
Hexachlorobutadiene	70 – 130%
N-Nitrosodiphenylamine*	40 – 130%
Naphthalene	70 – 130%
Nitrobenzene	70 – 130%
Pentachloronitrobenzene	70 – 130%
Phenacetin	70 – 130%
Pronamide	70 – 130%
Pyrene	70 – 130%

*Compound has interim limits until historical data may be generated.

APPENDIX C

FIELD SAMPLING AND ANALYSIS PLAN

FIELD SAMPLING AND ANALYSIS PLAN
REVISION 4.0

FORMER VERMONT BOSCH SITE
FOUNTAIN INN, SOUTH CAROLINA

Prepared for:

ROBERT BOSCH TOOL CORPORATION

1800 West Central Road
Mount Prospect, Illinois 60056

Prepared by:

AMEC Environment & Infrastructure, Inc.
555 North Pleasantburg Drive, Suite 202
Greenville, South Carolina

AMEC Project 6251121007.01.01

May 31, 2012

TABLE OF CONTENTS

Page

TABLES

FIGURES

1.0 INTRODUCTION

This Field Sampling and Analysis Plan (FSAP) has been prepared to describe the proposed remedial investigation (RI) activities at the Former Vermont Bosch Site (Site) located in Fountain Inn, South Carolina. The FSAP has been prepared by AMEC Environment & Infrastructure, Inc. (AMEC), formerly MACTEC Engineering and Consulting, Inc. (MACTEC), on behalf of Robert Bosch Tool Corporation (RBTC), as required by the Voluntary Cleanup Contract (VCC) #05-5613-RP, executed on August 29, 2005, and in accordance with the United States Environmental Protection Agency (USEPA) "Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA" (USEPA, 1988).

This FSAP presents the details associated with the site assessment activities to be performed during the RI. The following sections describe the proposed RI sampling program including sampling rationale, the locations, number and types of samples to be collected, and the analyses that will be performed. The general procedures for field sample collection and analysis, field documentation, RI investigation procedures, sample labeling, sample custody and data management are described in the project Quality Assurance Project Plan (QAPP) which is contained as Appendix B in the RI/FS Work Plan. When procedural information is provided in the FSAP, its inclusion is intended as a site-specific supplement to QAPP procedure. If site-specific sample collection requirements described in the FSAP conflict with the general requirements in the QAPP, the site-specific FSAP procedure will supersede the QAPP requirement for that activity.

2.0 SAMPLING OBJECTIVES

As defined in the VCC, the purpose of the proposed assessment activities is to determine the source, nature, and extent of contaminated media (soil, groundwater, surface water, and sediment) present at the Site.

3.0 WATER SUPPLY WELL INVENTORY

The first RI activity to be performed will be conducting an inventory of existing water supply wells located within a one-mile radius of the Site. Doing so will identify residences or businesses in the area that may be using water supply wells. The survey will include determining the water uses and well construction details if such information is readily available.

The proposed survey activities will consist of the following tasks:

- Contact the provider of potable water to Fountain Inn, South Carolina residents to obtain a map (if available) illustrating the availability of public water supply connections in the area, and to see if they can provide a listing of the customer names and/or addresses within one-mile of the Site.

- Perform a drive-by inspection within a one-mile radius of the site boundary to potentially identify homes and businesses utilizing a private water supply.

- Obtain available water supply well records for potential supply wells within one-mile of the Site from the South Carolina Department of Health and Environmental Control (SCDHEC).

- For each water well identified from the procedure above, prepare a list that contains the following information:

  - The general location of the well.

  - The address of the subject property (or the tax map number) and the name and address of the property owner, if available.

  - The use of the property as identified through the drive-by inspection or property records.

4.0 SAMPLING RATIONALE AND LOCATIONS

This section describes the second part of the field investigation which will include sampling of soil, groundwater, pore water, surface water, and sediment from the applicable AOCs to develop information on the nature and distribution of potential contaminants at the Site. These activities shall be performed in accordance with the Remedial Investigation/Feasibility Study (RI/FS) Work Plan, FSAP, QAPP, Health and Safety Plan (HASP), and in general accordance with the United States Environmental Protection Agency (USEPA), Region IV, Science and Ecosystem Support Division (SESD), "*Field Branches Quality System and Technical Procedures*."

The field activities include the collection of samples within identified AOCs. The sampling approach includes the collection of discrete samples. The RI program includes collection of three (3) surface soil samples, fifty (50) subsurface soil samples, fourteen (14) pore water samples, thirteen (13) surface water samples, nine (9) sediment samples, and 24 groundwater samples from within specific AOCs. Of the 24 groundwater samples, 23 will be collected from newly installed monitoring wells and one sample will be collected from existing monitoring well MW-1. The proposed sampling locations are shown on Figure 8 of the RI/FS Work Plan and are described in additional detail below. Sample locations will be marked in the field using flagging, a pin-flag, or a wooden stake for survey.

4.1 SURFACE SOIL SAMPLING

Surface soil samples will be collected to characterize the concentrations of constituents in soils adjacent to the northernmost storm water outfall (AOC #7 Storm Water Outfalls). Surface soil sampling procedures are described in Section B2 (page B-2) of the QAPP.

Storm water Outfall 002 is located in the northern portion of the site and discharges directly to a concrete drainage way that is situated perpendicular to the outfall. The concrete drainage way also receives discharges from the parking area of a baseball field located north of the site and from Woodside Avenue located northeast of the site. Since the outfall discharges directly to the concrete drainage way, no soil is present immediately downstream from the outfall. The soil downstream of the termination of the concrete drainage way was assessed during previous environmental investigations by collecting a surface soil sample.

Three surface soil samples (SS-07-01 through SS-07-03) will be collected at the locations shown on Figure 8 of the RI/FS Work Plan. Since the storm water outfall discharges directly to the concrete drainage way, an attempt will be made to collect a soil sample from the inside of outfall prior to its discharge point. If no soil is present within the outfall, then a soil sample will be collected downstream of the soil sample collected from the previous environmental investigation. Two surface soil samples will be collected upstream of the outfall at locations near the parking area near the baseball field and Woodside Avenue. The soil samples will be analyzed for polynuclear aromatic hydrocarbons (PAHs).

4.2 SUBSURFACE SOIL SAMPLING

Subsurface soil samples will be collected to characterize the concentration of constituents in soils from the specific AOCs as identified below. Subsurface soil sampling procedures are described in Section B2 (pages B-2 to B-3) of the QAPP.

4.2.1 AOC #4 Former Scrap Metal Rolloff

The Scrap Metal Rolloff is located adjacent to an asphalt driveway located to the southwest of the facility building. The Scrap Metal Rolloff received scrap steel from hydraulic press operations, metal swarf from grinding operations, and spent media from grit blast operations. Stained soil and asphalt were documented in previous environmental investigations and four soil samples were collected from the areas of stained soil. The visibly stained soils were excavated and four soil samples were collected from the base of the excavation at the approximate location of the previous soil samples. The excavation was subsequently backfilled with clean soil.

Three borings will be performed in the previously excavated area with a Geoprobe® direct-push drill rig or other similar equipment. The boring locations are identified as SB-04-01 through SB-04-03 on Figure 8 of the RI/FS Work Plan. The borings will be advanced from the ground surface to approximately four feet below ground surface (bgs). Continuous samples will be collected in four foot Macrocore® tubes. One composite soil sample will be collected from each boring from a depth of two to four feet bgs. The soil

samples will be analyzed for Target Compound List (TCL) volatile organic compounds (VOCs) and TCL semi-volatile organic compounds (SVOCs).

4.2.2 AOC #6 Compounding Room Blower Exhaust

The Compounding Room Blower Exhaust is located along the southwest side of the facility building. Exhaust vapors from the Compounding Room were observed to condense on the piping near the exhaust vents and drip on the ground. Two surface soil samples were collected from the areas where condensate dripped on the soil in previous environmental investigations.

Two borings will be performed near the previous sampling areas with a Geoprobe® direct-push drill rig or other similar equipment. The boring locations are identified as SB-06-01 and SB-06-02 on Figure 8 in the RI/FS Work Plan. The borings will be advanced from the ground surface to approximately four feet below bgs. Continuous samples will be collected in four foot Macrocore® tubes. Two composite soil samples will be collected from each boring from depths of one to two feet bgs and two to four feet bgs. The soil samples will be analyzed for TCL VOCs and TCL SVOCs.

4.2.3 AOC # 8 Former Oil/Water Separator Area

The former Oil/Water Separator was located below grade on the southeast side of the facility building. The separator was connected to the sanitary sewer discharge line from facility and received wastewater from floor drains inside the building (See Figure 17 of the RI/FS Work Plan). The subsurface soils adjacent to the separator were assessed during previous environmental investigations by collecting a soil sample on each side of the separator from the approximate depth of the bottom of the separator. The separator was subsequently removed and contaminated soils were excavated from around the separator to the apparent depth of groundwater in the area. The excavation was subsequently backfilled with clean soil.

Eight borings will be performed around the former location of the oil/water separator with a Geoprobe® direct-push drill rig or other similar equipment. The boring locations are identified as SB-08-01 through SB-08-08 on **Figure 1** of the FSAP and Figure 8 of the RI/FS Work Plan. The borings will be advanced from the ground surface to just above the

depth of the water table. The depth to the water table will be determined by obtaining a water table depth from monitoring well MW-1. Continuous samples will be collected from four foot Macrocore® tubes for lithologic description. Two discrete soil samples will be collected from each boring; one at the approximate depth of the base of the former oil/water separator and one from above the depth of the water table surface. The soil samples will be analyzed for TCL VOCs and TCL SVOCs.

4.2.4 AOC #9 Former Hazardous Waste Accumulation Building

The Former Hazardous Waste Storage Accumulation Building is located to the southwest of the facility building near the southwest property boundary. Various hazardous and non-hazardous wastes were accumulated in the building prior to being picked up for disposal. The soils at this AOC were assessed by collecting soil samples from beneath the concrete floor of the building. The former Hazardous Waste Accumulation Building is scheduled to be demolished prior to the start of the assessment.

Six borings will be performed within the footprint of the former Hazardous Waste Accumulation Building with a Geoprobe® direct-push drill rig or other similar equipment. The boring locations are identified as SB-09-01 through SB-09-06 on Figure 8 of the RI/FS Work Plan. The borings will be advanced from the ground surface to just above the depth of the water table. Continuous samples will be collected from four foot Macrocore® tubes for lithologic description. The soil samples will be screened for the presence of organic vapors using either a flame-ionization detector (FID) or photo-ionization detector (PID). One discrete soil sample will be collected for laboratory analysis from each four-foot sample interval. The soil samples will be analyzed for TCL VOCs and TCL SVOCs.

4.3 GROUNDWATER SAMPLING

Groundwater samples will be collected to characterize the concentration of constituents in groundwater from the specific AOCs as identified below. Borehole completion procedures and monitoring well installation procedures are described in Section B2 (pages B-6 to B-12) of the QAPP. Proposed monitoring well locations are shown on **Figure 2**. A summary of the monitoring well construction specifications is shown on **Table 1**. A proposed monitoring well construction diagram is provided as **Figure 3**.

4.3.1 AOC #2 Heat Treat Cleaning Water Disposal Area

The Heat Treat Cleaning Water Disposal Area is located near the northwest side of the facility building. The heat treat process consisted of a quench tanks and two rinse tanks. The quench tank held about 2,200 gallons of quench salt identified as Temper A Pink W/O that contained potassium nitrate, sodium nitrate, and sodium nitrite. Each rinse tank held about 1,150 gallons of water. Periodically, the quench tank and rinse tanks were cleaned and waste cleaning water and rinse water were discharged to the ground outside the heat treat area to the northwest of the facility building.

Stressed vegetation was documented during previous environmental investigations and 14 surface samples were collected to assess the horizontal extent of nitrate and nitrite contamination and seven soil test borings were conducted to assess the vertical extent of nitrate and nitrite contamination. One soil test boring was drilled as a control boring located on the opposite side of the facility from the Heat Treat Cleaning Water Disposal Area. Nitrate was detected in five of the 14 surface soil samples but nitrite was not detected in any of the surface soil samples. Both nitrate and nitrite were detected in the subsurface soil samples. Two temporary monitoring wells were installed and groundwater samples were collected to assess the nitrate and nitrite in Site groundwater. Both nitrate and nitrite were detected in the groundwater samples at concentrations below their respective MCLs. The areas with the highest concentrations of nitrate were excavated and disposed. The excavation was backfilled with clean soil.

The area with the highest concentrations of nitrate on the surface was excavated down to a depth of approximately one foot below ground surface (see limits of excavation on Figure 10) and to five feet below ground surface at the location of one soil test boring that had elevated nitrate concentrations at five feet below ground surface (see limits of excavation on Figure 10). Following completion of the excavation activities, eight grab soil samples were collected from the base of the excavation. Nitrate was detected in four of the eight samples and nitrite was detected in three of the samples. The excavation was subsequently backfilled with clean soil.

One shallow overburden monitoring well (MW-24 on **Figure 2**) will be installed downgradient from previous soil sample SS-6. A groundwater sample will be collected

from the monitoring well (MW-02-24 on Figure 8 of the RI/FS Work Plan) and analyzed for Target Analyte List (TAL) metals and TCL VOCs. Groundwater sampling procedures are described in Section B2 (pages B-12 to B-14) of the QAPP. For informational purposes, field measurements of pH, conductivity, temperature, and turbidity will be collected from the monitoring well.

4.3.2 AOC #3 Former Metals Baghouse

The Metals Baghouse is located along the northwest side of the facility building. The Metals Baghouse was used to collect dust from grinding and grit blasting operations for screwdriver blades, spade bits, and nut drivers. Stained soil was documented during previous environmental investigations and three soil samples were collected from the areas of stained soil. Potential contaminants are metals.

Two shallow overburden monitoring wells (MW-20 and MW-21 on **Figure 2**) will be installed at or near the water table surface near the location of the Former Metals Baghouse. Groundwater samples will be collected from each monitoring well (MW-03-20 and MW-03-21 on Figure 8 of the RI/FS Work Plan) and analyzed for TAL metals. Additionally, groundwater sample MW-03-20 will be analyzed for TCL VOCs and TCL SVOCs. Groundwater sampling procedures are described in Section B2 (pages B-12 to B-14) of the QAPP. For informational purposes, field measurements of pH, conductivity, temperature, and turbidity will be collected from each monitoring well.

4.3.3 AOC #4 Former Scrap Metal Rolloff

Two shallow overburden monitoring wells (MW-22 and MW-23 on **Figure 2**) will be installed at or near the water table surface in the excavation area of the Former Scrap Metal Rolloff. Groundwater samples will be collected from each monitoring well (MW-04-22 and MW-04-23 on Figure 8 of the RI/FS Work Plan) and analyzed for TAL metals. Groundwater sampling procedures are described in Section B2 (pages B-12 to B-14) of the QAPP. For informational purposes, field measurements of pH, conductivity, temperature, and turbidity will be collected from each monitoring well.

4.3.4 AOC # 8 Former Oil/Water Separator Area

A groundwater sample collected from monitoring well MW-1 detected concentrations of VOCs and SVOCs. Field screening groundwater samples from temporary monitoring wells installed downgradient from this AOC, both shallow (at or near the water table surface) and deep (depth of direct-push drilling equipment refusal), did not detect concentrations of VOCs or SVOCs.

One deep overburden monitoring well will be installed to the depth of hollow-stem auger refusal adjacent to existing monitoring well MW-1. The well is identified as MW-2D on **Figure 1** of this FSAP and Figure 8 of the RI/FS Work Plan and a groundwater sample (MW-08-02 on Figure 8 of the RI/FS Work Plan) will be collected from the well and analyzed for VOCs, SVOCs, and Total Petroleum Hydrocarbons-Diesel Range Organics (TPH-DRO). Groundwater sampling procedures are described in Section B2 (pages B-12 to B-14) of the QAPP.

Three shallow overburden monitoring wells (MW-3, MW-4, and MW-5 on **Figure 1** of this FSAP and Figure 8 of the RI/FS Work Plan) will be installed at or near the water table surface downgradient from the former location of the oil/water separator. Groundwater samples will be collected from each monitoring well (MW-08-03, MW-08-04, and MW-08-05 on Figure 8 of the RI/FS Work Plan) as well as from the existing monitoring well (MW-08-01 on Figure 8 of the RI/FS Work Plan) and analyzed for VOCs, SVOCs, and TPH-DRO. Groundwater sampling procedures are described in Section B2 (pages B-12 to B-14) of the QAPP. For informational purposes, field measurements of pH, conductivity, temperature, dissolved oxygen (DO), and oxidation-reduction potential (ORP) will be collected from each monitoring well.

4.3.5 AOC #9 Former Hazardous Waste Accumulation Building

VOCs were detected in the field-screening groundwater samples collected immediately downgradient from this AOC. VOCs were also detected in field-screening groundwater samples collected from the adjacent former Sherwin-Williams property, which is downgradient from the former Hazardous Waste Accumulation Building. It is noted that the Sherwin-Williams property is not believed to be a source of VOC contamination and the Sherwin-Williams property is only impacted by contaminated groundwater resulting

from the release at the former Hazardous Waste Accumulation Building located on the Site.

Nine shallow (at or near the water table surface) overburden monitoring wells (MW-6, MW-7, MW-9 through MW-11, MW-13 through MW-15, and MW-17 on **Figure 2**) and five deep (depth of hollow-stem auger refusal) overburden monitoring wells (MW-8D, MW-12D, MW-16D, MW-18D, and MW-19D on **Figure 2**) will be installed at this AOC. MW-9 will be installed as a background monitoring well to AOC #9. MW-7 and MW-8D will be installed within the footprint of the former Hazardous Waste Accumulation Building and the remainder of the wells will be installed downgradient from the building. Groundwater samples will be collected from each monitoring well (MW-09-06 through MW-09-19 on Figure 8 of the RI/FS Work Plan) and analyzed for VOCs. Groundwater sampling procedures are described in Section B2 (pages B-12 to B-14) of the QAPP. For informational purposes, field measurements of pH, conductivity, temperature, DO, and ORP will be collected from each monitoring well.

4.4 PORE WATER SAMPLING

Fourteen pore water samples (PW-09-01 through PW-09-14 on Figure 8 of the RI/FS Work Plan) will be collected from the bank of the unnamed tributary to Stoddard Creek. Pore water sampling procedures are described in Section B2 (pages B-5 to B-6) of the QAPP. The pore water samples will be screened for the presence of chlorinated solvents using the Color-Tec method, which is described in Section B4 (pages B-22 to B-24) of the QAPP. Up to eight pore water samples will be analyzed for VOCs in a fixed-base laboratory. Field measurements of pH, conductivity, temperature, DO, and ORP will be measured in each pore water sample.

4.5 SURFACE WATER AND SEDIMENT SAMPLING

Thirteen surface water and nine sediment samples will be collected from the unnamed tributary to Stoddard Creek. Sediment and surface water sampling procedures are described in Section B2 (pages B-3 to B-5) of the QAPP. Figure 8 of the RI/FS Work Plan illustrates the proposed surface water sampling locations (SW-09-01 through SW-09-013) and sediment sampling locations (SD-09-01 through SD-09-09). The surface water samples will be screened for the presence of chlorinated solvents using the Color-Tec

method, which is described in Section B4 (pages B-22 to B-24) of the QAPP. Up to nine of the surface water samples and all of the sediment samples will be analyzed for VOCs in a fixed-base laboratory. Field measurements of pH, conductivity, temperature, DO, and ORP will be collected from each surface water sampling location.

4.6 HYDRAULIC CONDUCTIVITY (SLUG) TESTING

Hydraulic conductivity (slug) tests will be performed in monitoring wells MW-1, MW-3, and MW-4 at AOC #8 and MW-7, MW-11, MW-15, MW-17, and MW-19D at AOC #9. The locations of the monitoring wells are shown on **Figure 2**. The hydraulic conductivity (slug) testing procedures are described in Section B2 (page B-15) of the QAPP.

5.0 SAMPLE EQUIPMENT AND HANDLING PROCEDURES

The sampling will be performed in accordance with procedures described in the QAPP and in general conformance with the USEPA Region IV, SESD, *Field Branches Quality System and Technical Procedures*.

The QAPP contains specific procedures for the collection of soil, groundwater, surface water, and sediment samples. The QAPP also contains specific procedures and requirements for data management which includes a format for unique sample identification numbers, initiating the sample custody process, and preparing field documentation on each sample collected. The field documentation includes use of field logbooks to describe and document the sequence of daily activities on-site and field data records to record observations and field measurements for each sample collected. These procedures, as well as data quality objectives, and details concerning the analytical program, including quality assurance/quality control (QA/QC) sample requirements are described in Section A7 (pages A-20 to A-26) of the QAPP.

Sampling related procedures contained in this FSAP are in addition to those contained in the QAPP.

5.1 SUBSURFACE SOIL SAMPLING PROCEDURE

The collection of subsurface soil samples includes samples to be collected at the Former Oil/Water Separator (AOC #8) and the Former Hazardous Waste Accumulation Building (AOC#9). The samples from the Former Oil/Water Separator will be analyzed for TCL VOCs and TCL SVOCs. The samples from the Former Hazardous Waste Accumulation Building will be analyzed for TCL VOCs. The subsurface soil samples will be collected in accordance with procedures described in Section B2 (pages B-2 to B-3) of the QAPP. The method of analysis, the sample bottle, sample preservation and sample hold time requirements are specified in Table B-2 of the QAPP. The method for collection and preservation of VOC samples by SW-846 Method 5035A is described in Section 6.1.2.

5.2 GROUNDWATER SAMPLING PROCEDURE

Groundwater samples will be collected in accordance with procedures described in Section B2 (pages B-12 to B-14) of the QAPP. The analytical methods, the sample bottle, sample preservation and sample hold time requirements are specified in Table B-2 of the QAPP.

5.3 SURFACE WATER AND SEDIMENT SAMPLING PROCEDURE

The sediment and surface water samples will be collected in accordance with procedures described in Section B2 (pages B-3 to B-5) of the QAPP. The analytical methods, the sample bottle, sample preservation and sample hold time requirements are specified in Table B-2 of the QAPP.

5.4 FIELD DOCUMENTATION SAMPLE NUMBERING

During sampling activities, field data will be documented in a field logbook and a field data record form. The use and content of the field logbook is described in Section A9 (pages A-27 to A-28) and Section B3 (pages B-20 to B-22) of the QAPP. The field data records are described in Section A9 (pages A-27 to A-28) of the QAPP.

Field monitoring equipment will be calibrated in accordance with manufacturer's procedures and the calibration results will be documented in a field data record.

A unique sample number will be used for each sample collected. The sample number code is described in Section B3 (pages B-18 to B-20) of the QAPP. These codes include identification of field samples and samples for QA/QC samples.

Sample labels will be prepared for each sample, which will include the sample number, the sample bottle and preservation requirements, analytical method(s), sample date, time, and sampler initials. Sample chain of custody procedures are described in Section B3 (page B-21) of the QAPP.

6.0 SAMPLE PRESERVATION AND ANALYSIS

A summary of the proposed samples, sample type, sample media and number of samples is presented in **Table 2**.

Based on previous data and the nature of the Site activities, the sampling plan will focus on VOCs and SVOCs.

6.1 SAMPLE ANALYSIS

Sample containers will be laboratory provided, cleaned, and prepared. Tables A-4 to A-6 of the QAPP present a summary of the analytical parameters associated with each of the proposed methods and the practical quantitation limit (PQL) associated with each respective compound. The sample container and preservation requirements are provided in Table B-2 of the QAPP.

6.1.1 Surface Soil Analytical Procedures

Surface soil samples will be analyzed in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods. Surface soil samples will be prepared and analyzed following USEPA Methods 3550B and 8270D for PAHs, a subset of SVOCs

6.1.2 Subsurface Soil and Sediment Analytical Procedures

Each subsurface soil and sediment sample for VOC analysis will be collected in accordance with SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 5035A as described in Section B4 (page B-24) of the QAPP. Method 5035A will include two clear 40-milliliter (ml) vials containing five ml of organic free reagent water for low concentration VOCs, one amber 40-ml vial with five ml of methanol for high concentration VOCs, and one 40-ml vial with no preservatives for screening and dry weight determination. The sample vials will be pre-prepared, weighed, and sealed by the laboratory. The soil samples will be collected using a disposable plastic syringe calibrated to obtain five grams of soil. The soil samples collected with the syringe will be immediately placed in the sample vials and capped.

The samples will be analyzed in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Subsurface soil and sediment samples will be prepared and analyzed following USEPA Methods 5035A and 8260B for VOCs and 3550B and 8270D for SVOCs.

6.1.3 Groundwater Sample Analytical Procedure

Groundwater will be analyzed in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Groundwater samples will be prepared and analyzed following USEPA Methods 8260B for VOCs, 8270D for SVOCs, 6010C/7470A for TAL metals, and 8015C for TPH-DRO.

6.1.4 Pore Water Sample Analytical Procedure

Pore water will be analyzed in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Pore water samples will be prepared and analyzed following USEPA Methods 8260B for VOCs.

6.1.5 Surface Water Analytical Procedures

Surface water will be analyzed in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Surface water samples will be prepared and analyzed following USEPA Methods 8260B for VOCs.

6.2 FIELD QUALITY ASSURANCE AND QUALITY CONTROL SAMPLES

Specific details of the project QA/QC are presented in the QAPP. The QC checks include the introduction of control samples into the sample analysis process in an effort to evaluate the accuracy, and precision of the sampling and analysis program.

Specific QC measures are developed for the field to help assure data quality falls within the parameters and guidelines in order to meet the intended data quality objectives (DQOs). Specifically, as described in Section 4.0 of this plan, analysis of surface and subsurface soil, groundwater, surface water, sediment, and pore water samples will be

performed to achieve Level 3 data reporting requirements. Thus, to achieve this level of control, the following field QC samples will be collected.

Field Blanks. A Field Blank (FB) is used to demonstrate the absence of contamination during sampling activities. It will be comprised of contaminant free water brought to the field by the sampling crew, and transferred to the proper sample container for shipment along with the other samples collected. One field blank sample will be collected per sampling day.

Rinsate Blanks. A Rinsate Blank (RB), commonly called an equipment blank, is used to demonstrate the effectiveness of field cleaning procedures for sampling equipment. A volume of rinse solution, consisting of contaminant free water, is poured over the equipment that has been cleaned in the field, and collected for analysis. One rinsate blank sample will be collected per each type of media-specific sampling equipment used during a sampling event when such sampling equipment is cleaned in the field.

Trip Blanks. A Trip Blank (TB) is utilized to detect possible VOC contamination of samples to be analyzed for VOCs. VOCs are susceptible to contamination by introduction or migration of contaminants through the volatile vial septum. Trip blanks will be prepared by filling volatile vials with purged organic free water. The trip blanks are prepared in the laboratory and accompany the sample collection vials to the sampling site, back to the laboratory, and are stored with the collected samples prior to analysis. Trip blanks will be shipped with each cooler containing field samples collected for VOC analysis.

Field Duplicate Samples. Field duplicate samples will be collected at a frequency of ten percent for each media sampled.

7.0 DECONTAMINATION PROCEDURES

7.1 SAMPLING EQUIPMENT

Soil sampling equipment will be decontaminated prior to the start of soil sampling and between each hole. Equipment will be decontaminated in accordance with the procedures described in Section B2 (pages B-15 to B-17) of the QAPP. Equipment utilized for the collection of samples to be analyzed for VOCs or SVOCs will be decontaminated as follows.

- Clean with tap water and soap using a brush if necessary to remove particulate matter and surface films. Equipment may be steam cleaned (soap and high pressure hot water) as an alternative to brushing. Sampling equipment that is steam cleaned will be placed on racks or saw horses at least two feet above the floor of the decontamination pad. Polyvinyl chloride (PVC) or plastic items will not be steam cleaned.
- Rinse thoroughly with tap water.
- Rinse thoroughly with analyte free water.
- Rinse thoroughly with solvent (e.g., pesticide-grade isopropanol). PVC or plastic items will not be rinsed with solvent.
- Rinse thoroughly with organic/analyte free water. If organic/analyte free water is not available, equipment will be allowed to completely dry. A final rinse **will not** be applied with analyte water.
- Remove the equipment from the decontamination area and cover with plastic. Equipment stored overnight will be wrapped in aluminum foil and covered with clean, unused plastic.

7.2 DECONTAMINATION PAD

A decontamination pad will be constructed for field cleaning of sampling equipment, including downhole drilling equipment. The decontamination pad will be constructed such that it that will meet the following requirements:

- The pad will be constructed in an area known or believed to be free of surface contamination.
- If feasible, the pad will be constructed on a level surface that will facilitate the collection of wastewater. This will be accomplished by

either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit will also be lined.

- The temporary pad will be lined with a water impermeable material with no seams within the pad. The material will be either easily replaced (disposable) or repairable. The pad will not leak excessively.

- Sawhorses or racks will be constructed to hold equipment while being cleaned and will be high enough off the ground to prevent equipment from being splashed.

- At the completion of the Site activities, the decontamination pad will be dismantled. Wastewater remaining in the pad will be removed and containerized for disposal.

- If solvents are not used during decontamination activities (i.e., pesticide-grade isopropanol which is to be used for decontamination of sampling equipment for VOCs and SVOCs), the decontamination water will not be collected

- When isopropanol is utilized for decontamination, the decontamination fluids will be collected and containerized for proper disposal.

The location of the decontamination pad will be determined prior to mobilization to the site for the sampling activities.

8.0 POST SAMPLING ACTIVITIES

8.1 SURVEYING

Following completion of the sampling activities, subsurface soil, surface water, and sediment sample locations will be surveyed by Global Positioning System (GPS) equipment and monitoring well locations will be surveyed for horizontal and vertical control by a South Carolina registered land surveyor.

8.2 INVESTIGATION DERIVED WASTE

During the field activities, investigative derived wastes (IDW) consisting of soil cuttings generated during the drilling of soil borings and installation of groundwater monitoring wells and development/purge water generated during installation and sampling of the monitoring wells. The IDW will be containerized in 55-gallon drums and stored within the locked security fence of the Site. At the conclusion of the soil boring, soil sampling, monitoring well installation, and groundwater sampling activities, composite soil or water samples will be collected from each drum (or set of drums generated from each location), as applicable, and sent to the laboratory for waste characterization purposes to determine proper disposal of the IDW. The drums will be sealed with the drum cover and cover ring. The nut on the cover ring will be tightened to the extent possible using a ratchet and socket or other similar hand tool. The drums will be labeled with an identification number, date, contents, and associated sampling locations pending the results of the laboratory analyses, at which time the drums will be scheduled for removal and proper disposal. Transportation manifests and certificates of disposal will be obtained. The IDW drum inventory will be maintained by the Field Operations Leader.

9.0 REFERENCES

United States Environmental Protection Agency, 1988, "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final," Office of Emergency and Remedial Response, OSWER Directive 9355.3-01 EPA/540/G-89/004.

United States Environmental Protection Agency, Region IV Science and Ecosystem Support Division, various dates, "Field Branches Quality System and Technical Procedures."

TABLES

TABLE 1

Summary of Monitoring Well Construction Specifications
Field Sampling and Analysis Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Well ID	Area	Well Depth	Screened Interval	Top of Sand	Top of Bentonite
MW-2D	Former Oil/Water Separator	55*	50-55	48	46
MW-3	Former Oil/Water Separator	25	15-25	13	11
MW-4	Former Oil/Water Separator	25	15-25	13	11
MW-5	Former Oil/Water Separator	25	15-25	13	11
MW-6	Chlorinated Solvent Plume	25	15-25	13	11
MW-7	Chlorinated Solvent Plume	25	15-25	13	11
MW-8D	Chlorinated Solvent Plume	55*	50-55	48	46
MW-9	Chlorinated Solvent Plume	25	15-25	13	11
MW-10	Chlorinated Solvent Plume	20	10-20	8	6
MW-11	Chlorinated Solvent Plume	20	10-20	8	6
MW-12D	Chlorinated Solvent Plume	50*	45-50	43	41
MW-13	Chlorinated Solvent Plume	20	10-20	8	6
MW-14	Chlorinated Solvent Plume	20	10-20	8	6
MW-15	Chlorinated Solvent Plume	20	10-20	8	6
MW-16D	Chlorinated Solvent Plume	55*	45-50	43	41
MW-17	Chlorinated Solvent Plume	20	10-20	8	6
MW-18D	Chlorinated Solvent Plume	55*	45-50	43	41
MW-19D	Chlorinated Solvent Plume	55*	45-50	43	41
MW-20	Former Metals Baghouse	25	15-25	13	11
MW-21	Former Metals Baghouse	25	15-25	13	11
MW-22	Former Scrap Metal Rolloff	25	15-25	13	11
MW-23	Former Scrap Metal Rolloff	25	15-25	13	11
MW-24	Heat Treat Cleaning Water	25	15-25	13	11

Notes:
Assumes eight inch nominal diameter borehole for surface completions.
"D" signifies deep monitoring wells (top of rock).
Assumes deep wells will be Type III wells with a four-inch surface casing installed approximately 10 feet above the top of bedrock surface.
* = top of bedrock assumed from Geoprobe refusal.
Depths reported in feet below ground surface.
Surface completions will be flush-mount or stickup as appropriate.

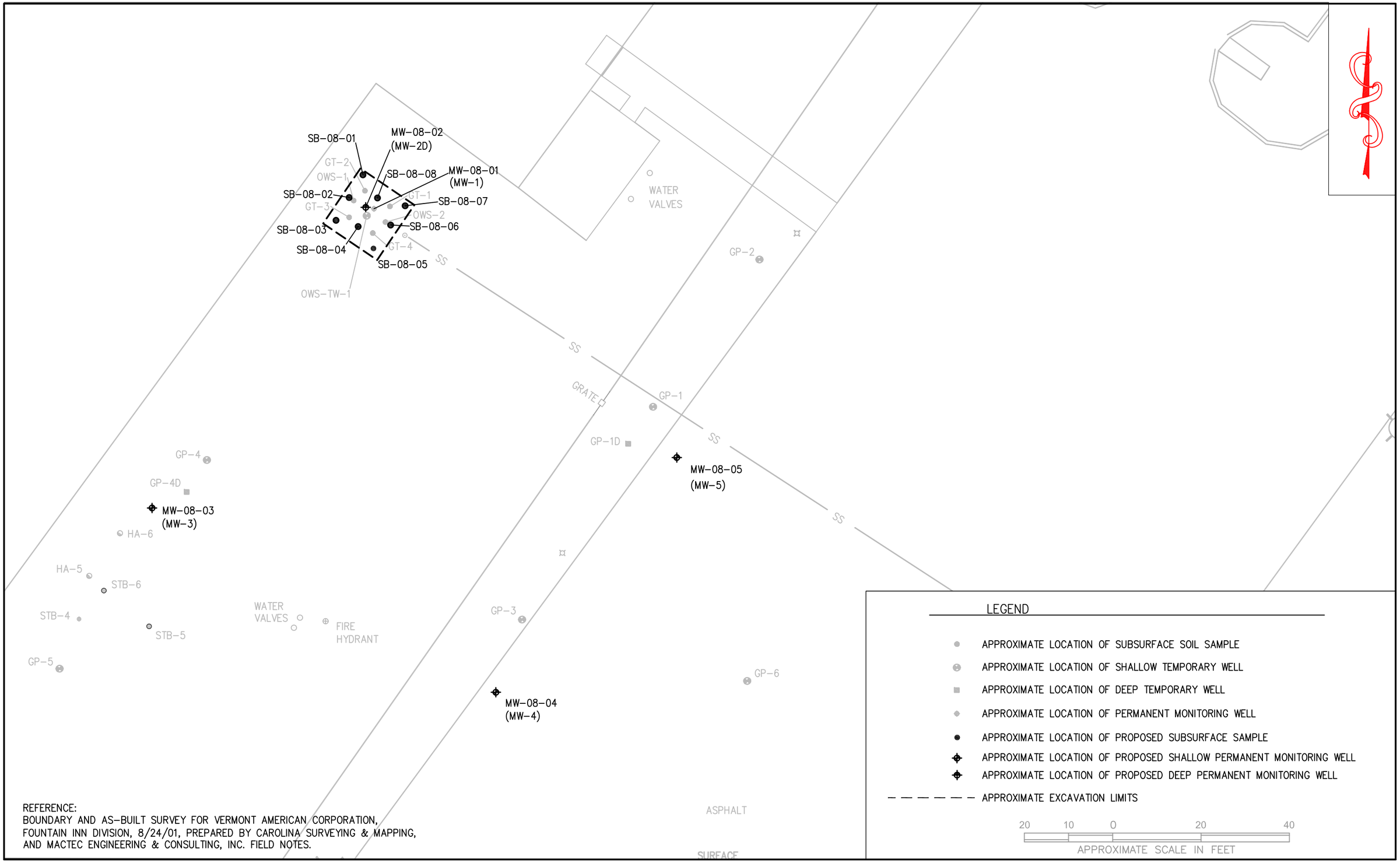
TABLE 2

Summary of Sample Matrix, Collection Method, and Analytical Procedures
Field Sampling and Analysis Plan
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

AOC Number	Area of Concern	Sample Matrix						Analytical Procedure
		Surface Soil	Subsurface Soil	Ground Water	Surface Water	Sediment	Pore Water	
3	Former Metals Baghouse			2				TAL Metals, TCL VOCs (1), TCL SVOCs (1)
4	Former Scrap Metal Rolloff		3	2				TCL VOCs, TCL SVOCs, TAL Metals
6	Compounding Room Blower Exhaust		4					TCL VOCs, TCL SVOCs
7	Storm Water Outfalls	3						PAHs
8	Former Oil/Water Separator Area		16	5				TCL VOCs, TCL SVOCs, TPH-DRO
9	Former Hazardous Waste Accumulation Building		27	14	9	9	8	TCL VOCs, TCL SVOCs

Notes:
AOC = Area of Concern
TAL = Target Analyte List
TCL = Target Compound List
VOCs = volatile organic compounds to be analyzed by SW-846 Method 5035A/8260B.
SVOCs = semi-volatile organic compounds to be analyed by SW-846 Method 8270C.
PAHs = polynuclear aromatic hydrocarbons to be analyzed by SW-846 Method 8270C.
TAL Metals = metals to be analyzed by SW-846 Method 6010B/7470A/7471A.
TPH-DRO = Total Petroleum Hydrocarbons-Diesel Range Organics to be analyzed by SW-846 Method 8015B.

FIGURES



REFERENCE:
BOUNDARY AND AS-BUILT SURVEY FOR VERMONT AMERICAN CORPORATION, FOUNTAIN INN DIVISION, 8/24/01, PREPARED BY CAROLINA SURVEYING & MAPPING, AND MACTEC ENGINEERING & CONSULTING, INC. FIELD NOTES.

LEGEND

- APPROXIMATE LOCATION OF SUBSURFACE SOIL SAMPLE
- APPROXIMATE LOCATION OF SHALLOW TEMPORARY WELL
- APPROXIMATE LOCATION OF DEEP TEMPORARY WELL
- APPROXIMATE LOCATION OF PERMANENT MONITORING WELL
- APPROXIMATE LOCATION OF PROPOSED SUBSURFACE SAMPLE
- APPROXIMATE LOCATION OF PROPOSED SHALLOW PERMANENT MONITORING WELL
- APPROXIMATE LOCATION OF PROPOSED DEEP PERMANENT MONITORING WELL
- APPROXIMATE EXCAVATION LIMITS

20 10 0 20 40
APPROXIMATE SCALE IN FEET

DRAWN	CHB	DATE	5/14/12
CHECKED	GWW	FILE	FIGURE 1.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

REVISIONS

No.	DESCRIPTION	BY

amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

OIL/WATER SEPARATOR AREA
FORMER VERMONT AMERICAN FACILITY
FOUNTAIN INN, SOUTH CAROLINA

FIGURE
1

RBTC FOUNTAIN INN DIVISION

FORMER SHERWIN WILLIAMS PROPERTIES

MW-20
MW-21
MW-24
MW-2D
MW-5
MW-3
MW-4
MW-9
MW-8D
MW-22
MW-23
MW-7
MW-19D
MW-10
MW-06
MW-11
MW-12D
MW-14
MW-13
MW-16D
MW-15
MW-18D
MW-17

LEGEND

MW-14 — PROPOSED MONITORING WELL LOCATION

100 50 0 100 200

SCALE 1" = 100'

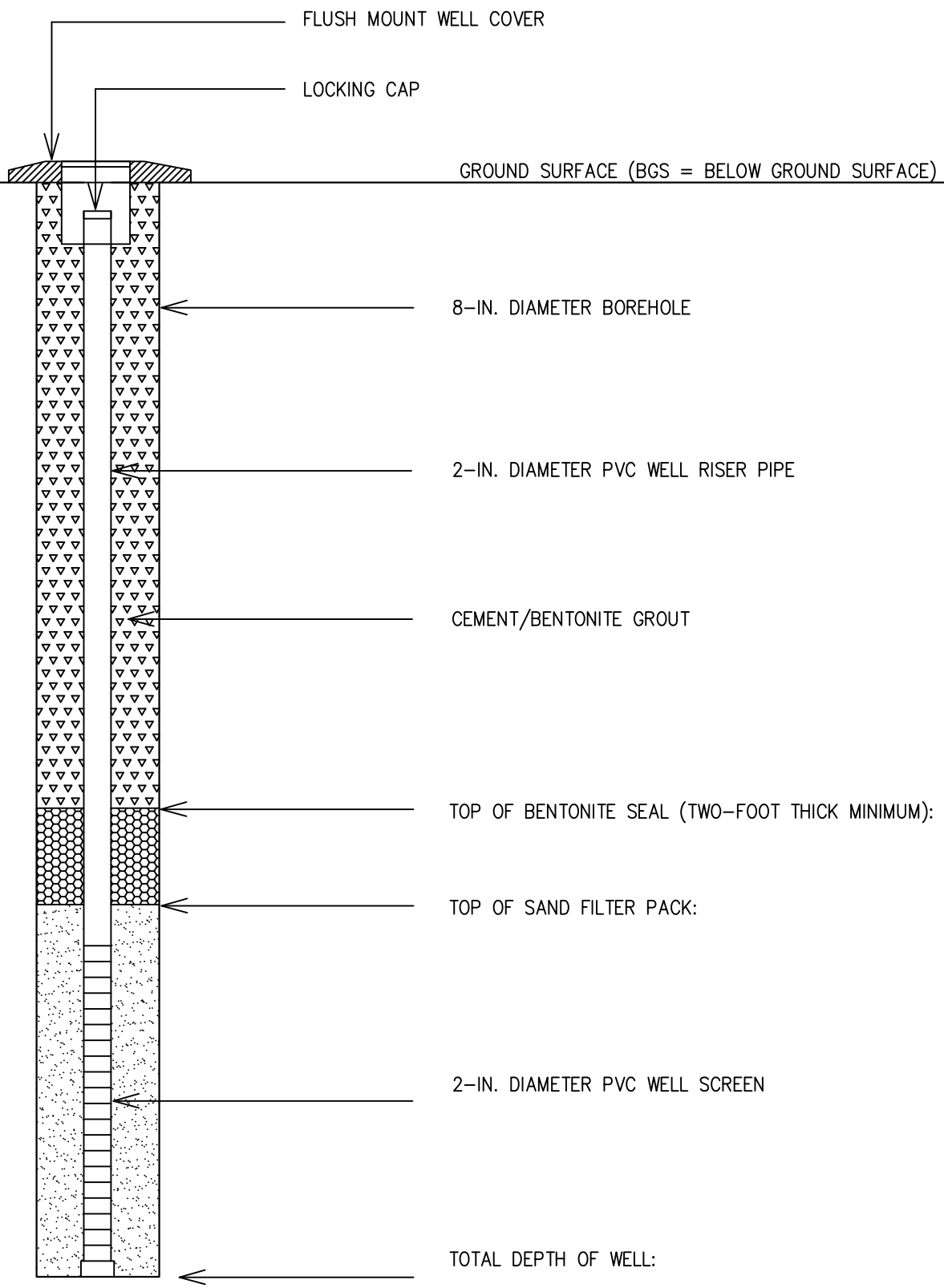
DRAWN	CHB	DATE	5/8/12
CHECKED	GWW	FILE	FIGURE 2.DWG
APPROVED	PSJ	JOB NO:	6251121007.01.01

REVISIONS		
No.	DESCRIPTION	BY

amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

PROPOSED MONITORING WELL LOCATION MAP
RBTC FOUNTAIN INN DIVISION/FORMER SHERWIN WILLIAMS PROPERTIES
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 2



CONTRACTOR:		CERTIFICATION NO. & STATE:	
DRILLER:		DRILLING EQUIPMENT:	
MACTEC FIELD PERSONNEL:		MW PERMIT NO. & STATE:	

NOT TO SCALE

INSTALLATION DATE:
GROUND SURFACE ELEVATION:
MEASURING POINT ELEVATION:
LATITUDE:
LONGITUDE:

amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

FIGURE 3
MONITORING WELL CONSTRUCTION DIAGRAM
RBTC FOUNTAIN INN
FOUNTAIN INN, SOUTH CAROLINA

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	PROJECT NO.:
FIGURE 3.DWG	CHB	GWW	PSJ	5/8/12	6251121007.01.01

APPENDIX D

HEALTH AND SAFETY PLAN

HEALTH AND SAFETY PLAN

FORMER VERMONT BOSCH SITE
FOUNTAIN INN, SOUTH CAROLINA

Prepared for:

ROBERT BOSCH TOOL CORPORATION

1800 West Central Road
Mount Prospect, Illinois 60056

Prepared by:

AMEC Environment & Infrastructure, Inc.
555 North Pleasantburg Drive, Suite 202
Greenville, South Carolina 29607

AMEC Project 6251121007.01.01

May 31, 2012

TABLE OF CONTENTS

Page

TABLE OF CONTENTS - Continued

TABLES

FIGURES

APPENDICES

SECTION I

EMERGENCY CONTACTS

EMERGENCY TELEPHONE NUMBERS

Medical	Emergency Assistance/Ambulance	911
	Hospital – Hillcrest Hospital 729 SE Main Street Simpsonville, South Carolina 29681	(864) 967-6100
Police	Emergency Assistance	911
	Fountain Inn Police Department	(864) 862-4461
Fire	Emergency Assistance	911
	Fountain Inn Fire Department	(864) 862-0010

Other:
National Poison Control Center (800) 492-2414
Chemical Manufacturing Association – Chemical Referral Center (800) 262-8200

AMEC Environment & Infrastructure, Inc.

		Telephone	Cell Phone
Office	Greenville, SC	(864) 552-9624	Not applicable
Site Health & Safety Officer (SHSO)	Gary W. Wise	(864) 552.9624	(864) 901-2965
Project Manager	Paul S. Johnstone	(864) 552-9624	(864) 616-4176
Project Principal	Paul S. Johnstone	(864) 552-9624	(864) 616-4176
Site Manager	Chris Bruce	(864) 552-9624	(864) 430-7415

Client

Robert Bosch Tool Corporation
Contact: David Luepke
Director, Environmental, Safety & Facility Services
(224) 232-2201

SECTION II

SITE SPECIFIC SAFETY AND HEALTH INFORMATION

A. Site Description

AMEC Project Number: 6251121007

Client: Robert Bosch Tool Corporation

Site Location: 800 Woodside Avenue
Fountain Inn, South Carolina

B. Scope of Work:

This Health and Safety Plan (HASP) is intended to address the details associated with the phases of site assessment activities described in the Remedial Investigation and Feasibility Study (RI/FS) Work Plan. However, in the event that the scope of services goes beyond what is covered under this present HASP, the document will be revised as appropriate prior to mobilization for subject work.

The scope of work to be performed in the investigation can be summarized as follows.

Scope of Work:

- Conduct a Site visit (to locate sample locations and identify background locations).

- Field Sampling:

  - Collect subsurface soil samples by means of direct-push drill rig.
  - Collect surface water, sediment, and pore water samples.
  - Install permanent groundwater monitoring wells.
  - Collect groundwater samples.
  - Perform hydraulic conductivity (slug) testing.

C. Key Personnel

AMEC Personnel:

Project Manager:	Paul S. Johnstone, P.G.
Project Principal:	Paul S. Johnstone, P.G.
SHSO:	Gary W. Wise, P.E.
Responsibilities	See Section 2.0
Training	See Section 3.0

Additional Personnel On Site:

Client representatives:	David Luepke Robert Bosch Tool Corporation (224) 232-2201
Contractors:	Probe Technology, Inc. Arlen Burney (704) 933-5538
	A.E. Drilling Services, Inc. William Barnes (864) 288-1986
	Freeland & Associates, Inc. Michael Austin (864) 217-4924
Regulatory Agency:	South Carolina Department of Health and Environmental Control (SCDHEC)
SCDHEC Project Manager:	Regina Brown Columbia, South Carolina (803) 896-4129

D. Hazard Evaluation

Chemical Hazards

The hazardous substances of concern at the Site are the volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) discovered during previous investigation of the site. These constituents were mobilized by past operational practices.

Samples collected during previous environmental assessments revealed elevated levels of acetone, 2-butanone, 1,1-dichloroethane, ethylbenzene, tetrachloroethene (perchloroethylene, or PCE), toluene, xylenes, diethylphthalate, bis(2-ethylhexyl)phthalate, and total petroleum hydrocarbons, oil and grease (TPH O&G) in soil and elevated levels of acetone, 2-butanone, chloroethane, 1,1-dichloroethane, 1,1-dichloroethene (1,1-DCE), ethylbenzene, naphthalene, styrene, PCE, toluene, 1,1,1-trichloroethane, trichloroethene TCE, xylenes, di-n-butyl phthalate, diethylphthalate, and TPH O&G in ground water. PCE, TCE, and 1,1-DCE were above South Carolina's promulgated Maximum Contaminant Levels (MCLs).

Surface water samples collected from an unnamed tributary to Stoddard Creek had elevated levels of PCE above the South Carolina Water Quality Criteria (WQC).

The anticipated concentrations of these compounds are summarized on Tables 1 and 2.

Physical Hazards:

- Heavy equipment – drill rig may be present.

- Slippery mud.

- Heat Stress - Field activities in hot climates create a potential for heat stress. The warning symptoms include fatigue; loss of strength; reduced accuracy, comprehension, and retention; and reduced alertness and mental capacity. To prevent heat stress, personnel shall receive adequate water supplies and electrolyte replacement fluids, and maintain scheduled work/rest periods. Pulse rate and body temperature shall also be monitored as appropriate.

- Cold Stress -

- Biological hazards – Bees, ticks, snakes.

E. Personal Protective Equipment

Clothing and Respirators	See Section 4.0
Decontamination Procedures	See Section 5.0
Medical Surveillance	See Section 6.0

Based on the evaluation of potential hazards at the site, the field work will be performed in Level D personal protective equipment (PPE). Level D PPE will consist of the following.

Level-D PPE:	Protective gloves (PVC, Nitrile or Butyl Rubber)
	Hard Hat (when working near heavy equipment like drill rigs or when other overhead hazards are present).
	Safety glasses or goggles
	Steel-toe safety shoes
	Long pants.
	Hearing protection if extended exposure to rig noise

F. Field Monitoring Procedures

Monitoring	See Section 7.0
Contingency Plan	See Section 13.0

VOCs have been identified at the site and are suspected to be present. Therefore, monitoring of the breathing zone is to include monitoring with an organic vapor analyzer, either a flame-ionization detector (FID) or photo-ionization detector (PID), and if appropriate, work will be performed in a higher level of protection (Level C).

SECTION III

GENERAL SAFETY, HEALTH, AND EMERGENCY INFORMATION

1.0 INTRODUCTION

The health and safety of site workers and the public is a primary concern and goal during project field activities. Thus, a comprehensive, carefully managed, and thoroughly documented site Health and Safety Plan (HASP) is prepared and implemented as part of project operations.

The following plan describes field implementation of the HASP, specific responsibilities, training requirements, protective equipment, and operating and emergency procedures. Specifically included are procedures for site and personnel monitoring during project field activities. Documentation of these and other site conditions during execution of the project is required. The plan's flexibility allows unanticipated site-specific problems to be addressed while assuring adequate and suitable worker protection. This plan is based on site specific information, actual site inspections, and determination of the potential hazardous conditions which may be encountered.

2.0 RESPONSIBILITIES

The Project Manager and Site Health and Safety Officer (SHSO) are responsible for formulating and enforcing health and safety requirements. These responsibilities include:

- assuring that all site team members have received the required health, safety and emergency response training;
- assuring that all site team members have completed the required medical examination and have met the appropriate qualification criteria for site work;
- assuring that all equipment used on site is suitable and adequate; and
- assuring that site standard operating procedures are followed at all items.

The SHSO will have direct responsibility for administering the HASP relative to all site activities. The Project Manager has general overview responsibility for the success of the project and will make occasional site visits during field operations. The Project Manager has prepared the HASP based on a format developed by AMEC and information obtained on site conditions. The SHSO has responsibility for reviewing and approving the HASP and responding to any non-routine matters that relate to health, safety, and emergency response during the project life. The Project Manager may visit the site at any time to monitor compliance with the provision of the HASP. Field personnel and subcontractors engaged in conducting site investigation activities are responsible for complying with the HASP.

3.0 PERSONNEL TRAINING

All personnel working at the site who potentially may be exposed to toxic substances or hazardous materials will participate in an initial and annual refresher and/or supervisory training (as appropriate), as well as site-specific training before commencement of any on-site assignment. The initial Health and Safety Training Program consists of the 40-hour training program required and designated by Occupational Safety and Health Administration (OSHA) standard 29 CFR 1910.120. In addition, AMEC uses eight-hour annual refresher and supervisory training elements, which are augmented by site-specific training and briefings. Site-specific training covers hazards and specialized protocols specific to the site. In addition it will cover major elements of the site HASP, as well as health and safety procedures regarding an individual's specific job responsibilities and tasks. Training, such as that defined under AMEC's Health and Safety Program and 29 CFR 1910.120 is required for all personnel entering work areas at the Site. Personnel without the required training will not be permitted in any work area at the Site. A thorough understanding of the types of hazards most likely to be encountered at contaminated work site and personal protection measures needed to protect personnel from hazards are the first requirements of a complete safety, health and emergency response plan. Each project team member shall have received instruction in health and safety procedures appropriate for conducting and participating in work at contaminated sites. Additional briefings and safety meetings will be held with field personnel before beginning project related work to explain and discuss site-specific health and safety matters.

A preliminary safety meeting between AMEC and subcontractor's personnel will be conducted before the initiation of any field activities. The initial briefing will discuss potential hazards which may be encountered, site safety, and the emergency response plan.

Subcontractors will be subject to all applicable health and safety regulations during field operation at the site. The SHSO or his representative is responsible for briefing the subcontractor's personnel on contamination that may be encountered on the site, site safety, and the emergency response plan. Each of the subcontractors will be under the direct supervision of the SHSO or his representative.

4.0 PERSONAL PROTECTIVE EQUIPMENT

Based on experience at similar sites and the anticipated work, it appears that the primary exposure to contaminants during field activities may occur through skin contact or through inhalation of vapor or dust while sampling and handling potentially contaminated soil or water.

The required personal protective and emergency equipment have been selected based on anticipated site conditions and work.

4.1 PERSONAL PROTECTIVE EQUIPMENT

The level of protection required is selected based on the anticipated hazards and site activities consistent with United States Environmental Protection Agency (USEPA) protocols and the provisions of OSHA 29 CFR 1910 and 1926. Anything less than maximum protection (Levels A or B) cannot be specified without (1) carefully defining site conditions, (2) allowing extra safety margins, (3) having higher level equipment readily

available, and (4) anticipated worst-case conditions. Because worker efficiency decreases in direct proportion to the amount of protective gear required, it is always desirable to use as little equipment as possible while providing adequate protection.

In the event of conflicting requirements, the most protective level shall apply. The required levels of protection are subject to change at any time by the SHSO or the Project Manager based on monitoring, visual observations, or changes in work or site conditions.

4.2 CLOTHING

4.2.1 Types of Protective Clothing Materials

The following materials are generally available for a variety of garments:

a. Non-elastomers: Tyvek, Nomex.

b. Elastomers: Polyethylene, Saranex, Polyvinyl Chloride (PVC), Neoprene, Chlorinated Polyethylene (CPE) or Chloropel, Butyl Rubber, Viton, Natural Rubber, Nitrile, Polyvinyl Alcohol (PVA).

4.2.2 Essential Considerations when selecting clothing

a. Work Mission Duration

1. Work rate
2. Physical fitness
3. Body size

b. Ensemble Permeation/Penetration by Contaminants

1. Valves
2. Fasteners

c. Ambient Temperature

1. Valves
2. Fasteners
3. Chemical behavior

4.2.3 Clothing Reuse

a. Chemicals can permeate after decontamination

b. Effects of contaminants on protective clothing must be known

4.2.4 Inspection

a. Inspection/testing of clothing received from factory

b. Inspection as it is issued to workers

c. Inspection after use

d. Periodic inspection during storage

e. Continuous inspection prior to, during, and after use

4.2.5 Storage

a. Separate from street clothes

b. Segregated by types and material

c. Store according to manufacturer (folded, hung, etc.)

4.2.6 Maintenance

a. Performed by qualified personnel

b. Authorized by manufacturer through sale of replacement parts

4.2.7 Clothing Selection

a. Principle factors

b. Physical integrity for intended tasks

c. Ease and cost of decontamination

d. Performance requirements of clothing material

4.2.8 Performance Characteristics of Clothing Material

a. Chemical Resistance

1. Degradation: breakdown of the material
2. Penetration: leakage through clothing seams, faults
3. Permeation: breakthrough depending on:

a). Material type
b). Degree of concentration gradient
c). Ambient temperature/contaminant temperature
d). Humidity
e). Barometric pressure

b. Flexibility (often depends on ambient temperatures)

c. Thermal limits

d. Durability (ability to resist physical damage)

4.3 RESPIRATORS

Respirators are used to help protect against inhalation hazards. The selection of a respirator usually involves three steps:

a. Identifying the hazards,

b. Evaluating the hazards,

c. Providing proper respiratory protective equipment to suit the conditions and the individual.

There are two general types of respirators: air purifying and supplied air respirators.

4.3.1 Air Purifying Respirators

- Removes hazardous contaminant from the air before it is inhaled.
- Consists of rubber facepiece and replaceable filters or cartridges.
- Can be full faced (covering all face from chin to forehead) or half faced (covering nose and mouth)
- Filters or cartridges must be chosen for specific contaminant. Different types are color coded for use with specific contaminant or group of contaminants.
- Negative pressure devices to insure air enters only through the filters.

4.3.1.1 Powered Air Purifying Respirators (PAPR)

- Positive pressure devices.
- Air is blown by the use of a battery pack through the filter and onto the face.
- Bulkier to wear than the half or full face type air purifying respirator but can be more comfortable in hot working conditions.

4.3.2 Supplied Air Respirators

- Air supplied to the respirator from a source independent of the immediate hazardous atmosphere.
- Air supplied through an airline or from compressed air from a tank worn on the back. This second type is known as Self Contained Breathing Apparatus (SCBA).

4.3.3 Wearing Respirators

- All respirators should be used in accordance with the OSHA respirator standard 29 CFR 1910.134.
- Individuals must be fit tested before being assigned a respirator.
- No smoking while using a respirator

- No eating or chewing
- Be clean shaven and free of face hair that may interfere with the respirator seal.

4.3.4 Inspections

All respirators should be routinely checked for:

- Dirt.
- Cracks, scratches and tears.
- Distortion.
- Broken or missing parts.

4.3.5 Cleaning and Disinfecting

Whenever possible, respirators should be assigned to one individual. Respirators should be cleaned after each use, rinsed with disinfectant, rinsed with clean water and left to air dry.

4.3.6 Storage

Respirators should be stored in clean plastic bags and left in a clean, dry area.

5.0 DECONTAMINATION PROCEDURES

Each individual shall conduct proper personal hygiene which may include washing any exposed skin prior to eating or smoking, consistent with site conditions.

A minimum decontamination for the Level D site work will consist of cleaning boots of loose soil or debris and discarding gloves before leaving the site to prevent spreading the contamination that may exist on the site. Decontamination procedures for higher levels of protection (i.e. Level C) are not presented. The HASP will be revised to include a description of these activities in the event that such action is warranted.

6.0 MEDICAL SURVEILLANCE

All personnel performing activities at the site must be medically qualified for site assignment as determined by an acceptable medical surveillance program. Personnel without medical clearance will not be permitted in work areas.

Symptoms of exposure to hazardous materials and physical stresses (e.g. heat stress or cold stress) will be reviewed with site personnel to indicate the recognized signs of possible exposure or physical stresses. The most likely physical stresses to occur at the site, heat stress and cold stress, are discussed in the following paragraphs.

Heat Stress: Field activities in hot climates create a potential for heat stress. The warning symptoms include fatigue; loss of strength; reduced accuracy, comprehension, and retention; and reduced alertness and mental capacity. To prevent heat stress, personnel shall receive adequate water supplies and electrolyte replacement fluids, and maintain scheduled work/rest periods. Pulse rate and body temperature shall also be monitored as appropriate.

Cold Stress: Field activities in cold weather create a potential for cold stress, including wind chill, frostbite, and hypothermia. The warning symptoms include heavy shivering, frostnip, excessive fatigue, irritability, or euphoria. To prevent cold stress, a work-warm regimen shall be instituted when work is being conducted in environments where the wind chill temperature is below 20º Fahrenheit (F). Heated warming shelters shall be available or provided on or nearby the work site, as appropriate. Heated shelters can include heated buildings, tents, cars, drill rigs, etc. Employees shall be given breaks at regular intervals to use the shelter. Breaks shall be at least 10 minutes in duration, and the interval to be used between breaks shall be noted in the project logbook.

7.0 EMERGENCY EQUIPMENT

The following emergency equipment will be available on the site during field operations:

7.1 FIRE EXTINGUISHERS

Because of the potential threat of fire at hazardous waste sites, fire extinguishers will be readily available and at hand throughout the investigation. All fire extinguishers will be Class ABC. The fire extinguishers will be kept with the field crew during any field activities.

7.2 FIRST AID KITS

An industrial first aid kit will be kept in the support area.

7.3 EYE WASH

An eyewash station (meeting the minimum requirements of ANSI Z358.1) and sufficient potable water for copious flushing will be readily available throughout the project operations.

7.4 COMMUNICATIONS

Emergency telephone numbers are included in the HASP (see Section I), which will be readily available on-site. Emergency communication will be discussed in the safety briefings with all work crews prior to initiating the field work. Mobile/cellular telephones will be used in the work area.

7.5 PERSONAL HYGIENE

A sufficient supply of clean, potable water and hand soap will be provided near the work area for the personal hygiene of field personnel. Personal hygiene primarily entails casual washing during site activities and is not strictly considered decontamination.

8.0 SITE CONTROL

All public will be kept out of the work zone. If necessary, physical barriers will be erected. Other means of segregating the public can include tape, cones and barriers. Warning signs can be erected.

A contaminated waste site may be divided into three specific zones established on the basis of contamination potential:

Zone 1 - exclusion zone;
Zone 2 - contamination reduction zone; and
Zone 3 - support zone.

Given the low-degree of hazards anticipated at the site, work zones will only be established around the drill rig for protection of workers and others from potential mechanical hazards.

The exclusion zone is the suspected area of greatest environmental contamination and presents the greatest potential for worker exposure. Personnel entering the area must wear the mandated level of protection. In certain instances, different levels of protection will be required depending on the tasks to be performed within that zone. The support zone serves as a clean, control area, where decontamination facilities are located. The contamination reduction zone serves as a transition area between the exclusion zone and the support zone. All areas will be defined and marked as appropriate by the Site Manager.

At the site, the establishment of these three specific zones may not be applicable. If hazardous conditions develop during site operations, work will be stopped and the three zones and any necessary additional zones will be developed. The zones will be marked by appropriate flags and stakes, and personnel will be briefed about activities and protective equipment for each zone.

9.0 ACCIDENT PREVENTION

9.1 BEFORE LEAVING FOR SITE

Review Site Information (see SHSO).

a. Expect hazards
b. Special conditions
c. Sampling procedures
d. Location of telephones and emergency equipment
e. Emergency medical information
f. Level of personnel protection required

Check safety gear and equipment. The following equipment will be used at the site, or will be available for issue, depending on site-specific conditions. The safety gear and equipment will be available on-site in a support vehicle.

a. Steel-toe safety boots
b. Neoprene or Nitrile rubber boots
c. Coveralls, Tyvek and Saranex coated Tyvek
d. Hard-hat
e. Goggles or Safety Glasses
f. Neoprene gloves
g. Half-face respirator with cartridges suitable for organic vapor, dusts,
h. Ziploc® baggies (quart and gallon size), aluminum foil, and plastic sheeting to keep equipment clean
i. Field standard operating procedures (Field Sampling and Analysis Plan, Quality Assurance Project Plan)

Back-up equipment and spares will be maintained, including:

a. Gloves
b. Duct tape
c. Trash barrel for return transportation of contaminated gear and
d. Extra respirator cartridges

9.2 BEFORE ENTERING SITE

a. No eating/drinking/smoking except away from the work area. Use good sanitary practices and wash hands and face thoroughly before eating/drinking/smoking.
b. Drink some salt replacement fluids, especially during hot weather conditions, and carry drinks for use in support area.
c. Place sample containers in field sample carrier (backpacks or carrier).
d. Do not place containers or equipment on potentially contaminated surfaces.
e. Check location of emergency eye wash supply and telephones.
f. Check alternate safety gear.

    - Respirator (test even if you are not going to wear it immediately)
    - Hard-hat
    - Goggles or safety glasses
    - Check gear for rips/tears/malfunctions.

g. Set up buddy system prior to proceeding with work.
h. Make preliminary site survey.

- Characterize physical conditions of site.
- Use as much excess caution as possible.
- Use caution - go slowly.

9.3 ON SITE

The following items are requirements to protect the health and safety of field workers and will be discussed in the safety briefing prior to initiation of work on the site.

- A **BUDDY SYSTEM** will be used. Hand signals will be established to maintain communication.

- During site operations, each worker will consider himself as a safety backup to his partner. Off-site personnel will provide emergency assistance. All personnel will be aware of dangerous situations that may develop.

- Visual contact will be maintained between buddies on-site when performing hazardous duties.

- Eating, drinking, chewing gum or tobacco, smoking, or any practice that increases the probability of hand-to-mouth transfer and ingestion of hazardous material is prohibited at the site.

- Prescription drugs will not be taken by personnel where the potential for contact with toxic substances exist, unless specifically approved by a qualified physician. Alcoholic beverage intake is prohibited during the work day.

- No facial hair which interferes with the face-to-face piece seal of the respirator will be permitted on personnel required to wear such equipment. Each staff member will be fit-tested for respirators by the Health and Safety Officer using an approved technique prior to arriving at the site. If Level D is initially specified at the site, the use of respirators is not required. However, organic vapor respirators will be available on-site in the event that upgrading to Level C is necessary. The respirators and spare cartridges will be available on-site in a support vehicle.

- Work areas for various operational activities (equipment testing, decontamination) will be established if higher levels of protection are implemented at the site.

- Procedures for leaving any contaminated area will be planned and reviewed prior to going on-site.

- Work areas and decontamination procedures have been established based on prevailing site conditions and are subject to change if site conditions change.

- No personnel will be admitted to the site without the proper safety equipment and training.

- Proper decontamination procedures must be followed before leaving the site. Decontamination in the Level D mode of operation will consist of good personal hygiene and cleaning boots and gloves before leaving the site.

- All personnel must comply with established safety procedures. Any staff member who does not comply with safety policy, as established by the Health and Safety Officer or the Project Manager, will be immediately dismissed from the site.

- Any medical emergency supersedes routine safety requirements.

- The Field Safety Coordinator will make regular safety inspection of the site to insure that operations are being conducted in accordance with established Safety Procedures.

9.4 PROCEEDING WITH WORK

- No eating/drinking/smoking while working in contaminated area.

- Use standard, specified work techniques (see work procedures or discuss with Site Manager).

- Use appropriate care in handing contaminated material. If the work site is not accessible using your gear, do not enter the work site. Confer with buddy and team leader about alternative work locations.

- Wipe off spills, dirt and residue immediately.

- If any gear or equipment damage develops, immediately repair or replace.

- If you experience any physical discomfort, abnormalities, or lightheadedness -stop work, tell your buddy, and go back to designated Support Zone.

10.0 CONTINGENCY PLAN

10.1 EMERGENCY ASSISTANCE

All emergency contacts are listed in Section I of this HASP.

10.2 HOSPITAL

Hospital emergency room personnel should be contacted and briefed regarding the scope of the work. The emergency route to the hospital shall be discussed with the field personnel prior to beginning any activities. Figure 1 presents a map to the nearest hospital as well as written directions.

10.3 ACCIDENTS/INJURIES

Depending on the severity of the injury, treatment may be given at the site by trained personnel, additional assistance may be required at the site (emergency medical technician), or the victim may have to be transported to the hospital. A first aid kit shall be maintained and readily available on-site.

In life threatening situations, care must begin WITHOUT considering decontamination. Outside protective clothing can be removed if it does not cause delays or aggravate the problem. Respirators must always be removed. Normal decontamination procedures should be followed when at all possible.

The SHSO shall be immediately notified of any accident/incident.

It will be the responsibility of the SHSO to investigate thoroughly the details of any accident or injury. Based on his findings, he will recommend any corrective action relative to field procedures to prevent recurrence.

10.4 FIRE

The potential for fire is significant at many contaminated waste sites. During subsurface operations, explosimeters and photoionization detectors are to be used to monitor levels of potentially combustible gases and volatile organics. Fire extinguishers (Class ABC) will be kept at the working locations. The local fire department will also be alerted to the nature and location of any field activities.

10.5 SITE EVACUATION

Three stages of site evacuation have been determined:

a. Withdraw from immediate work area
b. Withdraw from site
c. Withdraw from area

10.5.1 Withdrawal from Work Area

Withdrawal to a safe upwind location will be required if any of the following occur:

- Occurrence of a minor accident - Field operations will resume after first aid and/or decontamination procedures have been administered.
- Equipment malfunctions.

10.5.2 Evacuation of Site

The site will be evacuated in the following cases:

a. A major accident or injury occurs.

b. Fire and/or explosion occurs.

10.5.3 Evacuation of Areas Near the Facility

The SHSO is responsible for determining if circumstances exist for contamination of areas near the facility, and should always assume worst-case conditions until proven otherwise. Fire and police departments must be contacted. A list of their addresses and telephone numbers will be carried by the Site Manager.

10.6 SAFETY OF THIRD PARTIES

Site access may or may not be controlled at the site and only verified team members, and previously approved personnel will be allowed in work areas or areas containing potentially hazardous materials or conditions.

TABLES

TABLE 1

Summary of Solid Contaminant Concentrations
Former Vermont Bosch Site
Fountain Inn, South Carolina
AMEC Project 6251121007.01.01

Constituent	Units	Sample ID			
		OWS-1	OWS-2	GP-16	GP-17
Acetone	ug/kg	56	44	ND	ND
2-Butanone (MEK)	ug/kg	17	< 11	ND	ND
1,1-Dichloroethane	ug/kg	140	< 5.4	ND	ND
Ethylbenzene	ug/kg	7.4	< 5.4	ND	ND
Tetrachloroethene	ug/kg	ND	ND	1,200	64
Toluene	ug/kg	13	< 5.4	ND	ND
Xylenes (total)	ug/kg	100	< 5.4	ND	ND
Diethylphthalate	ug/kg	89,000	9,600	ND	ND
Bis(2-Ethylhexyl)phthalate	ug/kg	< 520	800	ND	ND
Oil and Grease	mg/kg	420	200	NA	NA

Notes:
ug/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
NA = not analyzed
ND = not detected

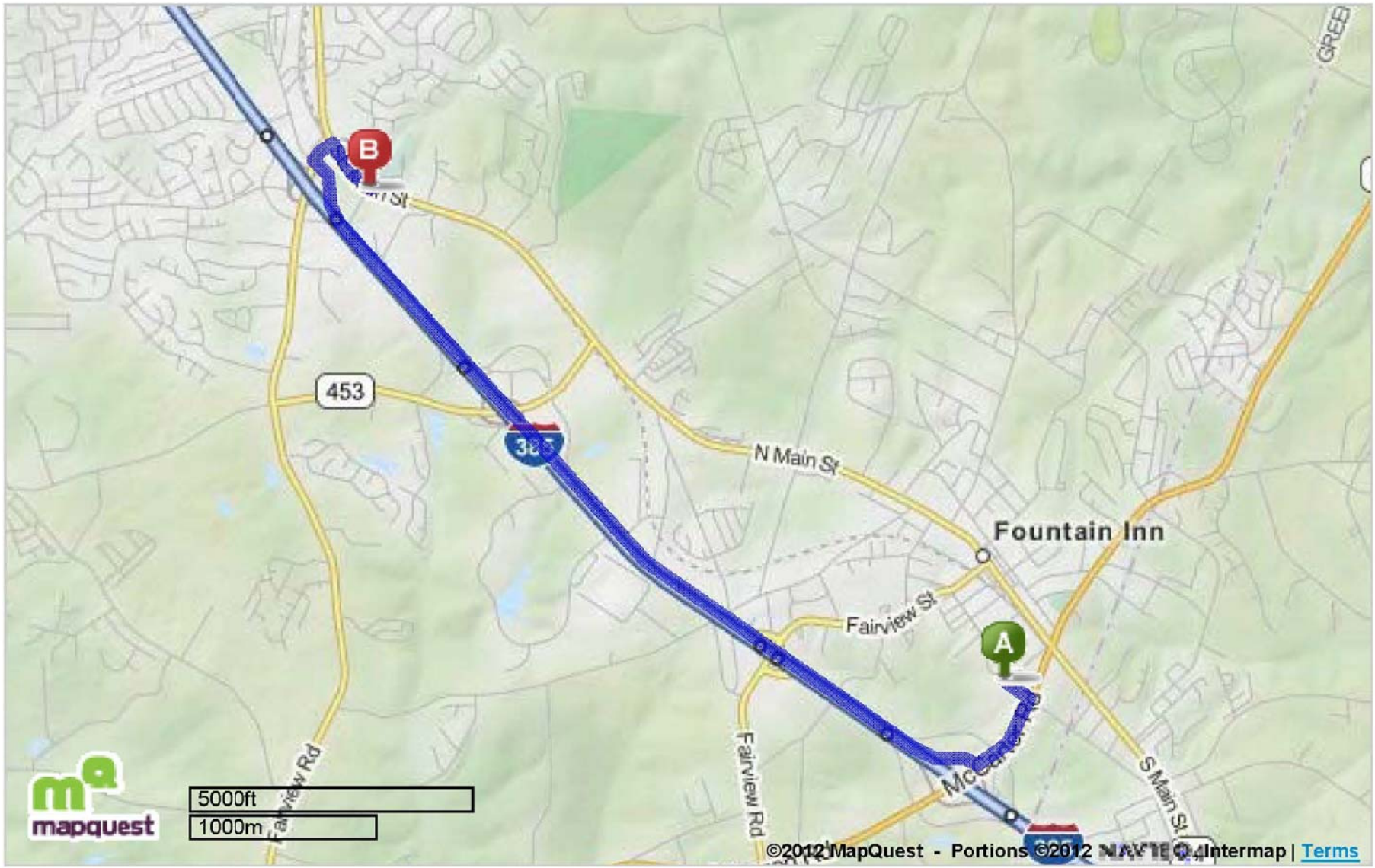
TABLE 2

Summary of Aqueous Contaminant Concentrations
Former Vermont Bosch Site
Fountain Inn, South Carolina
MACTEC Project 6680-05-9578-01

Constituent	Units	MW-1	GP-14	GP-14d	GP-15
Acetone	ug/l	350	ND	ND	ND
2-Butanone (MEK)	ug/l	62	ND	ND	ND
Chloroethane	ug/l	35	ND	ND	ND
1,1-Dichloroethane	ug/l	450	ND	ND	ND
1,1-Dichloroethene	ug/l	26	ND	ND	ND
Ethylbenzene	ug/l	30	ND	ND	ND
Naphthalene	ug/l	10	ND	ND	ND
Styrene	ug/l	76	ND	ND	ND
Tetrachloroethene	ug/l	ND	11,000	53	57
Toluene	ug/l	110	ND	ND	ND
1,1,1-Trichloroethane	ug/l	260	ND	ND	ND
Trichloroethene	ug/l	ND	16	ND	ND
Xylenes (total)	ug/l	450	ND	ND	ND
Di-n-butyl phthalate	ug/l	ND	ND	ND	ND
Diethylphthalate	ug/l	620,000	ND	ND	ND
Oil and Grease	mg/l	690	NA	NA	NA

Notes:
ug/l = micrograms per liter
mg/l = milligrams per liter
NA = not analyzed
ND = not detected

FIGURES



amec
555 N. Pleasantburg Drive
Suite 202
GREENVILLE, S.C. 29607
Phone: (864) 552-9624
Fax: (864) 552-9699

HOSPITAL LOCATION MAP
RBTC FOUNTAIN INN DIVISION
FOUNTAIN INN, SOUTH CAROLINA

FIGURE 1

FILE:	DRAWN BY:	CHECKED BY:	APPROVED BY:	DATE:	JOB NO:
FIGURE 1.DWG	CHB	PSJ	PSJ	9-30-05	6680-05-9578-01

Directions to Hillcrest Hospital

From intersection of Woodside Avenue and McCarter Road, turn left and go approximately 0.28 mile to traffic light at Main Street. Turn left on Main Street and go approximately 4.3 miles to Hillcrest Hospital.

Or

From intersection of Woodside Avenue and McCarter Road, turn right and go approximately 0.5 miles to I-385. Take I-385 North for approximately four miles to Fairview Road exit. Turn right on Fairview Road and go approximately 0.2 mile to South Street. Turn right on South Street and go approximately 0.1 mile to Plain Street. Turn left on Plain Street and go approximately 0.1 mile to SE Main Street. Turn left on SE Main Street and go approximately 0.1 mile to Hillcrest Hospital.

APPENDIX A

PERSONNEL SIGN OFF SHEETS

WORK PRECAUTIONS

1. Prior to beginning any work on the site, the Site Health and Safety Officer shall brief all field personnel on the contents of this plan.
2. No eating, drinking, smoking, chewing gum, or tobacco use is permitted at the work site unless in the designated “clean” area.
3. Wear prescribed safety equipment as directed in the HASP.
4. Remove and discard any clothing that becomes contaminated.
5. Wash exposed skin with soap and water before leaving site.
6. Potable water shall be provided in sufficient quantity to provide emergency washing.
7. Use safe and legal procedures for sample storage and shipment.

PERSONNEL AUTHORIZED TO ENTER SITE

By initialing and dating this form, the listed individual acknowledges that he/she has read and understands, and will comply with the requirements of this HASP.

Name	Date	Initials

APPENDIX B

JOB HAZARD ANALYSIS FORMS

Job Hazard Analysis Form

JHA No.: JHA - GRVL - 11 - 001 - 1

Job Title: Mobilization/Demobilization **Date of Analysis:** 11/10/11

Job Location: All Greenville Projects **Team Leader:** NA

Key Work Steps	Hazards/Potential Hazards	Safe Practices
1. Prepare for Site Visit	N/A	Prior to leaving for site ▪ Obtain and review HASP prior to site visit, if possible ▪ Identify all field personnel and provide a briefing of anticipated activities and hazards. ▪ Identify any medical conditions or sensitivities which could precipitate an emergency. Employees with known allergies are encouraged to make co-workers aware of their condition and ensure they know the correct procedures to take (e.g., how to use an Epi-pen, the location of other medication). Conditions may include: ▪ insect venom allergies ▪ plant allergies ▪ asthma ▪ food or drug allergies ▪ Determine medical and safety supply needs: ▪ epi-pens and Benadryl for allergic reactions [affected employee must provide and carry] ▪ wash water and soap ▪ insect repellant ▪ insecticide (ant, wasp, hornet…) ▪ first aid kits adequate for the number of personnel and nature of work ▪ Determine PPE needs – bring required PPE to the site, if not otherwise being provided at the site (e.g., gloves, steel toed boots) ▪ Determine training and medical monitoring needs and ensure all required Health and Safety training and medical monitoring has been received and is current ▪ If respiratory protection is required/ potentially required, ensure that training and fit-testing has occurred within the past year. ▪ Familiarize yourself with route to the site and the route from the site to designated local medical facilities. ▪ Identify potential shelter locations for hazardous weather conditions (e.g. lightning, hail, tornadoes)
	Vehicle defects	Inspect company owned/leased vehicle for defects or unsafe conditions using the daily and weekly inspection form. Correct all unsafe conditons prior to travel.

1. Prepare for Site Visit (continued)	Insufficient emergency equipment, unsecured loads	Insufficient emergency equipment, unsecured loads ▪ Cell phones are recommended to call for help in the event of an emergency ▪ All tools and equipment must be properly secured. Bungee cords, cargo straps, and a cargo net are available in the truck toolbox.
2. Operating vehicles – general	Collisions, unsafe driving conditions	Drive Defensively! ▪ Seat belts must be used at all times when operating any vehicle on company business. ▪ Drive at safe speed for road conditions ▪ Maintain adequate following distance ▪ Pull over and stop if you have to look at a map ▪ Parking brake must be engaged if the vehicle is to be left running, unattended. ▪ Try to park so that you don't have to back up to leave.
3. Driving to the jobsite	Dusty, winding, narrow roads	Dusty, winding, narrow roads ▪ Drive confidently and defensively at all times. ▪ Go slow around corners, occasionally clearing the windshield.
	Rocky or one-lane roads	Rocky or one-lane roads ▪ Stay clear of gullies and trenches, drive slowly over rocks. ▪ Yield right-of-way to oncoming vehicles--- find a safe place to pull over.
	Stormy weather, other motorists	Stormy weather, other motorists ▪ Inquire about conditions before leaving the office. ▪ Be aware of oncoming storms. ▪ Drive to avoid accident situations created by the mistakes of others.
	When angry or irritated	When angry or irritated ▪ Attitude adjustment; change the subject or work out the problem before driving the vehicle. Let someone else drive.
	Turning around on narrow roads	Turning around on narrow roads ▪ Safely turn out with as much room as possible. ▪ Know what is ahead and behind the vehicle. ▪ Use a backer if available.
	Sick or medicated	Sick or medicated ▪ Let others on the crew know you do not feel well. ▪ Let someone else drive.
	On wet or slippery roads	On wet or slippery roads ▪ Drive slow and safe, wear seatbelts.
3. Driving to the jobsite (continued)	Animals on road	Animals on road ▪ Drive slowly, watch for other animals nearby. ▪ Be alert for animals darting out of wooded areas

4. Gain permission to enter site	Hostile landowner, livestock, pets	Hostile landowner, livestock, pets ▪ Talk to land owner, be courteous and diplomatic ▪ Ensure all animals have been secured away from work area
5. Mobilization/ Demob-ilization of Equipment and Supplies	Struck by Equipment or Vehicles	Struck by heavy equipment ▪ Be aware of equipment/vehicle operations. ▪ Ground personnel in the vicinity of heavy equipment operations will be within the view of the operator at all times ▪ Employees shall wear a high visibility vest or T-shirt (reflective vest required if working at night). ▪ Ground personnel will be aware of the counterweight swing and maintain an adequate buffer zone.
	Struck by Equipment/Supplies	Struck by Equipment/Supplies ▪ Workers will maintain proper space around their work area, if someone enters it, stop work. ▪ When entering another worker's work space, give a verbal warning so they know you are there.
	Overexertion Unloading/Loading Supplies	Overexertion Unloading/Loading Supplies ▪ Train workers on proper body mechanics, do not bend or twist at the waist while exerting force or lifting. ▪ Tightly secure all loads to the truck bed to avoid load shifting while in transit.
	Caught in/on/between	Caught in/on/between ▪ Do not place yourself between two vehicles or between a vehicle and a fixed object.
	Slip/Trip/Fall	Slip/Trip/Fall ▪ Mark all holes and low spots in area with banner tape. Instruct personnel to avoid these areas. ▪ Drivers will maintain 3 point contact when mounting/dismounting vehicles/equipment. ▪ Drivers will check surface before stepping, not jumping down.
	Vehicle accident	Vehicle accident ▪ Employees should follow AMEC vehicle operation policy and be aware of all stationary and mobile vehicles.
6. Driving back from the jobsite	See hazards listed under item #3	See safe work practices under item #3

Form ESH-2.9.1-3.1

Job Hazard Analysis Form

IDENTIFY HAZARDS AND PPE

Complete the checklists for hazard identification and PPE requirements. Information from the RA and applicable permits are included in this section.

Standard Hazards			
☑ Falling Objects	☑ Slips and trips	☑Pinch points	☑ Rotating equipment
☑ Falls	☑ Power equipment/tools	☐ Elevated work surfaces	☐ _____
Eye Hazards			
☐ Particulates	☐ Liquid splashes	☐ Welding Arc	☐ _____
Hearing Hazards			
☑ None	☐ Impact noise	☐ High frequency noise	☐ High ambient noise
Respiratory Hazards			
☑ None	☐ Dust/particulates	☐ Organic Vapors	☐ Acid Gases
☐ Oxygen deficient	☐ Welding fumes	☐ Aerosols/Particulates	☐ Be, Hg, Cr, Pb
☐ _____	☐ Radon	☐ Asbestos	☐ _____
Chemical Hazards			
☑ None	☐ Organic solvents	☐ Reactive metals	☐ PCBs
☐ Acids / bases	☐ Oxidizers	☐ Volatiles / Semi-volatiles	☐ _____

Job Hazard Analysis Form

Environmental Hazards			
☐ None	☑ Temperature extremes or hazardous weather	☑ Wet location	☐ Bio hazards (snakes, insects, spiders, bird / mouse droppings, fungus, etc.)
☐ Explosive vapors	☐ Confined space	☐ Engulfment Hazard	☐ _____
Electrical Hazards			
☐ None	☐ Energized equipment or circuits	☐ Overhead utilities ☐ Underground utilities ☐ Hidden utilities	☐ Wet location
Fire Hazards			
☑ None	☐ Cutting, welding, or grinding generated sparks or heat sources	☐ Flammable materials present	☐ Oxygen enriched location
Ergonomic Hazards			
☑Lifting	☑ Bending	☑ Twisting	☐ Pulling/tugging
Computer Use in the: ☐ Office ☐ Field	☐ Repetitive motion	☐ _____	☐ _____
Radiological Hazards			
☑ None	☐ Loose contamination	☐ Fixed Contamination	☐ Radiation
☐ Airborne contamination	☐ Radon	☐ EMF	☐ Criticality
☐ Alpha	☐ Beta	☐ Gamma/X-rays	☐ Neutron
☐ Tritium	☐ TRU	☐ Depleted Uranium	☐ Enriched Uranium
Other Hazards			
☑ Allergic reaction to insect stings. See Attachments A and B to this JHA.			
☐			
☐			
☐			

Completed by: Gary W. Wise Date: 11/4/11

FORM ESH-2.9.1-3.3

Job Hazard Analysis Form

PPE AND MONITORING REQUIREMENTS

Standard PPE			
☐ Hard Hat	☐ Safety shoes	☐ Safety glasses	☐ Boot Covers
☐ Aprons	☐ Rubber Boots	☐ Other:_____	☐ Other:_____
Eye Protection			
☐ Welding glasses ☐ Welding helmet	☐ Face shield	☐ Chemical goggles	☐ Welding screens
Hearing Protection			
☐ Ear plugs	☐ Ear Muffs	☐ Ear plugs and muffs	☐ Other _____
Respiratory Protection			
☑ None	☐ Dust mask	☐ Full Face APR ☐ Half Face APR Cart. Type _____	☐ PAPR Cart. Type _____
☐ SCBA	☐ Airline respirator	☐ _____	☐ _____
Protective Clothing			
☐ Tyvek® coveralls	☐ Poly-coated Tyvek® Coveralls	☐ Saranex® Coveralls	☐ Fully encapsulating suit
☐ Cotton coveralls	☐ Modesty Clothing	☐ Fire resistant clothing	☐ Other _____
Hand Protection			
☑ None	☐ Cotton gloves	☐ Leather gloves	☐ Glove liners
☐ Nitrile gloves ☐ Viton® gloves ☐ Butyl gloves ☐ Neoprene gloves	Surgical gloves ☐ Latex ☐ Non-Latex	☐ Cut-resistant gloves	☐ Other _____
Monitoring Requirements			
☐ Oxygen	☐ Flammable gases/vapors	☐ Toxic Gas/vapors	☐ Hydrogen Sulfide/Carbon Monoxide
☐ Asbestos	☐ Full time IH coverage	☐ Part time IH coverage	☐ Be, Hg, Cr, Pb
☐ Metals Specify: _____			
☐ Organic vapors Specify: _____			
☐ Radioactive air particulates	☐ TLD required	☐ CAM	☐ Radon
☐ Full time RCT coverage	☐ Part time RCT coverage	☐ Radioactive air particulates	☐ Other _____
☐ Other _____		☐ Other _____	

PPE and monitoring requirements completed by: Gary W. Wise Date: 11/4/11

FORM ESH-2.9.1-3.4

Job Hazard Analysis Form

JHA Preparation Team

Gary W. Wise

Effective Date From: November 4, 2011 through November 3, 2012

Approval Signatures

Job Supervisor	Date	HSE Coordinator Gary Wise	11/14/11	RSO	Date
HSE Manager	Date	Project Manager	11/14/11	Other	Date

Form ESH-2.9.1-3.5

Pre-Job Brief Attendance Sheet

Documents included in this briefing: _____

Date of Briefing: _____

Print/Type Name	Signature	Employee Number

Briefing presented by: _____ **Signature:** _____

Form ESH-2.9.1- 4

ATTACHMENT A

Sarah Rice has a known allergy to the venom from bee, wasp, hornet, and fire ant stings. Ms. Rice will have a "sting kit" in her possession containing an "epi-pen" and/or liquid Benadryl. Prior to commencing work, all field personnel will be briefed by Ms. Rice on the proper use of the sting kit materials.

Precautionary Measures

- Ms Rice shall not be required to work alone in locations where stinging insects may be present.
- Look for insect activity continually and especially when moving or preparing to set up.
- Be prepared to withdraw and adjust work plans.

Response to a Sting

- If able to do so, Ms. Rice will administer the epi-pen herself
- If she is unable or requests assistance, apply the pen to the quadricep (front thigh muscle)
- Observe her condition for signs of anaphylactic shock (see Attachment B)
- Apply first aid – If embedded, remove the stinger with a scraper. Ice packs may reduce pain and swelling if available.
- Seek medical assistance:

  For a mild reaction with no evidence of shock, call WorkCare for guidance.
  For a severe reaction or potential onset of shock, call 911.
  Wait for transport or drive to the emergency room as directed by the operator.

Attachment B

SAFETY FLASH

July 26, 2004

amec

WORKING TOGETHER TO PROTECT PERSONNEL AND THE ENVIRONMENT

ALLERGIC REACTION TO A BEE STING[1]

Incident Description

On June 23, 2004 an AEE employee was stung by a bee during a wetland delineation and had severe allergic reaction (anaphylactic reaction) requiring two emergency hospital visits. The employee had no previous history or knowledge of her allergy to insect stings. The immediate reaction to the sting was characterized by hives, itchiness, and swelling in areas other than the sting site, burning sensation to the lips and mouth, and dizziness. The employee was working alone during the initial event. She was able to reach her car and drive to the field project site. The ambulance was called immediately and the employee was treated at the hospital and released. A second severe allergic reaction followed the next morning requiring another 911 call and an additional treatment at another hospital.

Quick Facts:

Up to 150 people a year die in the US only as a result of an allergic reaction to a sting from yellow jackets, hornets, wasps, bees, and fire ants.
Patients who have experienced a systemic allergic reaction to an insect sting have a 60% chance of a similar (or worse) reaction if stung again.

Types of Insect Sting Reactions

Most people are not allergic to insect stings and should recognize the difference between an allergic reaction (a life-threatening event) and a normal or large local reaction. The severity of an insect sting reaction varies from person to person. A normal reaction will result in pain, swelling, and redness confined to the sting site. Simply disinfect the area and apply ice to reduce the swelling. A large local reaction will result in swelling that extends beyond the sting site. For example, a person stung on the forearm may have his/her entire arm swell to twice it's normal size. Although alarming in appearance, this condition is often treated the same as a normal reaction. However, because this condition may persist for 2-3 days, antihistamines and steroids are sometimes prescribed to lessen the discomfort.
The most serious reaction to an insect sting is an allergic one. This condition requires immediate medical attention. Symptoms of an allergic reaction or "anaphylaxis" may include one or more of the following:

[1] Materials developed by the American College of Allergy, Asthma & Immunology used in this document.

- Hives, itching, and swelling in areas other than the sting site.
- Tightness in the chest and difficulty in breathing.
- Hoarse voice or swelling of the tongue.
- Dizziness or a sharp drop in blood pressure.
- Unconsciousness or cardiac arrest.

This type of reaction can occur within minutes after the sting and ***may be life threatening or even fatal***. People who have experienced an allergic reaction to an insect sting have a 60% chance of a similar or worse reaction if stung again.

Facts on Stinging Insects

The majority of insect stings come from yellow jackets, hornets, wasps, bees, and fire ants. These insects occur throughout the U.S. except for fire ants which are found only in the Southeastern states.

1. Over 2 million Americans are allergic to stinging insects (or approximately 0.7 percent. Applying this information to AEE we can estimate that approximately 14 AEE employees may be allergic to stinging insects and have an anaphylactic reaction when stung).
2. More than 500,000 people enter hospital emergency rooms every year suffering from insect stings and 40-150 people die as a result of an allergic reaction to these stings.
3. An allergic reaction to an insect sting can occur immediately, within minutes, or even hours after the sting (although never more than 24 hrs.). Such a reaction is characterized by hives, itchiness, and swelling in areas other than the sting site, difficulty in breathing, dizziness or a sharp drop in blood pressure, nausea, cramps or diarrhea, unconsciousness and cardiac arrest.
4. Patients who have experienced a systemic allergic reaction to an insect sting have a 60% chance of a similar (or worse) reaction if stung again.
5. An allergic reaction in progress can be stopped with epinephrine, either self-injected (prescribed) or administered by a doctor. People who carry these sting kits must keep them close at hand wherever they go and remember that one dose is not always enough to stop a reaction. If you are stung, seek medical attention immediately.
6. A person suffering from insect sting allergy can have this condition treated with venom immunotherapy (VIT), a 97% effective desensitization therapy administered by an allergist.
7. Stinging insects are most active during the summer and early fall when nest populations can exceed 60,000 insects.
8. These insects are most dangerous in the vicinity of their nests. A passer-by is viewed as a threat to the safety of their home and is often chased out of the area by a sting(s).
9. Yellow jackets, hornets, and wasps can sting repeatedly. Honeybees have barbed stingers which are left behind in their victims' skin. These stingers are best removed by a scraping action rather than a pulling motion which actually squeezes more venom into the skin.
10. Stinging insects are especially attracted to sweet fragrances (perfumes, colognes, and hair sprays), picnic food, open soda and beer containers, and

garbage areas. Avoiding these attractants will lessen a person's chance of being stung.

Tips on Avoiding Insect Stings

The following precautions are suggested:

1. In the field, always wear shoes that protect your whole foot[2]. Long pants and sleeves are recommended.
2. Insect repellents DO NOT work against stinging insects.
3. Never swat or flail at a flying insect. If need be, gently brush it aside or patiently wait for it to leave.
4. DO NOT drink from open beverage cans. Stinging insects will crawl inside a can attracted by the sweet beverage.
5. When eating outdoors, try to keep food covered at all times. Stinging insects are fond of the same foods you are.
6. Garbage cans stored outside should be covered with tight- fitting lids.
7. Avoid wearing sweet-smelling perfumes, hairsprays, colognes, and deodorants.
8. Avoid wearing bright colored clothing with flowery patterns. Bees may mistake you for a flower.
9. If you have had an allergic reaction to an insect sting, it is important that you see an allergist. You have a 60% chance of having a similar, or worse reaction if stung again. There is a treatment, venom immunotherapy, which is 97% effective

AEE Field Safety Practices

1. If a member of the field team is allergic to insect bites or stings, this should be made known to all members of the team and noted in the emergency information section of the field safety folder and/or Health and Safety Plan.
2. A member of the field team allergic to insect bites or stings should never work alone.
3. Treatment and emergency procedures should be reviewed before field activity begins.
4. Persons with known allergic reactions to insects should wear or carry on their person medical alert identification, and ***carry sting kits*** for use in emergencies.

Additional Information (Insect Venom Allergies by Harvard Medical School' Consumer Health Information)

For further information, please contact:

Lori Dowling Phone: (250) 564-3243 Email: lori.dowling@amec.com	Vladimir Ivensky Phone: (610) 810-6144 Email: vladimir.ivensky@amec.com

[2] Non work related recommendation: Avoid walking barefoot in the grass (honeybees and bumblebees forage on white clover, a weed that grows in lawns throughout the North America).

Job Hazard Analysis Form

JHA No.: JHA - GRVL - 12 - 004 - 0

Job Title: General Field Work **Date of Analysis:** 5/31/12

Job Location: Greenville, SC Projects **Team Leader:** Paul S. Johnstone

Key Work Steps	Hazards/Potential Hazards	Safe Practices
Mobilization / Demobilization	See Mobilization/Demobilization JHA	See Mobilization/Demobilization JHA
Communication	Safety, crew unity	▪ Each work day shall begin with a tailgate safety briefing. Topics shall include planned work, site and work hazards, PPE requirements, and communication methods. ▪ Let other crewmembers know when you see a hazard. ▪ Avoid working near known hazards. ▪ Always know the wherabouts of fellow crewmembers. ▪ Carry a radio and spare batteries or cell phone ▪ Review Emergency Evacuation Procedures (see below).
Walking and working in the field	Falling down, twisted ankles and knees, poor footing	▪ Slow down and use extra caution around logs, rocks, and animal holes. ▪ Extremely steep slopes (>50%) can be hazardous under wet or dry conditions; consider an alternate route. ▪ Wear laced boots with a minimum 8" high upper and non-skid soles for ankle support and traction.
	Falling objects	▪ Wear your hardhat for protection from falling limbs and pinecones, and from tools and equipment carried by other crewmembers. ▪ Stay out of the woods during extremely high winds.

	Damage to eyes	▪ Watch where you walk, ecpecially around trees and brush with limbs sticking out. ▪ Exercise caution when clearing limbs from tree trunks. Advise wearing eye protection. ▪ Ultraviolet light from the sun can be damaging to the eyes; look for sunglasses that specify significant protection from UV-A and UV-B radiation. If safety glasses required, use glasses with tinted lenses
	Bee and wasp stings	▪ Be alert to hives in brush or in hollow logs. Watch for insects travelling in and out of one location. ▪ If you or anyone you are working with is known to have allergic reactions to bee stings, tell the rest of the crew and your supervisor. Make sure you carry emergency medication with you at all times. ▪ Wear long sleeve shirts and trousers; tuck in shirt. Bright colors and metal objects may attract bees. ▪ If you are stung, cold compresses may bring relief. ▪ If a stinger is left behind, scrape it off the skin. Do not use a tweezers as this squeezes the venom sack, worsening the injury. ▪ If the victim develops hives, asthmatic breathing, tissue swelling, or a drop in blood pressure, seek medical help immediately. Give victim antihistamine, (Benadryl, chlo-amine tabs).
	Ticks and infected mosquitos	▪ Spray clothing with insect repellant as a barrier. ▪ Wear light colored clothing that fits tightly at the wrists, ankles, and waist. ▪ Each outer garment should overlap the one above it. ▪ Cover trouser legs with high socks or boots. ▪ Tuck in shirt tails. ▪ Search the body on a regular basis, especially hair and clothing; ticks generally do not attach for the first couple of hours.

	Ticks and infected mosquitos	▪ If a tick becomes attached, pull it by grasping it as close as possible to the point of attachment and pull straight out with gentle pressure. Wash skin with soap and water then cleanse with rubbing alcohol. Place the tick in an empty container for later identification, if the victim should have a reaction. Record dates of exposure and removal. ▪ Do not try to remove the tick by burning with a match or covering it with chemical agents. ▪ If you can not remove the tick, or the head detaches, seek prompt medical help. ▪ Watch for warning signs of illness: a large red spot on the bite area; fever, chills, headache, joint and muscle ache, significant fatigue, and facial paralysis are reactions that may appear within two weeks of the attack. Symptoms specific to Lyme disease include: confusion, short-term memory loss, and disorientation. ▪ Avoid heavy scents. ▪ Use insect repellants. If using permethrin, do not apply directly to skin, apply to clothing only. ▪ Carry after-bite medication to reduce skin irritation.
	Contact with poisonous plants or the oil from poisonous plants	▪ Look for signs of poisonous plants and avoid. ▪ Wear PPE as described in the HASP. ▪ Do not touch anything part of your body/clothing. ▪ Always wash gloves before removing them. ▪ Discard PPE in accordance with the HASP.
	Back Injuries	▪ Site personnel will be instructed on proper lifting techniques. ▪ Mechanical devices should be used to reduce manual handling of materials. ▪ Team lifting should be utilized if mechanical devices are not available.

	Slips/Trips/Falls	▪ Maintain work areas safe and orderly; unloading areas should be on even terrain; mark or repair possible tripping hazards. ▪ Site SHSO inspect the entire work area to identify and mark hazards.
	Vehicular Traffic	▪ Spotters will be used when backing up trucks and heavy equipment and when moving equipment. ▪ High visibility vests will be worn when workers are exposed to vehicular traffic at the site or on public roads.
	Overhead Hazards (if applicable) – includes drilling, heavy equipment operation, elevated work, and equipment or materials placed more than 5 feet above ground or floor level	▪ Personnel will be required to wear hard hats that meet ANSI Standard Z89.1. ▪ All ground personnel will stay clear of suspended loads. ▪ All equipment will be provided with guards, canopies or grills to protect the operator from falling or flying objects. ▪ All overhead hazards will be identified prior to commencing work operations.
	Dropped Objects (as applicable) – includes drum handling, drilling, and working near heavy equipment	▪ Steel toe boots meeting ANSI Standard Z41 will be worn.
	Noise (as applicable) – includes drilling, jackhammering, and powered equipment operation	▪ Hearing protection will be worn with a noise reduction rating capable of maintaining personal exposure below 85 dBA (ear muffs or plugs); all equipment will be equipped with manufacturer's required mufflers. Hearing protection shall be worn by all personnel working in or near heavy equipment.
	Eye Injuries (as applicable) – includes brush clearing, power tool use, drilling, hammering, etc.	▪ Safety glasses meeting ANSI Standard Z87 will be available and worn during tasks which may generate eye hazards.
	Heavy Equipment (overhead hazards, spills, struck by or against)	▪ Equipment will have seat belts. ▪ Operators will wear seat belts when operating equipment. ▪ Do not operate equipment on grades that exceed manufacturer's recommendations.

	Heavy Equipment (overhead hazards, spills, struck by or against)	▪ Equipment will have guards, canopies or grills to protect from flying objects. ▪ Ground personnel will not approach equipment until operator indicates it is safe. ▪ Ground personnel will stay clear of all suspended loads. ▪ Ground personnel will wear high visibility vests ▪ Spill and absorbent materials will be readily available. ▪ Drip pans, polyethylene sheeting or other means will be used for secondary containment. ▪ Ground personnel will stay out of the swing radius of excavators. ▪ Eye contact with operators will be made before approaching equipment. ▪ Operator will acknowledge eye contact by removing his hands from the controls. ▪ Equipment will not be approached on blind sides. ▪ All equipment will be equipped with backup alarms and use spotters when significant physical movement of equipment occurs on-site, (i.e., other than in place excavation or truck loading).
	Struck by vehicle/equipment	▪ Be aware of heavy equipment operations. ▪ Keep out of the swing radius of heavy equipment. ▪ Ground personnel in the vicinity of heavy equipment operations will be within the view of the operator at all times and will wear high visibility vests. ▪ Ground personnel will be aware of the counterweight swing and maintain an adequate buffer zone. ▪ Ground personnel will not stand directly behind heavy equipment when it is in operation. ▪ Drivers will keep workers on foot in their vision at all times, if you lose sight of someone, Stop!

	Struck/cut by tools	▪ Cut resistant work gloves will be worn when dealing with sharp objects. ▪ All hand and power tools will be maintained in safe condition. ▪ Guards will be kept in place while using hand and power tools.
	Caught in/on/between	▪ Workers will not position themselves between equipment and a stationary object. ▪ Workers will not wear long hair down (place in pony-tail and tuck into shirt), scarves, or jewelry if working with tools/machinery.
	Contact with Electricity/Lightning	▪ All electrical tools and equipment will be protected by GFCI. ▪ Electrical extension cords will be of the "Hard" or "Extra Hard" service type. ▪ All extension cords shall have a three-blade grounding plug. ▪ Personnel shall not use extension cords with damaged outer covers, exposed inner wires, or splices. ▪ Electrical cords shall not be laid across roads where vehicular traffic may damage the cord without appropriate guarding. ▪ All electrical work will be conducted by a licensed electrician. ▪ All utilities will be marked prior to excavation activities. ▪ All equipment will stay a minimum of 10 feet from overhead energized electrical lines (50 kV). This distance will increase by 4 inches for each 10 kV above 50 kV. Rule of Thumb: Stay 10 feet away from all overhead powerlines known to be 50 kV or less and 35 feet from all others.) ▪ The SHSO shall halt outdoor site operations whenever lightning is visible, outdoor work will not resume until 30 minutes after the last sighting of lightning.

	Equipment failure	▪ All equipment will be inspected before use. If any safety problems are noted, the equipment should be tagged and removed from service until repaired or replaced.
	Hand & power tool usage.	▪ Daily inspections will be performed. ▪ Remove broken or damaged tools from service. ▪ Use the tool for its intended purpose. ▪ Use in accordance with manufacturers instructions.
Environmental health considerations	Heat Stress	▪ Remain constantly aware of the four basic factors that determine the degree of heat stress (air temperature, humidity, air movement, and heat radiation) relative to the surrounding work environmental heat load. ▪ Know the signs and symptoms of heat exhaustion, heat cramps, and heat stroke. Heat stroke is a true medical emergency requiring immediate emergency response action. *NOTE:* The severity of the effects of a given environmental heat stress is decreased by reducing the work load, increasing the frequency and/or duration of rest periods, and by introducing measures which will protect employees from hot environments. ▪ Maintain adequate water intake by drinking water periodically in small amounts throughout the day (flavoring water with citrus flavors or extracts enhances palatability). ▪ Allow approximately 2 weeks with progressive degrees of heat exposure and physical exertion for substantial acclimatization. ▪ Acclimatization is necessary regardless of an employee's physical condition (the better one's physical condition, the quicker the acclimatization). Tailor the work schedule to fit the climate, the physical condition of employees, and mission requirements.

		▪ A reduction of work load markedly decreases total heat stress. ▪ Lessen work load and/or duration of physical exertion the first days of heat exposure to allow gradual acclimatization. ▪ Alternate work and rest periods. More severe conditions may require longer rest periods and electrolyte fluid replacement.
	Wet Bulb Globe Temperature (WBGT) Index	▪ Curtail or suspend physical work when conditions are extremely severe (see attached Heat Stress Index). ▪ Compute a Wet Bulb Globe Temperature Index to determine the level of physical activity (take WBGT index measurements in a location that is similar or closely approximates the environment to which employees will be exposed). WBGT THRESHOLD VALUES FOR INSTITUTING PREVENTIVE MEASURES 80-90 degrees F — Fatigue possible with prolonged exposure and physical activity. 90-105 degrees F — Heat exhaustion and heat stroke possible with prolonged exposure an 105-130 degrees F — Heat exhaustion and heat stroke are likely with prolonged heat exposure and
	Cold Extremes	▪ Cover all exposed skin and be aware of frostbite. While cold air will not freeze the tissues of the lungs, slow down and use a mask or scarf to minimize the effect of cold air on air passages. ▪ Dress in layers with wicking garments (those that carry moisture away from the body – e.g., cotton) and a weatherproof slicker. A wool outer garment is recommended.

	Cold Extremes	▪ Take layers off as you heat up; put them on as you cool down. ▪ Wear head protection that provides adequate insulation and protects the ears. ▪ Maintain your energy level. Avoid exhaustion and over-exertion which causes sweating, dampens clothing, and accelerates loss of body heat and increases the potential for hypothermia. ▪ Acclimate to the cold climate to minimize discomfort. ▪ Maintain adequate water/fluid intake to avoid dehydration.
	Wind	▪ Wind chill greatly affects heat loss (see attached Wind Chill Index). ▪ Avoid old, defective timber, especially hardwoods, during periods of high winds due to snag hazards.

Form ESH-2.9.1-3.1

Job Hazard Analysis Form

Identify Hazards and PPE

Complete the checklists for hazard identification and PPE requirements. Information from the RA and applicable permits are included in this section.

Standard Hazards			
☑ Falling Objects	☑ Slips and trips	☑ Pinch points	☐ Rotating equipment
☑ Falls	☐ Power equipment/tools	☐ Elevated work surfaces	☑ Vehicular traffic
Eye Hazards			
☐ Particulates	☐ Liquid splashes	☐ Welding Arc	☑ Other
Hearing Hazards			
☐ None	☐ Impact noise	☑ High frequency noise	☑ High ambient noise
Respiratory Hazards			
☑ None	☐ Dust/particulates	☐ Organic Vapors	☐ Acid Gases
☐ Oxygen deficient	☐ Welding fumes	☐ Aerosols/Particulates	☐ Be, Hg, Cr, Pb
☐ _____	☐ Radon	☐ Asbestos	☐ _____
Chemical Hazards			
☑ None	☐ Organic solvents	☐ Reactive metals	☐ PCBs
☐ Acids / bases	☐ Oxidizers	☐ Volatiles / Semi-volatiles	☐ _____
Environmental Hazards			
☐ None	☑ Temperature extremes	☐ Wet location	☑ Bio hazards (snakes, insects, spiders, bird / mouse droppings, fungus, etc.)
☐ Explosive vapors	☐ Confined space	☐ Engulfment Hazard	☐ _____
Electrical Hazards			
☐ None	☑ Energized equipment or circuits	☐ Overhead utilities ☐ Underground utilities ☐ Hidden utilities	☐ Wet location
Fire Hazards			
☑ None	☐ Cutting, welding, or grinding generated sparks or heat sources	☐ Flammable materials present	☐ Oxygen enriched location

Form ESH-2.9.1-3.2

Job Hazard Analysis Form

Ergonomic Hazards			
☑ Lifting	☑ Bending	☑ Twisting	☑ Pulling/tugging
Computer Use in the: ☐ Office ☐ Field	☐ Repetitive motion	☐ _____	☐ _____
Radiological Hazards			
☑ None	☐ Loose contamination	☐ Fixed Contamination	☐ Radiation
☐ Airborne contamination	☐ Radon	☐ EMF	☐ Criticality
☐ Alpha	☐ Beta	☐ Gamma/X-rays	☐ Neutron
☐ Tritium	☐ TRU	☐ Depleted Uranium	☐ Enriched Uranium
Other Hazards			
☑ Heavy equipment			
☑ Hand and power tool usage			
☑ Equipment failure			
☐			

Completed by: Gary W. Wise Date: 5/31/12

Form ESH-2.9.1-3.3

Job Hazard Analysis Form

PPE and Monitoring Requirements

Standard PPE			
☑ Hard Hat (as required)	☑ Safety shoes (as required)	☑ Safety glasses	☐ Boot Covers
☐ Aprons	☐ Rubber Boots	☐ Other:_____	☐ Other:_____
Eye Protection			
☐ Welding glasses ☐ Welding helmet	☐ Face shield	☐ Chemical goggles	☐ Welding screens
Hearing Protection			
☑ Ear plugs	☐ Ear Muffs	☐ Ear plugs and muffs	☐ Other _____
Respiratory Protection			
☑ None	☐ Dust mask	☐ Full Face APR ☐ Half Face APR Cart. Type _____	☐ PAPR Cart. Type _____
☐ SCBA	☐ Airline respirator	☐ _____	☐ _____
Protective Clothing			
☐ Tyvek® coveralls	☐ Poly-coated Tyvek® Coveralls	☐ Saranex® Coveralls	☐ Fully encapsulating suit
☐ Cotton coveralls	☐ Modesty Clothing	☐ Fire resistant clothing	☐ Other _____
Hand Protection			
☐ None	☐ Cotton gloves	☑ Leather gloves	☐ Glove liners
☐ Nitrile gloves ☐ Viton® gloves ☐ Butyl gloves ☐ Neoprene gloves	Surgical gloves ☐ Latex ☐ Non-Latex	☐ Cut-resistant gloves	☐ Other _____
Monitoring Requirements			
☐ Oxygen	☐ Flammable gases/vapors	☐ Toxic Gas/vapors	☐ Hydrogen Sulfide/Carbon Monoxide
☐ Asbestos	☐ Full time IH coverage	☐ Part time IH coverage	☐ Be, Hg, Cr, Pb
☐ Metals Specify: _____			
☐ Organic vapors Specify: _____			
☐ Radioactive air particulates	☐ TLD required	☐ CAM	☐ Radon
☐ Full time RCT coverage	☐ Part time RCT coverage	☐ Radioactive air particulates	☐ Other _____
☐ Other _____		☐ Other _____	

PPE and monitoring requirements completed by: Gary W. Wise Date: 5/31/12

Form ESH-2.9.1-3.4

Job Hazard Analysis Form

JHA Preparation Team

Paul S. Johnstone Gary W. Wise

Effective Date From: May 31, 2012 through May 30, 2013

Approval Signatures

Job Supervisor	Date	G Wise 5/31/12 LHSR	Date	RSO	Date
ES&H Manager	Date	P.S. Johnstone 5/31/12 Project Manager	Date	Other	Date

Pre-Job Brief Attendance Sheet

Documents included in this briefing: _____

Date of Briefing: _____

Print/Type Name	Signature	Employee Number

Briefing presented by: _____ **Signature:** _____

TABLE III:4-2. PERMISSIBLE HEAT EXPOSURE THRESHOLD LIMIT VALUE

	Work Load*		
Work/rest regimen	**Light**	**Moderate**	**Heavy**
Continuous work	30.0°C (86°F)	26.7°C (80°F)	25.0°C (77°F)
75% Work, 25% rest, each hour	30.6°C (87°F)	28.0°C (82°F)	25.9°C (78°F)
50% Work, 50% rest, each hour	31.4°C (89°F)	29.4°C (85°F)	27.9°C (82°F)
25% Work, 75% rest, each hour	32.2°C (90°F)	31.1°C (88°F)	30.0°C (86°F)

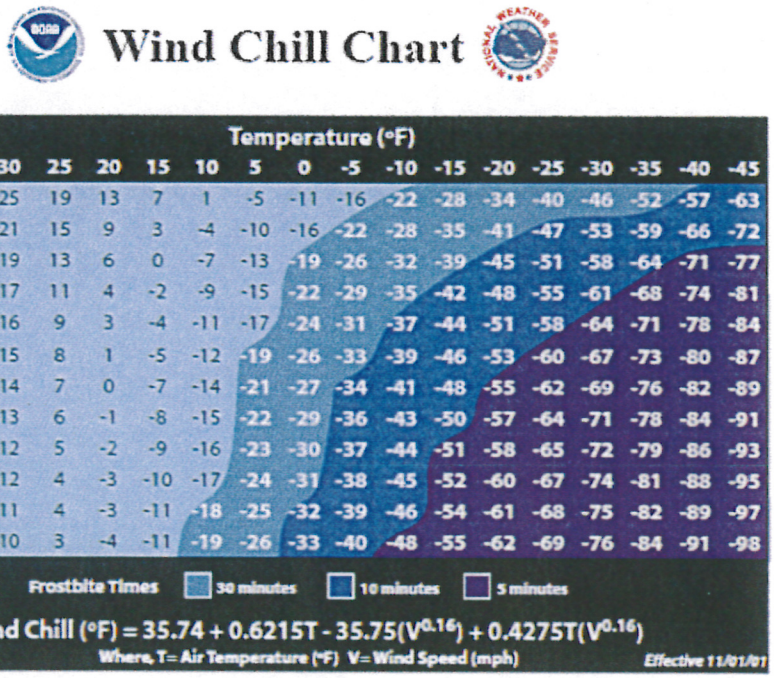
*Values are in °C and °F, WBGT.

These TLV's are based on the assumption that nearly all acclimatized, fully clothed workers with adequate water and salt intake should be able to function effectively under the given working conditions without exceeding a deep body temperature of 38°C (100.4° F). They are also based on the assumption that the WBGT of the resting place is the same or very close to that of the workplace. Where the WBGT of the work area is different from that of the rest area, a time-weighted average should be used (consult the ACGIH *1992-1993 Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices* (1992).

These TLV's apply to physically fit and acclimatized individuals wearing light summer clothing. If heavier clothing that impedes sweat or has a higher insulation value is required, the permissible heat exposure TLV's in Table III:4-2 must be reduced by the corrections shown in Table III:4-3.

Attachment 1

Source: OSHA Technical Manual (OTM)



Attachment 2

Source: http://www.nws.noaa.gov/om/windchill/images/windchillchart3.pdf

Job Hazard Analysis Form

JHA No.: JHA - GRVL - 12 - 003 - 0

Job Title: Groundwater Sampling **Date of Analysis:** 5/31/12

Job Location: All Greenville Projects **Team Leader:** Paul S. Johnstone

Key Work Steps	Hazards/Potential Hazards	Safe Practices
Prepare for sampling event.	Chemical exposure	Review constituents of concern and determine air monitoring and PPE needs.
Carry and load/unload sample coolers and other equipment.	Back or muscle strain	Employ proper ergonomics (lifting techniques); get help from a coworker to carry and load heavy coolers and other equipment; use mechanical aids if available.
Travel to and from site	Moving vehicles on road or in parking areas; traffic hazards	See Mobilization/Demobilization JHA
Calibrate monitoring equipment	Exposure to calibration gases	Review equipment manuals; calibrate in a clean, well-ventilated area
Opening the well cap, taking water level readings	Contact with poisonous plants or oil from poisonous plants. Contact with biting/stinging insects (i.e., spiders, bees, wasps, etc.) which may have constructed a nest in the well cap/well. Exposure to hazardous inhalation and contact with hazardous substances (contaminated ground-water); liquid splash; flammable atmospheres. Foot injuries from dropped equipment.	See General Field Work JHA See General Field Work JHA Allow well to vent for several minutes before obtaining water levels or sampling; wear PPE while sampling and decontaminating equipment; Be aware of moving objects; make sure you have a good grip when lifting and carrying objects; don't carry more than you can handle; wear steel-toed boots.
Collecting water samples	Exposure to contaminants. Exposure to water preservatives. Slips/ trips/falls.	Stand up wind when sampling; monitor breathing zone with appropriate monitoring equipment, if required; wear appropriate PPE. Work in well-ventilated area, up wind of samples; wear appropriate PPE. Ground can become wet/muddy due to spilled water; place all purged water in drums for removal; wear good, slip-resistant footwear.

	Repetitive motion and other ergonometric issues.	Use safe lifting techniques; use mechanical means, where possible, to raise and lower equipment into well; alternate raising and lowering equipment or bailing the well between field sampling team members.
	Electrocution.	Only use electrical equipment with a ground fault circuit interrupter (GFCI); use only correctly grounded equipment (never use three-pronged cords which have the third prong broken off); do not allow electrical cords to come in contact with water; do not stand in water while operating electrical equipment; when unplugging cord, pull on plug rather than cord; make sure all electrically-powered sampling equipment is in good repair; don't repair electrical equipment unless you are both authorized and qualified to do so.
	Fire/explosion/contamination hazard from refueling generators.	Turn generator off and allow it to cool down before refueling; segregate fuel and other hydrocarbons from samples to minimize contamination potential; transport fuels in approved safety containers.
Sample processing	Contaminated water	Wear appropriate PPE; prevent water from contacting skin; work in well-ventilated area; place contaminated water in appropriate labeled containers; stage containers for future pick up and disposal.
Shipping samples	Freeze burns, back strain, hazardous chemical exposure, sample leakage.	Wear appropriate PPE; wear protective gloves when handling ice; follow safe lifting techniques (get help lifting heavy coolers); samples that contain hazardous materials under the DOT definition must be packaged, manifested, and shipped by personnel that have had the appropriate DOT HAZMAT training.

Form ESH-2.9.1-3.1

Job Hazard Analysis Form

IDENTIFY HAZARDS AND PPE

Complete the checklists for hazard identification and PPE requirements. Information from the RA and applicable permits are included in this section.

Standard Hazards			
☑ Falling Objects	☑ Slips and trips	☐ Pinch points	☐ Rotating equipment
☑ Falls	☑ Power equipment/tools	☐ Elevated work surfaces	☐ _____
Eye Hazards			
☐ Particulates	☐ Liquid splashes	☐ Welding Arc	☐ _____
Hearing Hazards			
☑ None	☐ Impact noise	☐ High frequency noise	☐ High ambient noise
Respiratory Hazards			
☑ None	☐ Dust/particulates	☐ Organic Vapors	☐ Acid Gases
☐ Oxygen deficient	☐ Welding fumes	☐ Aerosols/Particulates	☐ Be, Hg, Cr, Pb
☐ _____	☐ Radon	☐ Asbestos	☐ _____
Chemical Hazards			
☐ None	☐ Organic solvents	☐ Reactive metals	☐ PCBs
☑ Acids / bases	☐ Oxidizers	☐ Volatiles / Semi-volatiles	☐ _____

Job Hazard Analysis Form

Environmental Hazards			
☐ None	☑ Temperature extremes	☑ Wet location	☑ Bio hazards (snakes, insects, spiders, bird / mouse droppings, fungus, etc.)
☐ Explosive vapors	☐ Confined space	☐ Engulfment Hazard	☐ _____
Electrical Hazards			
☐ None	☑ Energized equipment or circuits	☐ Overhead utilities ☐ Underground utilities ☐ Hidden utilities	☐ Wet location
Fire Hazards			
☐ None	☐ Cutting, welding, or grinding generated sparks or heat sources	☑ Flammable materials present	☐ Oxygen enriched location
Ergonomic Hazards			
☑Lifting	☑ Bending	☑ Twisting	☐ Pulling/tugging
Computer Use in the: ☐ Office ☐ Field	☐ Repetitive motion	☐ _____	☐ _____
Radiological Hazards			
☑ None	☐ Loose contamination	☐ Fixed Contamination	☐ Radiation
☐ Airborne contamination	☐ Radon	☐ EMF	☐ Criticality
☐ Alpha	☐ Beta	☐ Gamma/X-rays	☐ Neutron
☐ Tritium	☐ TRU	☐ Depleted Uranium	☐ Enriched Uranium
Other Hazards			
☐			
☐			
☐			
☐			

Completed by: Gary Wise Date: 5/31/12

FORM ESH-2.9.1-3.3

Job Hazard Analysis Form

PPE AND MONITORING REQUIREMENTS

Standard PPE			
☐ Hard Hat	☑ Safety shoes	☑ Safety glasses	☐ Boot Covers
☐ Aprons	☐ Rubber Boots	☐ Other:_____	☐ Other:_____
Eye Protection			
☐ Welding glasses ☐ Welding helmet	☐ Face shield	☐ Chemical goggles	☐ Welding screens
Hearing Protection			
☐ Ear plugs	☐ Ear Muffs	☐ Ear plugs and muffs	☐ Other _____
Respiratory Protection			
☑ None	☐ Dust mask	☐ Full Face APR ☐ Half Face APR Cart. Type _____	☐ PAPR Cart. Type _____
☐ SCBA	☐ Airline respirator	☐ _____	☐ _____
Protective Clothing			
☐ Tyvek® coveralls	☐ Poly-coated Tyvek® Coveralls	☐ Saranex® Coveralls	☐ Fully encapsulating suit
☐ Cotton coveralls	☐ Modesty Clothing	☐ Fire resistant clothing	☐ Other _____
Hand Protection			
☐ None	☐ Cotton gloves	☐ Leather gloves	☐ Glove liners
☐ Nitrile gloves ☐ Viton® gloves ☐ Butyl gloves ☐ Neoprene gloves	Surgical gloves ☐ Latex ☐ Non-Latex	☐ Cut-resistant gloves	☑ Other: As appropriate for contaminants of concern
Monitoring Requirements			
☐ Oxygen	☐ Flammable gases/vapors	☐ Toxic Gas/vapors	☐ Hydrogen Sulfide/Carbon Monoxide
☐ Asbestos	☐ Full time IH coverage	☐ Part time IH coverage	☐ Be, Hg, Cr, Pb
☐ Metals Specify: _____			
☐ Organic vapors Specify: _____			
☐ Radioactive air particulates	☐ TLD required	☐ CAM	☐ Radon
☐ Full time RCT coverage	☐ Part time RCT coverage	☐ Radioactive air particulates	☐ Other _____
☐ Other _____		☐ Other _____	

PPE and monitoring requirements completed by: Gary Wise Date: 5/31/12

FORM ESH-2.9.1-3.4

Job Hazard Analysis Form

JHA Preparation Team

Gary Wise	Paul S. Johnstone	

Effective Date From: May 31, 2012 through May 30, 2013

Approval Signatures

		G Wise 5/31/12			
Job Supervisor	Date	LHSR	Date	RSO	Date
		Paul S. Johnstone 5/31/12			
ES&H Manager	Date	Project Manager	Date	Other	Date

Pre-Job Brief Attendance Sheet

Documents included in this briefing: _____

Date of Briefing: _____

Print/Type Name	Signature	Employee Number

Briefing presented by: _____ **Signature:** _____

Job Hazard Analysis Form

JHA No.: JHA - GRVL - 12 - 002 - 0

Job Title: Environmental Drilling/Boring and Soil Sampling **Date of Analysis:** 5/31/12

Job Location: Greenville, SC Projects **Team Leader:** Paul S. Johnstone

Key Work Steps	Hazards/Potential Hazards	Safe Practices
Going to site, work preparation	Mobilization/Demobilization	See Mobilization/Demobilization JHA
All Drilling/Boring Activities	Slips, Trips, Falls	See General Field Work JHA
	Heat/Cold Stress	See General Field Work JHA
	Biological Hazards: Insects, Snakes, Wildlife, Vegetation	See General Field Work JHA
	Traffic (including pedestrian)	▪ Notify attendant or site owner/manager of work activities and location ▪ Use cones, signs, flags or other traffic control devices as outlined in the ▪ Traffic Control Plan ▪ Set up exclusion zone surrounding work area using cones, signs, flags or other traffic control devices ▪ Wear appropriate PPE including high visibility clothing such as reflective vest ▪ Inspect area behind vehicle prior to backing and use spotter
	Fire/ Explosion	▪ Post No Smoking signs around work area ▪ Establish designated smoking area away from work area ▪ Ensure type ABC, 20-lb, fully charged fire extinguisher on-site and within inspection period

	Fire/ Explosion (continued)	▪ As site conditions/activities warrant, establish Hot Work Permit including air monitoring using direct-reading, real-time instruments such as LEL/ O2 meter ▪ Stop work if hazardous conditions (explosive atmosphere) are identified
Ambient Air Monitoring	Vapors	▪ Approach area where vapors are suspected from upwind direction and stay upwind/crosswind of from potential sources of vapors (use flagging or similar device to indicate wind direction)
	Effective Air Monitoring	▪ Ensure personnel using have been trained on instrument use ▪ Calibrate instrument prior to use
Concrete Coring	Ignition Sources	▪ Ensure electrical equipment properly grounded ▪ Apply water as necessary to address surface sparking potential
	High Noise Levels	▪ Hearing protection required when working around operating equipment if levels are suspected to be >85 dBA (if have to yell to person at a dist of 3 ft to be heard, likely exceeding 85 dBA).
	Airborne Particulates and Debris	▪ Use water as necessary to control dust in area ▪ Wear appropriate PPE including face shield or safety glasses with side shields, dust mask, leather gloves and long sleeves
	Sharp Rough Materials	▪ Wear appropriate PPE including leather gloves, long sleeves and pants, and steel-toed boots
	Impact to Subsurface Lines	▪ Ensure all underground features have been identified in area per SCP prior to start of activities
Drill Rig Set-Up	Contact with Electric Lines and Other Overhead Obstacles	▪ Position rig to avoid overhead utility lines by distance defined by voltage and local regulations ▪ Use a spotter when raising mast to confirm clearance of overhead lines and other obstructions
	Rig Movement	▪ Heavy equipment should be equipped with back-up alarm or use horn when backing - use spotter when available ▪ Stay clear of operating equipment and rig when moving

Drill Rig Set-Up (continued)	Heavy Equipment Lifting/ Carrying	▪ Use at least 2 people to lift and carry sections, use mechanical lift devices whenever possible, bend and lift with legs and arms, not back
	Sharp or Elevated Equipment	▪ Wear appropriate PPE including steel-toed safety boots, leather gloves and hard hat ▪ Establish communication system between workers involved in moving/attaching sections
Ground Disturbance: Auger/Boring Advancement	Faulty or Inappropriate Equipment	▪ Qualified driller must inspect drill rig prior to use, if faulty or inappropriate, do not proceed until repaired or replaced ▪ Inspect all hand tools prior to use, if faulty or inappropriate, do not proceed until repaired or replaced. Tag out all defective tools
	Moving Equipment	▪ Clear area of obstructions and communicate with all workers involved that drilling is beginning ▪ Do not exceed manufacturer's recommended speed, force, torque, or other specifications. and penetrate the ground slowly with hands on the controls for at least the first foot of soil to minimize chance of auger kick-out ▪ Stay clear of rotating auger ▪ Use long-handled shovel to clear away cuttings when auger has stopped ▪ Do not wear loose clothing ▪ Wear appropriate PPE including leather gloves and steel-toed boots
	Suspended Loads	▪ Do not walk under suspended loads ▪ When possible, remove overhead hazards promptly ▪ Wear appropriate PPE including hard hat and steel-toed boots (See HASP)
	High Noise Levels	▪ Use hearing protection if within 20 feet of active drill rig
	Ground Disturbance: Auger/Boring Advancement Vapors and Airborne	▪ Monitor air concentrations using direct-reading, real-time instruments such as OVM and Dräger tubes (See HASP for required monitoring instruments and action limits)

	Particulates	▪ Stop work if hazardous conditions (explosive atmosphere, O2 deficient atmosphere) identified until precautions are taken ▪ Wear appropriate PPE including face shield or safety glasses with side shields, dust masks or respirators, long sleeves and pants ▪ Stay upwind (use flagging or similar device to indicate wind direction)
	Impact to Subsurface Lines/Tanks	▪ Only drill in areas where underground features have been identified and cleared per Subsurface Clearance Protocol (SCP) if hole has to be moved, clear new location first ▪ Wear appropriate PPE including insulating gloves or stand on an insulating mat when in contact with drill rig ▪ Ensure first aid responders are trained to deal with electric shock and flash burns
Ground Intrusion: Split Spoon	Faulty Equipment	▪ Inspect rope/cable/rod for wear, fraying, oils and moisture prior to use, do not use if faulty until repaired or replaced. ▪ Inspect cathead for rust and rope grooves prior to use, do not use if faulty until repaired or replaced ▪ Report any defects to your supervisor
	Moving Equipment	▪ Do not wrap rope around any part of the hand or body ▪ Maintain distance of at least 18-inches from in-running points on running/reciprocating equipment ▪ Eliminate excess rope ▪ Do not wear loose clothing ▪ Wear appropriate PPE including leather gloves
Soil Sampling	Contaminated Materials	▪ Wear appropriate PPE including Nitrile gloves
	Sharp Sampling Tools	▪ Use correct tools for opening sleeves ▪ When opening sleeve, cut away from body ▪ Place soil core on sturdy surface prior to cutting

Soil Sampling (continued)	Vapors	▪ Wear appropriate PPE including respirator if conditions warrant, or ▪ Move personnel to a location upwind or farther away from source of vapors
	Sample Cross Contamination	▪ Decontaminate or dispose of sampling equipment between sampling locations ▪ Double-check sample labels to ensure accuracy and adhesion to containers
Ground Disturbance: Hand-Auger/Boring and Monitoring Well Installation	Faulty or Inappropriate Equipment	▪ Inspect all hand tools prior to use, if faulty or inappropriate, do not proceed until repaired or replaced. Tag out all defective tools
	Impact to Subsurface Lines	▪ Ensure all underground features have been identified in area per SCP prior to start of activities
	Heavy Material Lifting/ Carrying	▪ Use at least 2 people to lift and carry bags weighing more than 50 pounds, use mechanical lift devices and transport aids (carts, dollies, etc.) whenever possible, bend and lift with legs and arms, not back
	Sharp or Rough Materials or Equipment	▪ Wear appropriate PPE including work boots and leather gloves
	Heat or Cold Stress	▪ See General Field Work JHA
Well Development	Electrical Shock or Burns	▪ Use care attaching pump leads to truck battery. Wear gloves and remove rings.
	Back and Shoulder Strain	▪ Stand straight and perform all pump surging with hands in the "strike zone" (between shoulders and knees). ▪ Change hand position frequently to reduce muscle and joint fatigue.
	Contaminated Water and Sediment	▪ Use appropriate PPE, including nitrile or other gloves. ▪ Manage waste liquids and solids per the approved work plan.
	Heat or Cold Stress	▪ See General Field Work JHA

Solid/Liquid Waste Management/ Disposal	Vapors and Airborne Particulates	▪ Monitor air concentrations using direct-reading, real-time instruments such as OVM and Dräger tubes ▪ Stop work if hazardous conditions (explosive atmosphere, O2 deficient atmosphere) identified until precautions are taken ▪ Wear appropriate PPE including safety glasses with side shields, dust masks and respirators ▪ Stay upwind (use flagging or similar device to indicate wind direction)
	Contaminated Materials and Container Pinch Points	▪ Wear appropriate PPE including Nitrile and leather gloves ▪ Position hands/fingers to avoid pinching/smashing/crushing when closing drum rings
	Heavy Materials and Containers Lifting/ Moving	▪ Do not lift or move heavy containers without assistance ▪ Use proper bending/lifting techniques by lifting with arms and legs and not with back ▪ If possible, use powered lift truck, drum cart, or other mechanical means Take breaks if feeling faint or overexerted ▪ Spot drums in storage area prior to filling ▪ Wear appropriate PPE including leather gloves and steel-toed boots

Form ESH-2.9.1-3.1

Job Hazard Analysis Form

Identify Hazards and PPE

Complete the checklists for hazard identification and PPE requirements. Information from the RA and applicable permits are included in this section.

Standard Hazards			
☑ Falling Objects	☑ Slips and trips	☑ Pinch points	☑ Rotating equipment
☑ Falls	☑ Power equipment/tools	☐ Elevated work surfaces	☐ _____
Eye Hazards			
☐ Particulates	☐ Liquid splashes	☐ Welding Arc	☐ _____
Hearing Hazards			
☐ None	☑ Impact noise	☐ High frequency noise	☑ High ambient noise
Respiratory Hazards			
☐ None	☑ Dust/particulates	☑ Organic Vapors	☐ Acid Gases
☐ Oxygen deficient	☐ Welding fumes	☐ Aerosols/Particulates	☐ Be, Hg, Cr, Pb
☐ _____	☐ Radon	☐ Asbestos	☐ _____
Chemical Hazards			
☐ None	☐ Organic solvents	☐ Reactive metals	☐ PCBs
☐ Acids / bases	☐ Oxidizers	☑ Volatiles / Semi-volatiles	☐ _____
Environmental Hazards			
☐ None	☑ Temperature extremes	☑ Wet location	☑ Bio hazards (snakes, insects, spiders, bird / mouse droppings, fungus, etc.)
☐ Explosive vapors	☐ Confined space	☐ Engulfment Hazard	☐ _____
Electrical Hazards			
☐ None	☐ Energized equipment or circuits	☑ Overhead utilities ☑ Underground utilities ☑ Hidden utilities	☐ Wet location
Fire Hazards			
☐ None	☐ Cutting, welding, or grinding generated sparks or heat sources	☑ Flammable materials present	☐ Oxygen enriched location

Form ESH-2.9.1-3.2

Job Hazard Analysis Form

Ergonomic Hazards			
☑ Lifting	☑ Bending	☑ Twisting	☑ Pulling/tugging
Computer Use in the: ☐ Office ☐ Field	☐ Repetitive motion	☐ _____	☐ _____
Radiological Hazards			
☑ None	☐ Loose contamination	☐ Fixed Contamination	☐ Radiation
☐ Airborne contamination	☐ Radon	☐ EMF	☐ Criticality
☐ Alpha	☐ Beta	☐ Gamma/X-rays	☐ Neutron
☐ Tritium	☐ TRU	☐ Depleted Uranium	☐ Enriched Uranium
Other Hazards			
☐			
☐			
☐			
☐			

Completed by: Gary Wise Date: 5/31/12

Form ESH-2.9.1-3.3

Job Hazard Analysis Form

PPE and Monitoring Requirements

Standard PPE			
☑ Hard Hat	☑ Safety shoes	☑ Safety glasses	☐ Boot Covers
☐ Aprons	☐ Rubber Boots	☐ Other:_____	☐ Other:_____
Eye Protection			
☐ Welding glasses ☐ Welding helmet	☐ Face shield	☐ Chemical goggles	☐ Welding screens
Hearing Protection			
☑ Ear plugs	☐ Ear Muffs	☐ Ear plugs and muffs	☐ Other _____
Respiratory Protection			
☐ None	☐ Dust mask	☐ Full Face APR ☐ Half Face APR Cart. Type _____	☐ PAPR Cart. Type _____
☐ SCBA	☐ Airline respirator	☑ As needed	☐ _____
Protective Clothing			
☐ Tyvek® coveralls	☐ Poly-coated Tyvek® Coveralls	☐ Saranex® Coveralls	☐ Fully encapsulating suit
☐ Cotton coveralls	☐ Modesty Clothing	☐ Fire resistant clothing	☐ Other _____
Hand Protection			
☐ None	☐ Cotton gloves	☐ Leather gloves	☐ Glove liners
☐ Nitrile gloves ☐ Viton® gloves ☐ Butyl gloves ☐ Neoprene gloves	Surgical gloves ☐ Latex ☐ Non-Latex	☐ Cut-resistant gloves	☑ Other As appropriate for contaminants of concern
Monitoring Requirements			
☐ Oxygen	☑ Flammable gases/vapors	☑ Toxic Gas/vapors	☐ Hydrogen Sulfide/Carbon Monoxide
☐ Asbestos	☐ Full time IH coverage	☐ Part time IH coverage	☐ Be, Hg, Cr, Pb
☐ Metals Specify: _____			
☐ Organic vapors Specify: _____			
☐ Radioactive air particulates	☐ TLD required	☐ CAM	☐ Radon
☐ Full time RCT coverage	☐ Part time RCT coverage	☐ Radioactive air particulates	☐ Other _____
☐ Other _____		☐ Other _____	

PPE and monitoring requirements completed by: Gary Wise Date: 5/31/12

Form ESH-2.9.1-3.4

Job Hazard Analysis Form

JHA Preparation Team

Gary Wise Paul S. Johnstone

Effective Date From: May 31, 2012 through May 30, 2013

Approval Signatures

Job Supervisor	Date	LHSR	5/31/12 Date	RSO	Date
ES&H Manager	Date	Project Manager	5/31/12 Date	Other	Date

Pre-Job Brief Attendance Sheet

Documents included in this briefing: _____

Date of Briefing: _____

Print/Type Name	Signature	Employee Number

Briefing presented by: _____ **Signature:** _____