Prepared for:
City of Fulton and Oswego County
Fulton, New York






Site-specific Sampling, Analysis, and Monitoring Plan

ERP Site ID #E7-38-039

308 Harrison Street, City of Fulton, New York

ENSR Corporation
March 2008
Document No.: 10683-008

ENSR | AECOM

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1.0 Project organization and responsibility

1.1 Project organization and responsibilities

- USEPA Review and Oversight — Jennifer Tsolisos
- NYSDEC Review and Oversight — Christopher F. Mannes, III PE
- NYSDOH Review and Oversight — Katie Comerford
- City of Fulton Review and Oversight — Ron Edick
- Oswego County Review and Oversight — Karen Noyes
- Overall Project Coordination — Luke P. McKenney
- Overall QA — Luke P. McKenney
- Systems Auditing — Luke P. McKenney
- Performance Auditing — Luke P. McKenney
- Sampling Operations — Denise Sero
- Sampling QC — Ray Smith
- Laboratory Analyses — TestAmerica
- Laboratory QC — Ray Smith
- Data Processing Activities — Denise Sero
- Data Processing QC — Denise Sero
- Data Quality Review — Waverly Braunstein

1.2 Personnel information

Name	Address	Responsibilities
Jenny Tsolisos, USEPA Brownfields Program Manager	290 Broadway, 18th Floor New York, NY 10007 Ph: (212) 637-4349	Ms. Tsolisos will represent the USEPA in its review and oversight function, in their financial sponsorship and arbiter on technical matters
Christopher F. Mannes, III PE, NYSDEC Project Manager	615 Erie Blvd. West Syracuse, NY 13204 Ph: (315) 426-7515	Mr. Mannes will represent the NYSDEC in its review and oversight function, in its financial sponsorship, and as arbiter on technical matters
Katie Comerford, Public Health Specialist NYSDOH Project Manager	217 S. Salina Street Syracuse, New York 13202 Ph: 315-477-8566	Ms. Comerford will represent NYSDOH in its review and oversight function, in its financial sponsorship, and as arbiter on technical matters

Name	Address	Responsibilities
Ronald C. Edick, City Engineer	Municipal Building 141 South First Street Fulton, New York 13069-1717 Ph: (315) 592-3454	Mr. Edick will represent the City in the review and oversight of the project, participate in citizen participation activities, and serve as the point of contact for the City
Karen Noyes, Oswego County Senior Planner	46 East Bridge Street Oswego, NY 13126 Ph: (315) 349-8295	Ms. Noyes will represent the City in the review and oversight of the project, participate in citizen participation activities, and serve as the point of contact for the County
Luke McKenney, ENSR Corporation, ISC Section Manager	5015 Campuswood Drive, Suite 104 East Syracuse, NY 13057 Ph. (315) 432-0506	Mr. McKenney will oversee the project, provide quality control on documents and determinations and mentor the daily task manager.
Ray Smith, ENSR Corporation, ISC Geologist	5015 Campuswood Drive, Suite 104 East Syracuse, New York 13057 Ph. (315) 432-0506	Mr. Smith will review contractor and subcontractor compliance with the SAMP.
Denise Sero, ENSR Corporation, ISC Geologist	5015 Campuswood Drive, Suite 104 East Syracuse, New York 13057 Ph. (315) 432-0506	Ms. Sero will supervise field investigation activities and will also serve as database manager.
Kathleen Harvey, ENSR Corporation, Regional Health and Safety Manager	2 Technology Park Drive Westford, MA 01886 Ph. (978) 589-3000 kharvey@ensr.aecom.com	Ms. Harvey will prepare the site-specific Health and Safety Plan and serve as the Health and Safety point of contact for ENSR staff.
Waverly Braunstein, ENSR Corporation, Senior Project Chemist	2 Technology Park Drive Westford, MA 01886 Ph. (978) 589-3000	Ms. Braunstein will act as Quality Assurance Officer (QAO) and will conduct data validation activities.

1.3 Laboratory information

Name	Contact and telephone number	Sample analyses
TestAmerica	Richard Lafond W: (315) 431-0171 F: (315) 431-0151 Richard.Lafond@testamericainc.com	Soil and groundwater samples: TCL VOCs + MTBE via USEPA Method 8260B, TCL SVOCs via USEPA Method 8270C, TAL metals and cyanide via USEPA Methods 6010, 6020, 7470, and 9012A, and PCBs via USEPA Method 8082. Soil vapor samples: VOCs via USEPA Method TO-15.

2.0 Project definition

2.1 Site background

This Site-specific Brownfields Sampling, Analysis, and Monitoring Plan (SAMP) for the project site, located at 308 Harrison Street (Site ID#E7-38-039) in the City of Fulton, County of Oswego, New York, contains a Historical Data Review and Site Reconnaissance reports. These reports contain extractions from the Phase I Environmental Site Assessment conducted on the property on behalf of Oswego County, as part of its USEPA Brownfield Program.

As part of Oswego County's USEPA Brownfield Assessment Program, ENSR conducted a Phase I Environmental Site Assessment (ESA) in March 2005, in an effort to develop an initial understanding of the environmental conditions associated with the property. The Phase I ESA was conducted in accordance with the scope and limitations of the ASTM Standard Practice E 1527-00 for ESAs and USEPA's Proposed All Appropriate Inquiries (AAI), as described in 40 CFR Part 312. A summary of ENSR's historical review and site reconnaissance is included below. The Phase I ESA was previously provided to the NYSDEC and USEPA and is available upon request.

2.2 Historical data review report

The project site is located at 308 Harrison Street (ERP Site #E7-38-039) in the City of Fulton, Oswego County, New York. A site location map has been included as Figure 1. The vacant site is approximately 2.2 acres in size, and consists of a one-story corrugated-metal building (approximately 1,500 square feet). The remainder of the site consists of slightly overgrown grassy areas. Several piles of construction materials and some surface debris remain on the property. The site is located in a commercial area near a residential neighborhood, and is currently vacant.

The subject property was purchased by the City of Fulton Community Development Agency in July 2003. In March 2005, ENSR conducted a Phase I Environmental Site Assessment (ESA) of the property on behalf of Oswego County, as part of their USEPA Brownfield Assessment Program, in an effort to develop an initial understanding of the environmental conditions associated with the property. The Phase I ESA was conducted in accordance with the scope and limitations of the ASTM Standard Practice E for ESAs and USEPA's Proposed All Appropriate Inquiries (AAI), as described in 40 CFR Part 312. The Phase I ESA included a review of available historical and environmental documentation, an on-site inspection of the property, and a sidewalk inspection of neighboring properties. The Phase I ESA was provided to the NYSDEC in the City's ERP Grant Application.

The Phase I ESA revealed that the property was used as an illegal solid waste dump in 1916 and for manufacturing operations (i.e. paper bags and packing pads) from 1907 through 1955. Information pertaining to the type of materials processed, handled and/or stored during that time is unknown. C&S Companies, Inc. (C&S) conducted a Phase I ESA in October 2002 and a limited Phase II ESA in August 2003. Ash and fill materials were discovered in eleven test trenches installed during the Phase II ESA. A geophysical survey, also conducted as part of C&S's investigation, identified the presence of a 1000-gallon underground storage tank (UST) west of the corrugated metal building.

Historical documents reviewed by ENSR during the Phase I ESA included Sanborn Maps, aerial photographs, city directories and newspaper articles. Based on this historical review, three buildings existed on the property. The main building (Building 1) was used for manufacturing operations from 1907 until approximately 1955. From the mid-1960s through the mid-1970s, the main building was used as a moving and storage facility. The main building later served as the Fulton Mall from 1984 until the early 1990's and was demolished in 2004.

The existing corrugated metal building (Building 2) appeared in an aerial photograph taken in 1955 and was used for storage. The third building (Building 3) was listed in Sanborn Maps dated 1911 and 1924 as the "Former Fulton Toilet Paper Company" and "L.W. Emerick Warehouse and Machinery," respectively. This building was not evident in an aerial photograph dated 1939. The former land use of adjacent properties was identified as paper mills, which operated from the 1850s through the 1960s. ENSR identified this historic land use in the Phase I ESA as an environmental concern.

ENSR's historical review included a search of historic newspapers. An article from the Fulton Times Newspaper in 1916 indicated that the site had been used as an illegal municipal solid waste dumping site. The dumping site covered a space equivalent to three city lots to a depth of over four feet. The article states that solid waste was disposed of at the site for over one year by garbage collectors until health officers threatened arrest and fines. The Board of Health was considering a clean-up plan to be completed and paid for by the guilty garbage collectors, but no subsequent information could be found to determine if the clean up was ever completed. ENSR identified this illegal dump in the Phase I ESA as an environmental concern.

ENSR also reviewed historical environmental reports, including C&S's Phase I Environmental Site Assessment and a Limited Phase II Environmental Assessment for the Site dated October 22, 2002 and August 27, 2003, respectively. Based on the findings of the 2002 Phase I ESA, C&S Engineers, Inc. completed a limited Phase II investigation in August 2003. A geophysical survey was conducted which confirmed the presence of a 1,000-gallon UST located at the northwest corner of the building. A trench was excavated to a depth of approximately four feet parallel to the tank. Photoionization detector (PID) readings of the soil surrounding the tank and visual observations did not indicate petroleum contamination in the tank area. Eleven test trenches were also advanced in areas associated with debris piles. Areas of non-native fill materials, including suspected coal ash and cinders, were observed beneath the root zone. Reportedly, no soil or groundwater samples were collected for laboratory analysis. C&S recommendations included the removal of the UST and that further subsurface investigation be conducted to characterize and delineate the extent of the suspected coal ash or cinder material.

2.3 Site reconnaissance report

2.3.1 Land areas

The vacant site is approximately 2.2 acres in size, and consists of a one-story corrugated-metal building (approximately 1,500 square feet). The remainder of the property consists of slightly overgrown grassy areas. During ENSR's Phase I site inspection, ENSR observed discarded equipment, tires, and construction debris along the south wall of the building. In addition, a pile of miscellaneous debris including appliances, mattresses, tires, and some surface debris were observed elsewhere on the property. Several areas of puddling and ponding of water were observed on the northern portion of the property. ENSR also observed a one-inch vent pipe located on the west wall of the metal building. Based on C&S's previous investigations, it is ENSR's understanding that this vent pipe is connected to the 1,000-gallon UST.

2.3.2 Buildings

The one-story building is constructed of a wood frame with corrugated metal on a concrete slab foundation and is approximately 1,500 square feet in size. The main entrance consists of two bay doors on the north side of the building. The interior space includes an open bay area which, at the time of the Phase I ESA, was used to store obsolete equipment, building materials, and tires. During the Phase I site inspection, ENSR also observed several large piles of construction materials (wood, plastic, vinyl etc), surface debris, and several smaller piles of construction materials in the building. No hazardous substances were noted in the building at the time of the inspection; however due to the amount of construction and debris piles, ENSR was not able to thoroughly evaluate the interior areas of the metal building.

2.3.3 Utilities

The site is serviced by municipal water and sewer. Based on information gathered during ENSR's Phase I ESA, the site was connected to the municipal sewer in 1930. During the Phase I ESA site inspection, no floor drains or sumps were observed in the building; however, due to the piles of construction materials, the inspection of the floor area was limited.

National Grid Company provided the subject property with electricity and natural gas for power and heating purposes. Telephone service is provided by Alltel, Inc of New York. Two utility poles are located along the eastern edge of the property. No transformers were observed during the ENSR's Phase I site inspection.

2.3.4 Physical characteristics

Topography on-site is relatively flat with a gentle slope to the north-northeast. Vegetation observed on-site consisted of bushes, grass and shrubs located in the east, west and southern portions of the property. None of the vegetation appeared to be environmentally stressed during the Phase I site inspection. In addition, no stained soil was observed during the inspection.

Based on topographic information and field observations, groundwater is estimated to be at a depth of 1 to 4 feet below surface grade. Local groundwater flow beneath the site is inferred to be in a northwesterly to northeasterly direction, either toward Oswego Canal or towards Waterhouse Creek, which is located approximately 650 feet northeast of the subject site. Surface water flow is towards Waterhouse Creek.

According to the US Department of Agriculture – Soil Conservation Service's Publication *Soil Survey of Oswego County*, the subject property is located upon Amboy-Williamson subsurface soils. These soils are well drained, and have a high content of silt and very find sand. Site soils are categorized as urban land. The local bedrock formation is Lower Silurian (Medina Group and Queenston Formation).

According to the Flood Insurance Rate Map for the City of Fulton (Community Panel Number 360649 0002 B), the subject site lies within Zone AO; an area of 100-year shallow flooding. A wetlands delineation was not performed by a wetlands specialist as part of ENSR's Phase I ESA.

2.3.5 Surrounding properties

The subject site is located at the southwest corner of the intersection of Harrison Street and North Fourth Street in a mixed commercial/residential area. The site is bounded to the north by Harrison Street, beyond which are Mimi's Storage (a commercial storage building) and Oswego Falls Body and Paint. To the northeast, the site is abutted by Harrison Street, beyond which is Haun Welding and Equipment. The site is bordered to the south-southwest by Alltel Service, Inc. beyond which is Ontario Street. To the southeast is vacant land. The site is abutted to the east by North Fourth Street, beyond which is the City of Fulton Municipal Garage property. To the west, the site is bordered by Third Street, beyond which is Galizia's All-Safe Storage. An aerial photograph of the site and neighboring properties has been provided in Figure 3.

2.4 Project definition

The objective of the Remedial Investigation (RI) for the site is to characterize the nature and extent of contamination of soils at the site surrounding the UST and the former illegal municipal solid waste dump, and to determine whether the UST, former dump may have impacted groundwater at the site. The former land use of upgradient and adjacent properties as paper mills for the 1850s through the 1960s is an additional environmental concern at this site. The Remedial Investigation Work Plan describes in detail the specific tasks to be completed at the site. A brief summary is provided below.

To characterize the vertical and horizontal extent of impacts associated with the UST and to confirm or deny the presence of other contaminants of concern (COCs) related to previous site usage, ENSR will advance 15 soil borings and collect one soil sample from each of five test pits. To determine the presence of groundwater impacts resulting from the former UST and/or from previous site usage, four groundwater monitoring wells will be installed on the subject property. Additionally, six soil vapor probes will be installed around the perimeter of the site, and one soil vapor probe will be installed inside the current site building to evaluate COCs in soil vapor. Soil and groundwater samples will be submitted to an off-site NYSDOH ELAP certified laboratory for analysis of target compound list (TCL) volatile organic compounds (VOCs) and Methyl-Tert-Butyl-Ether (MTBE) via USEPA Method 8260B, TCL semi-volatile organic compounds (SVOCs) via USEPA Method 8270C, target analyte list (TAL) metals and cyanide via the appropriate USEPA Methods 6010, 6020, 7470/7471, and 9012A, and polychlorinated biphenyls (PCBs) via USEPA Method 8082. Soil vapor samples will be analyzed for VOCs by USEPA Method TO-15.

2.4.1 Scope of work

The detailed scope of work at the site is described in the project Work Plan. Samples will be collected from soil borings and monitoring wells. These samples will be analyzed using the USEPA SW-846 "Test Methods for Evaluating Solid Waste," November 1986, 3rd edition (and subsequent updates) and by USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.

In addition to the remedial investigation activities, an interim remedial measure will be conducted to remove the 1000 gallon UST, which is a potential source of contamination.

3.0 Project description

3.1 Data use objectives

The following items are the objectives of this Site-specific SAMP for the project site, located at 308 Harrison Street, (Site ID#E7-38-039) in the City of Fulton, County of Oswego, New York.

- Delineate horizontal and vertical contaminant concentrations, identify clean areas.
- Locate and identify sources of contamination.
- Ascertain if there is a risk to human health or the environment, including fish and wildlife resources.
- Determine if additional remediation is required.

3.2 Brownfield site investigation reports

Upon the completion of the Brownfields Remedial Investigation sampling project, the Remedial Investigation Report (RIR) will be developed. The RIR will include one or more of the following recommendations to summarize the environmental condition of the property:

- Additional sampling is required.
- Undertake remediation.
- No additional actions are required.

The Remedial Investigation Report will base any of the aforementioned recommendations on the data collected during the Remedial Investigation, and on other data or facts that have been collected on the subject property.

3.3 Quality of data needed for environmental data measuring

The sampling results must be of sufficient quality to ensure that the sampling results accurately characterize site conditions, as these sampling results may be used to make important and potentially costly decisions concerning the re-development of Brownfields sites. To ensure an accurate characterization of environmental conditions for the subject property, this Site-specific Brownfields SAMP is based on the following procedures:

- Logical evaluation of available site information.
- Selection of an appropriate sampling design.
- Selection and utilization of suitable analytical field screening and sampling techniques.
- Proper sample collection, preservation, and transportation techniques are planned.
- Collection and analysis of appropriate quality assurance/quality control (QA/QC) samples.
- Planning of proper fixed laboratory analyses.
- Logical presentation and interpretation of analytical data.
- Definition of data usability criteria.

3.4 Project description

A more detailed description of the work to be performed at the site is included in the Remedial Investigation Work Plan. To characterize the vertical and horizontal extent of impacts associated with the UST and to confirm or deny the presence of other contaminants of concern (COCs) related to previous site usage, ENSR will advance fifteen soil borings and collect one soil sample from each of five test pits. To determine the presence of groundwater impacts resulting from the former UST and/or from previous site usage, four groundwater monitoring wells will be installed on the subject property. Additionally, six soil vapor probes will be installed around the perimeter of the site and one soil vapor probe will be installed inside the current site building to evaluate COCs in soil vapor. Soil and groundwater samples will be submitted to an off-site NYSDOH ELAP certified laboratory for analysis of TCL VOCs via USEPA Method 8260B, MTBE, TCL SVOCs via USEPA Method 8270C, TAL metals and cyanide via the appropriate USEPA Methods 6010, 6020, 7470/7471, and 9012A, and PCBs via USEPA Method 8082. Soil vapor samples will be analyzed for VOCs by USEPA Method TO-15.

Soil samples will be screened in the field using a photoionization detector (PID) to detect the presence of volatile organic compounds (VOCs). Subsurface soil samples will be collected for analysis using disposable plastic trowels from an interval, at the discretion of the ENSR geologist/engineer, dependent on observations during sampling (odors, discoloration, etc) or elevated photoionization detector (PID) field screening results.

Soil vapor sampling points will be tested to ensure that no outside air is infiltrating the sample by utilizing a tracer gas (helium). The area where the sampling point intersects the ground surface will be enclosed in a sealed vessel, and the atmosphere surrounding the sampling point will be enriched with helium gas. A vapor sample from the probe will be analyzed with a portable monitoring device before and after sampling for the compounds of concern.

Groundwater samples will be collected in accordance with EPA Region 2 Low Flow Sampling SOP; therefore, during sampling, groundwater quality parameters (pH, temperature, conductivity, oxidation-reduction potential, turbidity and dissolved oxygen) will be recorded periodically using a water-quality meter with a flow-through cell (such as a Horiba U-22 meter). The well is considered stabilized and ready for sample collection when the indicator parameters have stabilized for three consecutive readings (±0.1 SU for pH, ±3% for specific conductivity, ±10 mv for oxidation-reduction potential, and ±10% for turbidity and dissolved oxygen).

3.5 Project time line

The following schedule has been developed assuming Agencies' approval of the IRM and Remedial Investigation Work Plan is received by April 1, 2008. ENSR has allotted for a 45-day Agency review period of the Remedial Investigation and Remedial Alternatives Report.

Table 3-1 Project schedule

Task	Start Date	Completion Date	Notes
Mail a fact sheet to the Mailing List and announce through local news media the availability of the Work Plan at local repositories.	April 15, 2008	April 15, 2008	To commence within two weeks of receipt of USEPA and NYSDEC approval of Work Plans
Conduct Remedial Investigation and IRM	April 29, 2008	May 27, 2008	To commence within four weeks of receipt of USEPA and NYSDEC approval of Work Plans. Estimated up to four weeks in the field.
Prepare and submit draft Remedial Investigation Report including IRM Report	May 28, 2008	July 9, 2008	To be submitted within six weeks following the completion of field activities
Prepare and submit draft Remedial Alternatives Report	July 10, 2008	August 6, 2008	To be submitted within four weeks of submittal of Draft Remedial Investigation Report
Prepare and submit final Remedial Investigation and Remedial Alternatives Report	September 22, 2008	October 6, 2008	Assumed 45-day USEPA and NYSDEC review period. Final RI/RAR to be submitted within two weeks of receipt of USEPA and NYSDEC comments

4.0 Sampling design

4.1 Sampling and analysis

The purpose of performing a Brownfields site investigation is to determine the presence and identity of contaminants, as well as the extent to which they have become integrated into the surrounding environment. The objective of this effort will be to collect and analyze environmental samples that are representative of the media under investigation. The methods and equipment used for collecting environmental matrices of concern will vary with the associated physical and chemical properties of each media designated for sampling.

To ensure sampling and analytical protocols are appropriate, it is necessary to describe the objectives and details comprising these activities. As a result, the design of a proper sampling scheme, including protocols for collecting rinse blanks, trip blanks, duplicates, and background samples should be derived from an accepted guidance. As such, the *USEPA Superfund Program Representative Sampling Guidances, Volume 1: Soil; Volume 5: Water and Sediment, Part II - Ground Water*, and the New York State Department of Health's *Guidance for Evaluating Soil Vapor Intrusion in the State of New York* are included as attachments to this Site-specific Brownfields SAMP. These media specific guides are formal sampling guidance that outline protocols for the collection of representative samples to ensure the accurate characterization of site conditions. Therefore, following these guides will assist in the design of a fitting sampling network that is thoroughly justified and documented in this Site-specific Brownfields SAMP.

4.2 Sampling design

The sampling program will provide data concerning the presence and the nature and extent of contamination of groundwater, soil and soil vapor, if any. Sample locations, analytical parameters, and collection rationale are provided in Table 4-1. One soil field duplicate sample, one groundwater field duplicate sample, and one soil gas field duplicate will be collected. One equipment blank sample will be collected for each type of sampling equipment used, except soil gas, as no sampling equipment is applicable to soil gas sample collection. A trip blank will accompany each shipment of aqueous samples analyzed for VOCs.

Table 4-1 Summary of samples and analysis

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
SB-1	Soil	Highest PID or other location as determined by ENSR field geologist	Northwest corner of property	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Northwest adjacent properties historically used as paper mills, northern adjacent properties used as chemical and oil storage.	COCs associated with paper manufacturing, such as mercury and other metals, PCBs, VOCs, and PAHs.
SB-2	Soil	Highest PID or other location as determined by ENSR field geologist	North perimeter of property, adjacent to Harrison Street	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Properties located to the north across Harrison Street were historically used as undefined chemical and oil storage areas, and an automotive paint and repair shop is located across Harrison Street. In addition, SB-2 is in the area of the former illegal dumping activities.	COCs associated with oil storage include VOCs and SVOCs. COCs associated with automotive operations include chlorinated VOCs, VOCs, and metals. COCs associated with former dumping activities are unknown.
SB-3	Soil	Highest PID or other location as determined by ENSR field geologist	Northeast corner of property.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Properties located to the northeast include an automotive repair and paint shop, and a property referred to as a "municipal garage," but it is not known whether the City uses the property for vehicle storage only or for maintenance. In addition, SB-3 is located in the area of the former illegal dumping activities.	COCs associated with automotive operations include chlorinated VOCs, SVOCs, and metals. COCs associated with the possible vehicle maintenance include chlorinated VOCs, BTEX, SVOCs, and metals. COCs associated with former dumping activities are unknown.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
SB-4	Soil	Highest PID or other location as determined by ENSR field geologist	West perimeter of the property, adjacent to Third Street	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historical use of the adjacent properties to the west as paper mills.	COCs associated with paper manufacturing, such as mercury and other metals, PCBs, VOCs, and PAHs.
SB-5	Soil	Highest PID or other location as determined by ENSR field geologist	Southwest corner of property	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Current and historic uses of properties to the south and southwest do not represent a REC; SB-5 is placed to minimize data gaps and provide a thorough understanding of the subsurface conditions throughout the entire site, including historic site usage as an illegal solid waste dump.	Unknown; COCs may relate to any upgradient sources and previous site use as an illegal dump.
SB-6	Soil	Highest PID or other location as determined by ENSR field geologist	Southern perimeter of property.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Current and historic uses of properties to the south do not represent a REC; SB-6 is placed to minimize data gaps and provide a thorough understanding of the subsurface conditions throughout the entire site, including historic site usage as an illegal solid waste dump.	Unknown; COCs may relate to any upgradient sources and previous site use as an illegal dump.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
SB-7	Soil	Highest PID or other location as determined by ENSR field geologist	Southern perimeter of property.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Current and historic uses of properties to the south do not represent a REC; SB-6 is placed to minimize data gaps and provide a thorough understanding of the subsurface conditions throughout the entire site, including historic site usage as an illegal solid waste dump.	Unknown; COCs may relate to any upgradient sources and previous site use as an illegal dump.
SB-8	Soil	Highest PID or other location as determined by ENSR field geologist	Southeast corner of property	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of impacts relating to historic site use generally, and specifically with the former Building 3. Historic uses of the building include a toilet paper company, a machinery shop, and a warehouse.	COCs associated with the machinery operations include VOCs, SVOCs, and metals. If toilet paper manufacturing was carried out, COCs typical of paper manufacturing may be present.
SB-9	Soil	Highest PID or other location as determined by ENSR field geologist	Northwestern portion of property in former location of Building 1.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historic use of the former Building 1 included as a paper bag manufacturer, a packing pad manufacturer, and moving/trucking operations. In more recent times, the building was the Fulton Mall, which housed a variety of retail operations.	COCs associated with historic use of Building 1 include VOCs, SVOCs, metals, and PCBs.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
SB-10	Soil	Highest PID or other location as determined by ENSR field geologist	Northwestern portion of property in former location of Building 1.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historic use of former Building 1 included as a paper bag manufacturer, a packing pad manufacturer, and moving/trucking operations. In more recent times, the building was the Fulton Mall, which housed a variety of retail operations.	COCs associated with historic use of Building 1 include VOCs, SVOCs, metals, and PCBs.
SB-11	Soil	Highest PID or other location as determined by ENSR field geologist	Central portion of the property, inside existing building	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	The historic use of the existing building is uncertain. SB-11 is placed to gain a general understanding of any environmental conditions associated with Building 2.	Unknown; COCs may relate to any upgradient sources, historic uses of Building 2, and previous site use as an illegal dump.
SB-12	Soil	Highest PID or other location as determined by ENSR field geologist	Central portion of the property, inside existing building	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	The historic use of the existing building is uncertain. SB-11 is placed to gain a general understanding of any environmental conditions associated with the building.	Unknown; COCs may relate to any upgradient sources, historic uses of Building 2, and previous site use as an illegal dump.
SB-13	Soil	Highest PID or other location as determined by ENSR field geologist	East-central portion of the property.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historic use of the site as an illegal solid waste dump.	COCs associated with historic illegal dumping activities are unknown.
SB-14	Soil	Highest PID or other location as determined by ENSR field geologist	Eastern portion of the site, in former location of Building 3	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of impacts relating to historic site use generally, and specifically with the former Building 3. Historic uses of the building include a toilet paper company, a machinery shop, and a warehouse.	COCs associated with the machinery operations may include VOCs, SVOCs, and metals. If toilet paper manufacturing was carried out, COCs typical of paper manufacturing may be present.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
SB-15	Soil	Highest PID or other location as determined by ENSR field geologist	Southeastern-central portion of the site.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historic use of the site as an illegal solid waste dump.	COCs associated with historic illegal dumping activities are unknown.
MW-1	Soil	Highest PID or other location as determined by ENSR field geologist	West perimeter of the property, adjacent to Third Street	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historical use of the adjacent properties to the west as paper mills.	COCs associated with paper manufacturing, such as mercury and other metals, PCBs, VOCs, and PAHs.
MW-2	Soil	Highest PID or other location as determined by ENSR field geologist	Southern perimeter of property.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Current and historic uses of properties to the south do not represent a REC; MW-2 is placed to minimize data gaps and provide a thorough understanding of the subsurface conditions throughout the entire site, including historic site usage as an illegal solid waste dump.	Unknown; COCs may relate to any upgradient sources and previous site use as an illegal dump.
MW-3	Soil	Highest PID or other location as determined by ENSR field geologist	Central portion of the property, south of existing building	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of impacts at the site generally and specifically associated with the illegal dumping activities. Historic use of the existing building is unknown.	Unknown; COCs may relate to any upgradient sources, historic uses of the existing building, and previous site use as an illegal dump.
MW-4	Soil	Highest PID or other location as determined by ENSR field geologist	Northeastern portion of the property, north of the existing building	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of impacts at the site generally and specifically associated with the UST and the existing building.	COCs associated with the UST historic use of Building 1 include VOCs, SVOCs, metals, and PCBs.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
MW-5	Soil	Highest PID or other location as determined by ENSR field geologist	Northwestern portion of the property, adjacent to former location of Building 1	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of impacts at the site generally and specifically associated with the UST and former Building 1.	COCs associated with the UST and the historic use of Building 1 include VOCs, SVOCs, metals, and PCBs.
SV-1	Soil Vapor	5 ft bgs	Northeast corner of property.	VOCs	Assess the presence of soil vapor impacts in the northeastern portion of the site, as well as determine the potential for off-site migration of any impacts.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.
SV-2	Soil Vapor	5 ft bgs	Northwest corner of property	VOCs	Assess the presence of soil vapor impacts in the northwestern portion of the site, as well as determine the potential for off-site migration of any impacts.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.
SV-3	Soil Vapor	5 ft bgs	West perimeter of the property, adjacent to Third Street	VOCs	Assess the presence of soil vapor impacts in the western portion of the site, as well as determine the potential for off-site migration of any impacts.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.
SV-4	Soil Vapor	5 ft bgs	Southwest corner of property, along southern property line	VOCs	Assess the presence of soil vapor impacts in the southern portion of the site, as well as determine the potential for off-site migration of any impacts.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.
SV-5	Soil Vapor	5 ft bgs	Southern property boundary, southeastern portion of site	VOCs	Assess the presence of soil vapor impacts in the southern portion of the site, as well as determine the potential for off-site migration of any impacts.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
SV-6	Soil Vapor	5 ft bgs	Eastern perimeter of property	VOCs	Assess the presence of soil vapor impacts in the eastern portion of the site, as well as determine the potential for off-site migration of any impacts.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.
SV-7	Soil Vapor	2 inches below slab material	Interior of existing building	VOCs	Assess the presence of contaminated soil vapors and/or potential for future exposures inside the site building.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.
SV-8 (Contingent)	Soil Vapor	2 inches below slab material	Interior of existing building	VOCs	If the soil investigation reveals impacts associated with the existing building, an additional soil vapor sample will be collected from beneath the building foundation to assess the presence of contaminated soil vapors and/or potential for future exposures inside the site building.	Soil vapor impacts would be restricted to VOCs; no other COCs are a soil vapor concern.
TP-1	Soil	Highest PID or other location as determined by ENSR field geologist	Northwest corner of the property, former location of Building 1	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historic use of the former Building 1 included as a paper bag manufacturer, a packing pad manufacturer, and moving/trucking operations. In more recent times, the building was the Fulton Mall, which housed a variety of retail operations. Test pits will specifically look for buried debris or gross subsurface contamination.	COCs associated with historic use of Building 1 include VOCs, SVOCs, metals, and PCBs.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
TP-2	Soil	Highest PID or other location as determined by ENSR field geologist	Southeast corner of property	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	TP-2 will be installed in areas of debris dumping and possible buried debris.	COCs associated with dumping activities are unknown.
TP-3	Soil	Highest PID or other location as determined by ENSR field geologist	Central portion of the site, south of existing building	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	TP-3 is located in the area historically used as an illegal solid waste dump, and will look for buried debris or gross subsurface contamination related to the illegal dumping activities.	COCs associated with illegal dumping activities are unknown.
TP-4	Soil	Highest PID or other location as determined by ENSR field geologist	Eastern portion of the site, adjacent to former location of Building 3	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of impacts relating to historic site use (including illegal solid waste dumping) and specifically with former Building 3. Historic uses of the building include a toilet paper company, a machinery shop, and a warehouse.	COCs associated with the machinery operations may include VOCs, SVOCs, and metals. If toilet paper manufacturing was carried out, COCs typical of paper manufacturing may be present.
TP-5	Soil	Highest PID or other location as determined by ENSR field geologist	Northeast portion of the property.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	TP-5 is located in the area of the site historically used as an illegal solid waste dump, and will look for buried debris or gross subsurface contamination related to the illegal dumping activities.	COCs associated with dumping activities are unknown.
MW-1	Ground water	NA	West perimeter of the property, adjacent to Third Street	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Historical use of the adjacent properties to the west as paper mills.	COCs associated with paper manufacturing, such as mercury and other metals, PCBs, VOCs, and PAHs.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Site Investigation Samples						
MW-2	Ground water	NA	Southern perimeter of property.	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess upgradient groundwater quality	Ideally, no COCs will be associated with the upgradient well location; historic uses of properties to the south do not represent RECs.
MW-3	Ground water	NA	Central portion of the property, south of existing building	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of groundwater impacts at the site generally and specifically associated with the illegal dumping activities. Historic use of the existing building is unknown.	Unknown; COCs may relate to any upgradient sources, historic uses of the existing building, and previous site use as an illegal dump.
MW-4	Ground water	NA	Northeastern portion of the property, north of the existing building	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of groundwater impacts at the site generally and specifically associated with the UST and the existing building.	COCs associated with the UST historic use of Building 1 include VOCs, SVOCs, metals, and PCBs.
MW-5	Ground water	NA	Northwestern portion of the property, adjacent to former location of Building 1	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Assess the presence of groundwater impacts at the site generally and specifically associated with the UST and former Building 1.	COCs associated with the UST and the historic use of Building 1 include VOCs, SVOCs, metals, and PCBs.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
IRM Confirmatory Soil Samples						
IRM-1	Soil	West wall of tank excavation.	UST area	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Post-excavation confirmatory soil samples are required to determine whether the UST has impacted the surrounding soils. In addition, samples will be analyzed for wide array of analytes to assist in thorough site characterization.	Original contents of the UST are unknown; COCs may include VOCs, SVOCs, and metals.
IRM-2	Soil	East wall of tank excavation.	UST area	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Post-excavation confirmatory soil samples are required to determine whether the UST has impacted the surrounding soils. In addition, samples will be analyzed for wide array of analytes to assist in thorough site characterization.	Original contents of the UST are unknown; COCs may include VOCs, SVOCs, and metals.
IRM-3	Soil	North wall of tank excavation.	UST area	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Post-excavation confirmatory soil samples are required to determine whether the UST has impacted the surrounding soils. In addition, samples will be analyzed for wide array of analytes to assist in thorough site characterization.	Original contents of the UST are unknown; COCs may include VOCs, SVOCs, and metals.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
IRM Confirmatory Soil Samples						
IRM-4	Soil	South wall of tank excavation	UST area	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Post-excavation confirmatory soil samples are required to determine whether the UST has impacted the surrounding soils. In addition, samples will be analyzed for wide array of analytes to assist in thorough site characterization.	Original contents of the UST are unknown; COCs may include VOCs, SVOCs, and metals.
IRM-5	Soil	Bottom of tank excavation.	UST area	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Post-excavation confirmatory soil samples are required to determine whether the UST has impacted the surrounding soils. In addition, samples will be analyzed for wide array of analytes to assist in thorough site characterization.	Original contents of the UST are unknown; COCs may include VOCs, SVOCs, and metals.
IRM-6	Soil	Beneath tank piping.	UST area	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Post-excavation confirmatory soil samples are required to determine whether the UST has impacted the surrounding soils. In addition, samples will be analyzed for wide array of analytes to assist in thorough site characterization.	Original contents of the UST are unknown; COCs may include VOCs, SVOCs, and metals.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
IRM Confirmatory Soil Samples						
IRM-7	Soil	Beneath tank piping.	UST area	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Post-excavation confirmatory soil samples are required to determine whether the UST has impacted the surrounding soils. In addition, samples will be analyzed for wide array of analytes to assist in thorough site characterization.	Original contents of the UST are unknown; COCs may include VOCs, SVOCs, and metals.

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Quality Assurance/Quality Control Samples						
QA/QC Duplicate	Soil	Location to be determined by ENSR field geologist	Determined by ENSR field geologist	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Field duplicates ensure that laboratory analyses are accurate.	NA
QA/QC Duplicate	Ground water	NA	Determined by ENSR field geologist	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Field duplicates ensure that laboratory analyses are accurate.	NA
MS/MSD	Soil	Location to be determined by ENSR field geologist	Determined by ENSR field geologist	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	MS/MSD samples are analyzed by the laboratory to ensure that the material sampled does not have qualities that cause interference with the analysis.	NA

Sample ID	Matrix	Sample Depth	Sample Location	Analytical Parameters	Rationale	Contaminants of Concern
Quality Assurance/Quality Control Samples						
MS/MSD	Ground water	NA	Determined by ENSR field geologist	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	MS/MSD samples are analyzed by the laboratory to ensure that the material sampled does not have qualities that cause interference with the analysis.	NA
Rinse Blank	Water	NA	NA	TCL VOCs + MTBE, TCL SVOCs, TAL Metals + Cyanide, PCBs	Rinse blanks are collected by pouring clean, deionized water over field sampling equipment to ensure that sampling equipment is not contaminating samples.	NA
Trip Blank	Water	NA	NA	TCL VOCs + MTBE	Trip blanks are 40 mL vials containing clean, deionized, VOCs-free water, which accompany sample bottles to ensure that ambient VOCs sources are not impacting samples.	NA

5.0 Method and SOP reference table

5.1 Standard operating procedures

Often many routine laboratory and field operations are cataloged to form Standard Operating Procedures (SOPs). Whenever SOPs are applicable and available, they should always be incorporated into the overall data collection activities inherent to performing a Brownfields site investigation. Site-specific Brownfields SAMPs should delineate all activities that could directly or indirectly influence data quality. This should include a determination of all operations that can be covered by SOPs. Therefore, all Site-specific Brownfields SAMPs should contain, at a minimum, SOPs for the following operations:

- Sampling and analytical methodologies.
- Field equipment selection and use.
- Field equipment calibration and standardization.
- Field equipment preventive maintenance.
- QC procedures for intra-laboratory and intra-field activities.
- Data validation.
- Document control procedures.

5.2 Sampling SOPs

To ensure environmental sample collection efforts are representative of site conditions, it is customary to utilize accepted SOPs to optimize sampling activities. Sampling SOPs are proven protocols that may be varied or changed, as required, depending upon site conditions and/or equipment limitations imposed by the procedure. In all instances, those sampling procedures which will be employed to collect environmental samples for a given site investigation must be documented in the Site-specific Brownfields SAMP.

To facilitate the selection of appropriate sample collection techniques, it is advantageous that the sampling SOPs employed for a site-specific Brownfields investigation be derived from an accepted guide. As such, the *U.S.EPA Compendia of Emergency Response Team (ERT) Sampling Procedures* including *Soil Sampling and Surface Geophysics Procedures* and *Groundwater Sampling Procedure,* and the *NYSDOH Guidance for Evaluating Soil Vapor Intrusion in the State of New York* are included as attachments to this Site-specific Brownfields SAMP. The ERT media-specific sampling protocols are the U.S. EPA's accepted SOPs for collecting potentially contaminated environmental matrices of concern such as soil and water. Therefore, to optimize sample collection efforts, these protocols are to be used in conjunction with the *Superfund Program Representative Sampling Guidances*.

5.3 SOP reference table

Project sampling SOPs
1a. Contract Laboratory Program Guidance for Field Samplers (Attachment A)
2a. NYSDOH Guidance for Evaluating Soil Vapor Intrusion in the State of New York (Attachment B)
3a. Superfund Program Representative Sampling Guidances –(Attachment C)
4a. MiniRAE 2000 Instrument Manual, May 2000, prepared by RAE Systems (manufacturer) – (Attachment D)
5a. ENSR SOP #7315 – Operation/Calibration of a Photoionization Detector (PID) (2002) – (Attachment E)
6a. Horiba U-22XD Water Quality Meter Instrument Manual, (no date) – (Attachment F)
7a. GasCheck 5000I Instruction Manual – (Attachment G)
8a. U.S.EPA Compendia of Emergency Response Team (ERT) Sampling Procedures (Attachment H)
9a. ENSR SOP # 7115 – Subsurface Soil Sampling by Split Spoon (1994) – (Attachment I)
10a. ENSR SOP #7116 – Subsurface Soil Sampling by Geoprobe Methods (2006) – (Attachment J)
11a. EPA Region 2 Low Flow Sampling SOP – (Attachment K)
12a. ENSR SOP #7220 – Monitoring Well Construction and Installation (2006) – (Attachment L)
13a. ENSR SOP #7221 – Monitoring Well Development (2006) – (Attachment M)
14a. ENSR SOP #7230 – Test Pit/Trench Subsurface Exploration (2002) – (Attachment N)
15a. ENSR SOP #7510 – Packaging and Shipment of Samples (1999) – (Attachment O)
16a. ENSR SOP #7600 – Decontamination of Equipment (1994) – (Attachment P)
17a. Example ENSR Field Data Sheets – Boring Log, Well Installation Log, Test Pit Log, Monitoring Well/Piezometer Development Record, Low-Flow Groundwater Sample Collection Record (Attachment Q)
18a. Example Sample Label – (Attachment R)
19a. Example of Laboratory Chain of Custody Record and Custody Seal – (Attachment S)
Analytical Method Reference
1b. Volatile Organic Compounds – SW-846 Method 8260B
2b. Semi-Volatile Organic Compounds – SW-846 Method 8270C
3b. Polychlorinated Biphenyls as Aroclors – SW-846 Method 8082

4b. Target Analyte List Metals – SW-846 Methods 6010B/6020/7470A/7471A
5b. Cyanide – SW-846 9012A
6b. Volatile Organic Compounds in Air – USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-15
Project Analytical SOPs
1c. Laboratory Quality Manual – SevernTrent (Currently TestAmerica) Laboratories: Buffalo (2005), New York and Knoxville, Tennessee (2006) – (Attachment T)
2c. Laboratory Standard Operating Procedures for Analytic Methods – SevernTrent (Currently TestAmerica) Laboratories: EPA Methods 8260B (2006), 8270C (2005), 6010B (2006), 6020 (2006) 9012A (2006), 7470A (2006), 7471A (2006), 8082 (2003) – (Attachment U)
3c. Laboratory Standard Operating Procedures for Support Equipment Maintenance, Record Keeping, and Corrective Actions of Analytical Balances, Temperature Control Devices, and Reagent Water (2006) – SevernTrent (Currently TestAmerica) Laboratories – (Attachment V)
4c. Laboratory Control Limits for Organics and Inorganics (2007) – SevernTrent (Currently TestAmerica) Laboratories – (Attachment W)
5c. NYS DEC Analytical Service Protocol Exhibit B, Reporting and Deliverables Requirement (2005) – (Attachment X)

6.0 Sampling and analytical methods requirements

6.1 Sample container preparation and sample preservation

Only new, certified clean (as per OSWER directive 9240.0-05A), sample containers will be used to collect samples for this project. The laboratory will maintain certificates of analysis for each lot of sample containers used and canisters, copies of which will be provided by the laboratory upon request. The appropriate preservatives will be added to each container by the laboratory just prior to shipment to the site. The types of containers are shown in Tables 6-1, 6-2, and 6-3. TCLP sample container types and holding times are presented as contingency samples, in the event that TCLP analysis be required (i.e., waste characterization samples).

Samples shall be preserved according to the preservation techniques given in Tables 6-1, 6-2, and 6-3. Preservatives will be added to the sample bottles by the laboratory prior to their shipment in sufficient quantities to ensure that proper sample pH is met. Following sample collection, the sample bottles used for collection of water and soil/solid waste samples should be placed on ice in the shipping cooler, cooled to 4^{o}C with ice or "blue ice," and delivered to the laboratory within 24 hours of collection. Chain-of-custody procedures are described in Section 9.0. [Note: soil vapor samples do not require cooling, and should be shipped at ambient temperature.]

Table 6-1 Water sample containerization, preservation, and holding times

Six groundwater samples (including one field duplicate) will be collected for analysis. In addition, three rinse blanks and one trip blank (VOC only) will be analyzed:

Analysis	Bottle type	Preservation [(a)]	Holding time [(b)]
Volatile Organic Compounds (VOCs) and MTBE	2-40 mL glass vial w/ Teflon septum	Cool to 4^{o}C HCl to pH <2	10 days
Other Organic Compounds [(c)]	1000 mL glass w/ Teflon lined cap	Cool to 4^{o}C	5 days*
Metals	1000 mL plastic bottle Cool to 4^{o}C	Nitric Acid to pH < 2	6 months, except mercury (26 days)
Cyanide	500 mL plastic bottle	NaOH to pH > 12 Cool to 4^{o}C	12 days

(a) All samples to be preserved in ice during collection and transport.
(b) Days from validated time of sample receipt (VTSR), or 2 days after collection, whichever is less.
(c) Semi-volatile organic compounds or PCBs.
* Holding time to extraction; extracts must be analyzed within 40 days of extraction.

Table 6-2 Soil and waste sample containerization and holding times

Twenty-eight soil samples (including three field duplicates) will be collected for analysis (TCLP analysis presented in the event that waste characterization samples are required):

Analysis	Bottle type	Preservation [a]	Holding time [b]
Volatile Organic Compounds (VOCs) and MTBE	Wide-mouth glass w/ Teflon lined cap	Cool to 4°C	10 days
Other Organic Compounds [c]	Wide-mouth glass w/ Teflon lined cap	Cool to 4°C	10 days*
Metals	Wide-mouth plastic or glass	Cool to 4°C	6 months, except mercury (26 days)
Cyanide	Wide-mouth plastic	Cool to 4°C	12 days
TCLP Organic [d] Compounds	Wide-mouth glass w/ Teflon lined cap	Cool to 4°C	See Table 6-4
TCLP Metals [d]	Wide-mouth plastic or glass	Cool to 4°C	See Table 6-4

(a) All samples to be preserved in ice during collection and transport.
(b) Days from date of VTSR or 2 days after sample collection whichever is less.
(c) Semi-volatile organic compounds and PCBs.
*Holding time to extraction; extracts must be analyzed within 40 days of extraction.
(d) Contingency – TCLP analysis may be required if waste characterization samples are necessary.

Table 6-3 Soil vapor sample containerization, preservation, and holding times

Eight soil vapor samples (including one field duplicate) will be collected for analysis:

Analysis	Bottle type	Preservation	Holding time [a]
Volatile Organic Compounds (VOCs)	6 L pre-cleaned SUMMA® canister	None	30 days

(a) Days from time of sample collection.

6.2 Sample holding times

The sample holding times for organic and inorganic parameters are given in Tables 6-1, 6-2, and 6-3 and must be in accordance with the NYSDEC ASP requirements. Holding times for Toxicity Characteristic Leaching Procedure (TCLP) samples are given in Table 6-4. The NYSDEC ASP holding times must be strictly adhered to by the laboratory. Any holding time exceedances must be reported. As discussed above, TCLP analysis may be required if waste characterization sampling becomes necessary. No samples are currently planned for TCLP analysis.

Table 6-4 TCLP sample holding times

Analytical parameter	From: VSTR To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis
Volatiles	5 days	NA	7 days
Semi-volatiles	5 days	12 days	40 days
Mercury	5 days	NA	26 days
Metals (except Mercury)	180 days	NA	180 days

TCLP - Toxicity Characteristic Leaching Procedure
NA - Not Applicable

All NYSDEC holding times are presented as time elapsed from validated time of sample receipt (VTSR) at the laboratory. Note that NYSDEC requires samples to be delivered to the laboratory within 48 hours of sample collection. While every attempt will be made to deliver samples within 48 hours, this may not always be possible. In those cases, holding times will begin two days after sample collection.

7.0 Preventative maintenance – laboratory and field equipment

7.1 Preventive maintenance procedures

Equipment, instruments, tools, gauges, and other items requiring preventive maintenance will be serviced in accordance with the manufacturer's specified recommendations and written procedure developed by the operators. SOPs 4a, 5a, 6a, and 7a discuss the calibration and operation of field equipment, (See Section 5.3; these SOPs are provided as Appendices D, E, F, and G) and will be used to ensure proper functioning.

A list of critical spare parts will be established by the operator. These spare parts will be available for use in order to reduce the downtime. A service contract for rapid instrument repair or backup instruments may be substituted for the spare part inventory.

Preventative maintenance of laboratory equipment is discussed in Section 5.4.2 of the laboratory-provided Laboratory Quality Manual, Attachment T.

7.2 Schedules

Written procedures will establish the schedule for servicing critical items in order to minimize the downtime of the measurement system. The laboratory will adhere to the maintenance schedule, and arrange any necessary and prompt service. Required service will be performed by qualified personnel.

7.3 Records

Logs shall be established to record and control maintenance and service procedures and schedules. All maintenance records will be documented and traceable to the specific equipment, instruments, tools, and gauges. The field team will maintain and file all logs related to field equipment, and the logs will be reviewed periodically during the project by the QAO.

Laboratory records produced shall be reviewed, maintained, and filed by the operators at the laboratory. The Laboratory Quality Manual (Attachment T) details the processes for reviewing, maintaining, and filing these records (Section 4). The QAO may audit these records to verify complete adherence to these procedures.

8.0 Calibration and corrective action

8.1 Calibration

8.1.1 Field instruments

All field analytical equipment will be calibrated immediately prior to each day's use. The calibration procedures will conform to manufacturer's standard instructions. This calibration will ensure that the equipment is functioning within the allowable tolerances established by the manufacturer and required by the project. Records of all instrument calibration will be maintained by the Field Team Leader. Copies of all the instrument manuals will be maintained on-site by the Field Team Leader.

Calibration procedures for instruments used for monitoring health and safety hazards (e.g., photoionization detector and explosimeter) are provided in the Health and Safety Plan.

8.1.2 Laboratory instruments

The laboratory will follow all calibration procedures and schedules as specified in the sections of the USEPA SW-846 and subsequent updates that apply to the instruments used for the analytical methods given in Section 6.0. Copies of laboratory-provided SOPs are included in Attachment U.

8.2 Internal quality control checks and frequency

8.2.1 Field sample collection

The assessment of field sampling precision will occur through the collection and analysis of field duplicate and MS/MSD samples. The accuracy of field sampling will be evaluated by trip blanks, equipment blanks, and matrix spikes. The procedures associated with the collection of these samples, and the frequency of collection, are defined in Section 11.0 of this SAMP.

8.2.2 Field measurement

QC procedures for pH, temperature, turbidity, specific conductance, water level measurements, and soil VOC headspace measurements will include one or more of the following QC procedures: checking the reproducibility of the measurement by obtaining duplicate readings on a single sample, calibrating the instrument or by comparing it to a known standard, and analyzing QC check samples.

8.2.3 Laboratory analysis

Precision and accuracy determinations for laboratory measurements will be in accordance with the methodologies cited in Section 10.0 of this SAMP. These parameters will be assessed through the use of method blanks, surrogate spikes, internal standard areas, gas chromatograph/mass spectrometer tuning, gas chromatograph/flame ionization detector tuning, laboratory duplicates, LCS, MS, and MS/MSD samples. The types of QC samples for each analytical method are presented in Table 11-1. Corrective action is discussed in Section 8.3 of this SAMP. Qualification of the data based on the QC results is discussed in Section 17 of this SAMP.

8.3 Corrective action

8.3.1 Introduction

The following procedures have been established to ensure that conditions adverse to quality, such as malfunctions, deficiencies, deviations, and errors, are promptly investigated, documented, evaluated, and corrected.

8.3.2 Procedure description

When a significant condition adverse to quality is noted at site, laboratory, or subcontractor location, the cause of the condition will be determined and corrective action will be taken to preclude repetition. Condition identification, cause, reference documents, and corrective action planned to be taken will be documented and reported to the QAO, Project Manager, Field Team Leader and involved contractor management, at a minimum. Implementation of corrective action is verified by documented follow-up action. Section 4.10 of the laboratory-provided Laboratory Quality Manual (Attachment T) describes the laboratory's internal Corrective Action process.

All project personnel have the responsibility, as part of the normal work duties, to promptly identify, solicit approved correction, and report conditions adverse to quality. Corrective actions will be initiated as follows:

- When predetermined acceptance standards are not attained.
- When procedure or data compiled are determined to be deficient.
- When equipment or instrumentation is found to be faulty.
- When samples and analytical test results are not clearly traceable.
- When quality assurance requirements have been violated.
- When designated approvals have been circumvented.
- As a result of system and performance audits.
- As a result of a management assessment.
- As a result of laboratory/field comparison studies, and
- As required by USEPA SW-846, and subsequent updates, the USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, or by the NYSDEC ASP.

Project management and staff, such as field investigation teams, remedial response planning personnel, and laboratory groups monitor on-going work performance in the normal course of daily responsibilities. Project audits are not anticipated to occur during the completion of the Remedial Investigation; however, if conditions adverse to quality are detected, or if the Project Manager requests, audits may occur. Activities or documents ascertained to be noncompliant with quality assurance requirements will be documented. Corrective actions will be mandated through audit finding sheets attached to the audit report. Audit findings will be logged and maintained in hard copy, and controlled by the Task Manager.

Personnel assigned to quality assurance functions will have the responsibility to issue and control Corrective Action Request (CAR) Forms (Figure 8-1 or similar). The CAR identifies the out-of-compliance condition, reference document(s), and recommended corrective action(s) to be administered. The CAR is issued to the personnel responsible for the affected item or activity. A copy is also submitted to the Project Manager.

The individual to whom the CAR is addressed returns the requested response promptly to the QA personnel, affixing his/her signature and date to the corrective action block, after stating the cause of the conditions and corrective action to be taken.

The QA personnel maintain the log for status of CARs, confirms the adequacy of the intended corrective action, and verifies its implementation. CARs will be retained in the project file for the records.

Any project personnel may identify noncompliance issues; however, the designated QA personnel are responsible for documenting, numbering, logging, and verifying the close out action. The Project Manager will be responsible for ensuring that all recommended corrective actions are implemented, documented, and approved.

Figure 8-1 Corrective action request

CORRECTIVE ACTION REQUEST
Number: _____ Date: _____
TO: _____ You are hereby requested to take corrective actions indicated below and as otherwise determined by you to (a) resolve the noted condition and (b) to prevent it from recurring. Your written response is to be returned to the project quality assurance manager by _____
CONDITION:
REFERENCE DOCUMENTS:
RECOMMENDED CORRECTIVE ACTIONS:
_____ _____ _____ _____ _____ _____ Originator Date Approval Date Approval Date
RESPONSE
CAUSE OF CONDITION
CORRECTIVE ACTION (A) RESOLUTION (B) PREVENTION (C) AFFECTED DOCUMENTS
C.A. FOLLOWUP: CORRECTIVE ACTION VERIFIED BY: _____ DATE:_____

9.0 Sample handling and chain of custody requirements

9.1 Sample documentation and handling

An essential element of any Brownfields sampling/analytical scheme is to maintain sample integrity from collection to data reporting. This involves tracing the possession and handling of samples from the time of collection through analysis and final disposition. The documentation used to track a sample's history is referred to as the "chain-of-custody." To facilitate sample chain-of-custody efforts, it is essential to record all inspections, investigations, and photographs that are taken, as well as perform a thorough review of all notes before leaving the site.

To promote the management of sample integrity, it is important that all parties involved understand that a sample is considered to be under a person's custody if; (a) it is in a person's physical possession, (b) in view of that person after he/she has taken possession, (c) secured by that person so that no one can tamper with the sample, or (d) secured by that person in an area which is restricted to authorized personnel. A person who has samples under their custody must always comply with these procedures in order to assure sample integrity.

9.2 Sample documentation

All sample documents should always be legibly written in ink. Any corrections or revisions to sample documentation shall be made by lining through the original entry, initialing, and dating any changes. To elaborate on these requirements, the following sub-sections are provided to outline sample documentation procedures, which should be employed when conducting a Brownfields investigation.

9.2.1 Field logbook

The field logbook is a descriptive notebook detailing site activities and observations so that an accurate and factual account of field procedures may be reconstructed. All entries should be signed by the individuals who are making them. Nonetheless, all field logbook entries should always document the following specific information:

- Site name and project number.
- Contractor name and address.
- Names of personnel on site.
- Dates and times of all entries.
- Descriptions of all site activities, including site entry and exit times.
- Noteworthy events and discussions.
- Weather conditions.
- Site observations.
- Identification and description of samples and locations.
- Subcontractor information and names of on-site personnel.
- Dates and times of sample collections and chain of custody information.
- Records of photographs.
- Site sketches.

- All relevant and appropriate information delineated in field data sheets and sample labels.

9.2.2 Field data sheets and sample labels

Field data sheets, along with corresponding sample labels, are routinely used to identify samples and document field sampling conditions and activities. Examples of the following field data sheets are included as Attachment Q: boring log, monitoring well construction log, test pit log, monitoring well/piezometer development log, and low-flow groundwater sample collection record.

Field data sheets should be completed at the time of sample collection and should always include the following information:

- Site name.
- Contractor name and address.
- Samplers name.
- Sample location and sample identification number.
- Date and time the sample was collected.
- Type of sample collected.
- Brief description of the site.
- Weather conditions.
- Analyses to be performed.
- Sample container, preservation, and storage information.

Sample labels are always to be securely affixed to the sample container. They must always clearly identify the particular sample, and delineate the following information:

- Site name and designated project number.
- Sample identification number.
- Date and time the sample was collected.
- Sample preservation method.
- Sample pH.
- Analysis requested.
- Sampling location.

A representative example of a sample label from the selected laboratory is provided in Attachment R.

9.2.3 Chain of custody record

A chain-of-custody record must always be maintained from the time of sample collection until final deposition. Every transfer of custody will be noted and signed for with a copy of the record being kept for each individual that endorsed it. It is integral that the chain-of-custody record should always include the following information:

- Contractor name and address.
- Sample identification number.
- Sample location.

- Sample collection date and time.
- Sample information (matrix type, number of bottles collected, container type, etc).
- Names and signatures of samplers.
- Signatures of all individuals who have had custody of the samples.

An example of the laboratory chain of custody is provided in Attachment S.

9.2.4 Custody seals

Custody seals are used to demonstrate that a sample container has not been opened or tampered with. When samples are not under direct control of the individual currently responsible for them, they will be stored in a locked container, which is to be affixed with a custody seal. The individual who has sample custody shall always sign, date, and affix the custody seal to the locked container in such a manner that it cannot be opened unless it is broken. An example is presented in Attachment S.

9.2.5 Sample handling and shipment

It is customary for field sampling personnel to always transport environmental samples directly to the laboratory within 24 hours of sample collection. To assist in these efforts, field sampling personnel should consider utilizing an overnight delivery service within 24 hours of sample collection.

When preparing sample coolers for shipment they should always be securely closed with a custody seal affixed to each shipping container. All sample containers will be labeled as described above. Subsequently, they are to be placed in an appropriate transport container and packed with an absorbent material such as vermiculite. All sample containers will be packed with ice to maintain a temperature of 4°C. All sample documentation will then be affixed to the underside of each transport container lid. The transport container lid will then be closed and affixed with a custody seal accordingly.

Regulations for packaging, marking/labeling, and shipping hazardous materials and wastes are issued by the U.S. Department of Transportation (U.S. DOT). Air carriers that transport hazardous materials, such as Federal Express, may also require compliance with the current edition of the International Air Transport Association (IATA) Dangerous Goods Regulations. The IATA protocol details the procedures for the shipment and transportation of hazardous materials by a common air carrier. It is important to note that following all current IATA regulations will ensure compliance with U.S. DOT protocol.

9.3 Site-specific sample handling and chain of custody requirements

This section presents sample custody procedures for both the field and laboratory. Implementation of proper custody procedures for samples generated in the field is the responsibility of field personnel. Both laboratory and field personnel involved in the Chain-of-custody (COC) and transfer of samples will be trained as to the purpose and procedures prior to implementation.

Evidence of sample traceability and integrity is provided by COC procedures. These procedures document the sample traceability from the selection and preparation of the sample containers by the laboratory, to sample collection, to sample shipment, to laboratory receipt and analysis. The sample custody flowchart is shown in Figure 9.1. A sample is considered to be in a person's custody if the sample is:

- In a person's possession.
- Maintained in view after possession is accepted and documented.
- Locked and tagged with Custody Seals so that no one can tamper with it after having been in physical custody, or

- In a secured area which is restricted to authorized personnel.

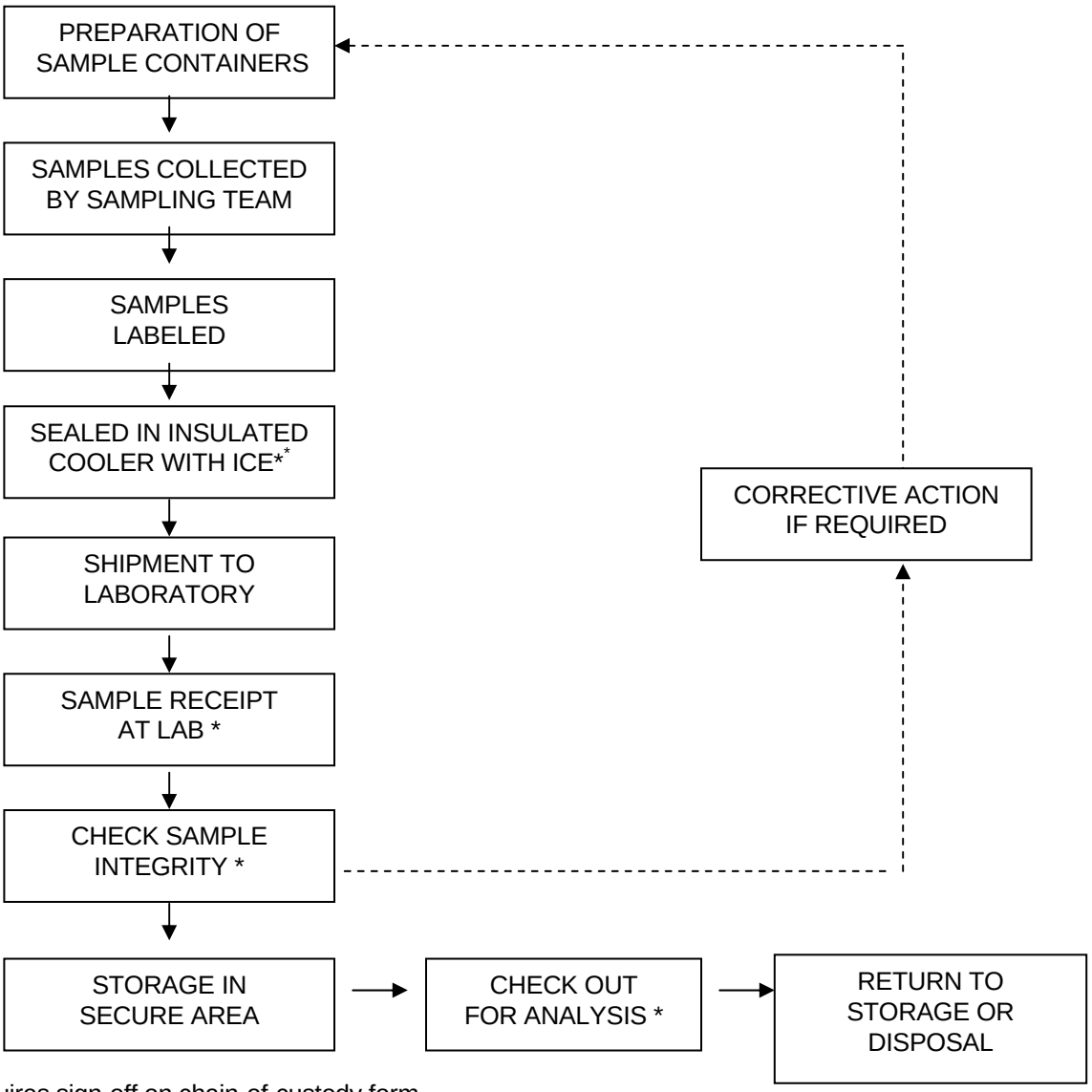
9.3.1 Field sample custody

A COC record accompanies the sample containers from selection and preparation at the laboratory, during shipment to the field for sample containment and preservation, and during return to the laboratory. Triplicate copies of the COC must be completed for each sample set collected.

The COC lists the field personnel responsible for taking samples, the project name and number, the name of the analytical laboratory to which the samples are sent, and the method of sample shipment. The COC also lists a unique description of every sample bottle in the set. If samples are split and sent to different laboratories, a copy of the COC record will be sent with each sample.

The REMARKS space on the COC is used to indicate if the sample is a matrix spike, matrix spike duplicate, or any other sample information for the laboratory. Once all bottles are properly accounted for on the form, a sampler will write his or her signature and the date and time on the first RELINQUISHED BY space. The sampler will also write the method of shipment, the shipping cooler identification number, and the shipper air bill number on the top of the COC. Mistakes will be crossed out with a single line in ink, initialed, and dated by the author.

Figure 9-1 Sample custody flowchart



*Requires sign-off on chain-of-custody form

** Ice not used for soil vapor samples

One copy of the COC is retained by sampling personnel and the other two copies are put into a sealable plastic bag and taped inside the lid of the shipping cooler. The cooler lid is closed, custody seals provided by the laboratory are affixed to the latch and across the back and front lids of the cooler, and the person relinquishing the samples signs their name across the seal. The seal is taped, and the cooler is wrapped tightly with clear packing tape. It is then relinquished by field personnel to personnel responsible for shipment, typically an overnight carrier. The COC seal must be broken to open the container. Breakage of the seals before receipt at the laboratory may indicate tampering. If tampering is apparent, the laboratory will contact the Project Manager, and the sample will not be analyzed.

9.4 Laboratory sample custody

The Project Manager or Field Team Leader will notify the laboratory of upcoming field sampling activities, and the subsequent shipment of samples to the laboratory. This notification will include information concerning the number and type of samples to be shipped as well as the anticipated date of arrival.

The following laboratory sample custody procedures will be used:

- The laboratory will designate a sample custodian who will be responsible for maintaining custody of the samples, and for maintaining all associated records documenting that custody.
- Upon receipt of the samples, the custodian will check cooler temperature, and check the original COC documents and compare them with the labeled contents of each sample container for correctness and traceability. The sample custodian will sign the COC record and record the date and time received.
- Care will be exercised to annotate any labeling or descriptive errors. In the event of discrepant documentation, the laboratory will immediately contact the Project Manager or Field Team Leader as part of the corrective action process. A qualitative assessment of each sample container will be performed to note any anomalies, such as broken or leaking bottles. This assessment will be recorded as part of the incoming chain-of-custody procedure.
- The samples will be stored in a secured area at a temperature of 4 ± 2ºC (except soil vapor samples) until analyses commence.
- A laboratory tracking record will accompany the sample or sample fraction through final analysis for control.
- A copy of the tracking record will accompany the laboratory report and will become a permanent part of the project records.

10.0 Analytical precision and accuracy

10.1 Analytical data quality requirements and assessments

An important aspect in the Brownfields project planning process is to define what levels of data are required. These data quality requirements are to be based on a common understanding of its intended use, the complexity of the measurement process, and the availability of resources. Once data quality requirements are clearly determined, QC protocols are to be defined for measuring whether these environmental monitoring acceptance/performance criteria are being met.

10.2 Data acceptance/performance criteria

When conducting a Brownfields site investigation, it is essential to collect data that are of sufficient quantity and quality to support accurate decision-making. The most effective way to accomplish these objectives is to determine the type, quantity, and quality of environmental measurement data that are necessary to achieve monitoring goals prior to the commencement of sampling. To assure the level of detail is commensurate with the objectives of a Brownfields site investigation, a common sense "systematic planning" approach should be followed. This process is useful in promoting the development of "acceptance and/or performance criteria" for gauging the collection, evaluation, and use of environmental measurement data.

Data "acceptance and/or performance criteria" are prerequisites established to specify the quality of Brownfields site investigation environmental monitoring results required to support decisions. Data acceptance/performance criteria are predicated in accordance with the anticipated end uses of the information that are to be collected. The establishment of data acceptance/performance criteria is applicable to all phases and aspects of the remediation process including site investigation, design, construction, and clean up operations. It is important to note that the level of detail and quality needed will often vary with the intended use of the data. Consequently, in most instances QA/QC activities involving precision and accuracy determinations are relied upon to assess acceptance/performance criteria.

10.3 Analytical precision

Analytical precision measurements are typically determined when performing instrumental analyses to assess the errors associated with analyte interferences, sample heterogeneity, and poor laboratory practices. They are commonly undertaken by incorporating matrix spike, matrix spike duplicate, and/or matrix duplicate quality control sample analyses into the analytical scheme. Precision measures are often best expressed by calculating the Relative Percent Difference (RPD) between a sample and its duplicate determination. The Relative Percent Difference (RPD) between the two results will be calculated as follows and used as an indication of the precision of the analyses performed:

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

S = Sample
D = Duplicate
| | = Indicates absolute value of the difference to express RPD as a positive value.

10.4 Analytical accuracy

Analytical accuracy determinations are typically undertaken when performing instrumental analyses to assess the proficiency of the measurement process.

They are commonly undertaken by incorporating calibration verification, method blank, calibration blank, method control, surrogate spike, and/or matrix spike quality control sample analyses into the analytical scheme. Accuracy measures are often best expressed by calculating the Percent Recovery (%R) between true and found values as follows:

% R = A/B x 100

A = The found analyte concentration determined experimentally.
B = The true analyte concentration.

10.5 Analytical precision and accuracy requirements

The quality assurance and quality control objectives for all measurement data include precision, accuracy, representativeness, completeness, and comparability. These objectives are defined in following subsections. The analytical methods and their Contract Required Quantitation Limits (CRQLs) are given in Section 10.0.

10.5.1 Precision

Precision is an expression of the reproducibility of measurements of the same parameter under a given set of conditions. Specifically, it is a quantitative measurement of the variability of a group of measurements compared to their average value (USEPA, 1987). Precision is usually stated in terms of standard deviation, but other estimates such as the coefficient of variation (relative standard deviation), range (maximum value minus minimum value), relative range, and relative percent difference (RPD) are common.

For this project, field sampling precision will be determined by analyzing coded duplicate samples (labeled so that the laboratory does not recognize them as duplicates) for the same parameters, and then, during data validation (Section 12.0), calculating the RPD for duplicate sample results.

Field duplicate samples will be collected at a frequency of one for every 20 or fewer investigative samples of each matrix. Therefore, one field duplicate will be collected from the soil boring samples, one field duplicate will be collected from the monitoring well samples, and one field duplicate will be collected for the soil vapor samples.

Field duplicates for solid samples should have an RPD of less than or equal to 50%; field duplicates for aqueous samples should have an RPD of less than or equal to 30%; field duplicates for soil vapor samples should have an RPD of less than or equal to 50%. These criteria apply only when analyte concentrations in both the sample and duplicate are greater than five times the detection limit. If the analyte concentration in the sample and/or duplicate is less than five times the detection limit, but greater than the detection limit, the criteria will be doubled. These criteria apply to both inorganic and organic analyses.

Analytical precision will be determined by the laboratory by calculating the RPD for the results of the analysis of internal QC duplicates (inorganics) and matrix spike duplicates (organics). The formula for calculating RPD is as follows:

$$RPD = \frac{|V1 - V2|}{(V1 + V2)/2} \times 100$$

Where:

RPD = Relative Percent Difference.
V1, V2 = The two values to be compared.
|V1 - V2| = The absolute value of the difference between the two values.

The precision (RPD) and accuracy (percent recovery) criteria for inorganics are defined in the respective methods and are therefore presented in the text. In accordance with section 9.7 of SW-846 Method 8000C, criteria for organic analyses are developed by the laboratory and are statistically derived values based on historical data. As such, they may vary over time. ENSR has provided a copy of the laboratory's current limits in Attachment W. It will be the responsibility of ENSR's QAO to determine if variations in the laboratory's acceptance limits are reasonable. If the QAO determines that the changes are significant, the laboratory will be required to submit an explanation for the changes, and ENSR may require that the laboratory use the limits provided at the time this SAMP is approved. The RPD criteria for inorganic analytes are 20% for aqueous samples and 50% for solid samples and 25% for soil vapor samples.

10.5.2 Accuracy

Accuracy is a measure of the degree of agreement of a measured value with the true or expected value of the quantity of concern (Taylor, 1987), or the difference between a measured value and the true or accepted reference value. The accuracy of an analytical procedure is best determined by the analysis of a sample containing a known quantity of material, and is expressed as the percent of the known quantity which is recovered or measured. The recovery of a given analyte is dependent upon the sample matrix, method of analysis, and the specific compound or element being determined. The concentration of the analyte relative to the detection limit of the analytical method is also a major factor in determining the accuracy of the measurement. Concentrations of analytes which are close to the detection limits are less accurate because they are more affected by such factors as instrument "noise". Higher concentrations will not be as affected by instrument noise or other variables and thus will be more accurate.

Sampling accuracy may be determined through the assessment of the analytical results of equipment blanks and trip blanks for each sample set. Analytical accuracy is typically assessed by examining the percent recoveries of surrogate compounds that are added to each sample (organic analyses only), and the percent recoveries of matrix spike compounds added to selected samples and laboratory blanks. Additionally, initial and continuing calibrations must be performed and accomplished within the established method control limits to define the instrument accuracy before analytical accuracy can be determined for any sample set.

Accuracy in the laboratory can be measured as the percent recovery (%R) of a known amount of analyte, called a spike, added to a sample (matrix spike [MS]) or to a blank (blank spike or laboratory control sample [LCS]). The %R is calculated as follows:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where:

- %R = Percent recovery.
- SSR = Spike sample result: concentration of analyte obtained by analyzing the sample with the spike added.
- SR = Sample result: the background value, i.e., the concentration of the analyte obtained by analyzing the sample.
- SA = Spiked analyte: concentration of the analyte spike added to the sample.

The laboratory must analyze MS and LCS samples at the frequency specified in the method (usually 1 per 20 samples or one per analytical batch). The laboratory must spike the full target compound list into each MS and LCS sample for all analyses.

The precision (RPD) and accuracy (percent recovery) criteria for inorganics are defined in the respective methods and are therefore presented in the text. In accordance with section 9.7 of SW-846 Method 8000C, criteria for organic analyses are developed by the laboratory and are statistically derived values based on historical data. As such, they may vary over time. ENSR has provided a copy of the laboratory's current limits in Attachment W. It will be the responsibility of ENSR's QAO to determine if variations in the laboratory's acceptance limits are reasonable. If the QAO determines that the changes are significant, the laboratory will be required to submit an explanation for the changes, and ENSR may require that the laboratory use the limits provided at the time this SAMP is approved. The lower acceptance limit must be greater than or equal to 10% for all analytes.

Note that the matrix spike may or may not be performed on a project sample due to the limited number of samples being collected in this investigation, and that MS samples are not applicable to soil gas analysis by method TO-15.

10.5.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program (USEPA, 1987). Samples must be representative of the environmental media being sampled. Selection of sample locations and sampling procedures will incorporate consideration of obtaining the most representative sample possible.

Field and laboratory procedures will be performed in such a manner as to ensure, to the degree that is technically possible, that the data derived represents the in-place quality of the material sampled. Every effort will be made to ensure chemical compounds will not be introduced into the sample via sample containers, handling, and analysis. All soil gas canisters must free of contamination prior to sampling. Decontamination of sampling devices and digging equipment will be performed between samples as outlined in the Field Sampling Plan. Analysis of equipment blanks, trip blanks, and method blanks will also be performed to monitor for potential sample contamination from field and laboratory procedures.

The assessment of representativeness also must consider the degree of heterogeneity in the material from which the samples are collected. Sampling heterogeneity will be evaluated during data validation through the analysis of coded field duplicate samples. The analytical laboratory will also follow acceptable procedures to assure the samples are adequately homogenized (except for VOC analyses) prior to taking aliquots for analysis, so the reported results are representative of the sample received.

Chain-of-custody procedures will be followed to document that contamination of samples has not occurred during container preparation, shipment, and sampling. Details of blank, duplicate and Chain-of-custody procedures are presented in Sections 9.0 and 11.0.

10.5.4 Completeness

Completeness is defined as the percentage of measurements made which are judged to be valid (USEPA, 1987). The QC objective for completeness is generation of valid data for at least 90 percent of the analyses requested. Completeness is defined as follows for all sample measurements:

$$\%C = \frac{V}{T} \times 100$$

where: %C = Percent completeness.
V = Number of measurements judged valid.
T = Total number of measurements.

10.5.5 Comparability

Comparability expresses the degree of confidence with which one data set can be compared to another (USEPA, 1987). The comparability of all data collected for this project will be ensured by:

- Using identified standard methods for both sampling and analysis phases of this project;
- Requiring traceability of all analytical standards and/or source materials to the U.S. Environmental Protection Agency (USEPA) or National Institute of Standards and Technology (NIST);
- Requiring that all calibrations be verified with an independently prepared standard from a source other than that used for calibration (if applicable);
- Using standard reporting units and reporting formats including the reporting of QC data;
- Performing a data package review as described in Section 16, including the use of data qualifiers in all cases where appropriate; and
- Requiring that all validation qualifiers be used any time an analytical result is used for any purpose.

These steps will ensure all future users of either the data or the conclusions drawn from them will be able to judge the comparability of these data and conclusions.

10.6 Analytical procedures

Samples will be analyzed according to the USEPA SW-846 "Test Methods for Evaluating Solid Waste," November 1986, 3rd edition and subsequent updates. The methods to be used for the laboratory analysis of water and soil samples and the estimated quantitation limits are presented in the tables below. The corresponding State of New York Standards (where available) are presented for comparison.

Table 10-1 Quantitation limits

Volatile Organics (5 mL purge)						
		Estimated Quantitation Limits			State of New York Standards	
	Analysis/Compound	Method	Water (ug/L)	Soil (ug/kg)	Water (ug/L) [a]	Soil (ug/kg) [b]
1	1,1,1-Trichloroethane	SW8260B	1	5	5	680
2	1,1,2,2-Tetrachloroethane	SW8260B	1	5	5	
3	1,1,2-Trichloroethane	SW8260B	1	5	1	
4	1,1-Dichloroethane	SW8260B	1	5	5	270
5	1,1-Dichloroethene	SW8260B	1	5	5	330
6	1,2-Dichloroethane	SW8260B	1	5	0.6	20 or SB
7	1,2-Dichloroethene(total)	SW8260B	1	5	5	
8	1,2-Dichloropropane	SW8260B	1	5	1	
9	2-Butanone (MEK)	SW8260B	10	25		120
10	2-Hexanone	SW8260B	10	25		
11	4-Methyl-2-pentanone(MIBK)	SW8260B	5	25		
12	Acetone	SW8260B	10	25		50
13	Benzene	SW8260B	1	5	1	60
14	Bromodichloromethane	SW8260B	1	5		
15	Bromoform	SW8260B	1	5		
16	Bromomethane	SW8260B	2	10	5	
17	Carbon Disulfide	SW8260B	1	5		
18	Carbon Tetrachloride	SW8260B	1	5	5	760
19	Chlorobenzene	SW8260B	1	5	5	1100
20	Chloroethane	SW8260B	2	10	5	
21	Chloroform	SW8260B	1	5	7	370
22	Chloromethane	SW8260B	2	10	5	
23	cis-1,3-Dichloropropene	SW8260B	1	5	0.4	
24	Dibromochloromethane	SW8260B	1	5	5	
25	Ethyl Benzene	SW8260B	1	5	5	1000
26	Methyl-Tert-Butyl-Ether	SW8260B	1	5		930
27	Methylene Chloride	SW8260B	1	5	5	50
28	Styrene	SW8260B	1	5	5	
29	Tetrachloroethene	SW8260B	1	5	5	1300
30	Toluene	SW8260B	1	5	5	700
31	trans-1,3-Dichloropropene	SW8260B	1	5	0.4	
32	Trichloroethene	SW8260B	1	5	5	470
33	Vinyl Chloride	SW8260B	2	10	2	20
34	Xylenes(total)	SW8260B	1	5	5	260

Volatile Organics in Soil Vapor				
		Estimated Quantitation Limits		State of New York Background Concentrations
	Analysis/Compound	Method	Soil Vapor (ug/m3)	Soil Vapor (ug/m3) [C]
1	1,1,1-Trichloroethane	TO-15	1	0.3
2	1,1,2,2-Tetrachloroethane	TO-15	1	0.1
3	1,1,2-Trichloroethane	TO-15	1	0.2
4	1,1-Dichloroethane	TO-15	1	0.1
5	1,1-Dichloroethene	TO-15	1	0.1
6	1,2-Dichloroethane	TO-15	1	0.1
7	1,2-Dichloroethene(total)	TO-15	1	0.2 (as cis-1,2-dichloroethene)
8	1,2-Dichloropropane	TO-15	1	0.4
9	2-Butanone (MEK)	TO-15	1	6.2
10	2-Hexanone	TO-15	1	
11	4-Methyl-2-pentanone(MIBK)	TO-15	1	0.8
12	Acetone	TO-15	5	16
13	Benzene	TO-15	1	1.9
14	Bromodichloromethane	TO-15	1	
15	Bromoform	TO-15	1	
16	Bromomethane	TO-15	1	0.4
17	Carbon Disulfide	TO-15	1	
18	Carbon Tetrachloride	TO-15	1	0.4
19	Chlorobenzene	TO-15	1	0.1
20	Chloroethane	TO-15	1	0.2
21	Chloroform	TO-15	1	0.2
22	Chloromethane	TO-15	1	1.3
23	cis-1,3-Dichloropropene	TO-15	1	0.2
24	Dibromochloromethane	TO-15	1	
25	Ethyl Benzene	TO15	1	0.8
26	Methyl-Tert-Butyl-Ether	TO-15	1	
27	Methylene Chloride	TO-15	1	0.8
28	Styrene	TO-15	1	0.2
29	Tetrachloroethene	TO-15	1	0.6
30	Toluene	TO-15	1	11
31	trans-1,3-Dichloropropene	TO-15	1	0.1
32	Trichloroethene	TO-15	1	0.2
33	Vinyl Chloride	TO-15	1	0.2
34	Xylenes(total)	TO-15	1	0.8 (m,p-xylene) 0.7 (o-xylene)

Semi volatile Organics						
		Estimated Quantitation Limits			State of New York Standards	
	Analysis/Compound	Method	Water (ug/L)	Soil (ug/kg)	Water (ug/L) [a]	Soil (ug/kg) [b]
1	1,2,4-Trichlorobenzene	SW8270C	10	330	5	
2	1,2-Dichlorobenzene	SW8270C	10	330	3	1100
3	1,3-Dichlorobenzene	SW8270C	10	330	3	2400
4	1,4-Dichlorobenzene	SW8270C	10	330	3	1800
5	2,2'-oxybis(1-chloropropane)	SW8270C	10	330	5	
6	2,4,5-Trichlorophenol	SW8270C	25	330	1	
7	2,4,6-Trichlorophenol	SW8270C	10	330	1	
8	2,4-Dichlorophenol	SW8270C	10	330	1	
9	2,4-Dimethylphenol	SW8270C	10	330	1	
10	2,4-Dinitrophenol	SW8270C	25	330	1	
11	2,4-Dinitrotoluene	SW8270C	10	330	5	
12	2,6-Dinitrotoluene	SW8270C	10	330	5	
13	2-Chloronaphthalene	SW8270C	10	330		
14	2-Chlorophenol	SW8270C	10	330	1	
15	2-methyl-4,6-Dinitrophenol	SW8270C	25	330		
16	2-Methylnaphthalene	SW8270C	10	330		
17	2-Methylphenol	SW8270C	10	330	1	330
18	2-Nitrolaniline	SW8270C	25	330	5	
19	2-Nitrophenol	SW8270C	10	330	1	
20	3,3'-Dichlorobenzidine	SW8270C	10	330	5	
21	3-Nitroaniline	SW8270C	25	330	5	
22	4-Bromophenyl-phenyl ether	SW8270C	10	330		
23	4-Chloro-3-methylphenol	SW8270C	10	330		
24	4-Chloroaniline	SW8270C	10	330	5	
25	4-Chlorophenyl-phenyl ether	SW8270C	10	330		
26	4-Methylphenol	SW8270C	10	330	1	330
27	4-Nitroaniline	SW8270C	25	330	5	
28	4-Nitrophenol	SW8270C	25	330	1	
29	Acenaphthene	SW8270C	10	330		20000
30	Acenaphthylene	SW8270C	10	330		100000
31	Anthracene	SW8270C	10	330		100000
32	Benzo(a)anthracene	SW8270C	10	330		1000 or SB
33	Benzo(a)pyrene	SW8270C	10	330		1000 or SB
34	Benzo(b)fluoranthene	SW8270C	10	330		1000 or SB
35	Benzo(g,h,i)perylene	SW8270C	10	330		100000
36	Benzo(k)fluoranthene	SW8270C	10	330		800 or SB
37	bis(2-Chloroethoxy) methane	SW8270C	10	330	5	
38	bis(2-Chloroethyl) ether	SW8270C	10	330	1	
39	bis(2-ethylhexyl)phthalate	SW8270C	10	330	5	
40	Butylbenzylphthalate	SW8270C	10	330		
41	Carbazole	SW8270C	10	330		
42	Chrysene	SW8270C	10	330		1000 or SB
43	Di-n-butylphthalate	SW8270C	10	330	50	

Semi volatile Organics						
		Estimated Quantitation Limits			State of New York Standards	
	Analysis/Compound	Method	Water (ug/L)	Soil (ug/kg)	Water (ug/L) [(a)]	Soil (ug/kg) [(b)]
44	Di-n-octylphthalate	SW8270C	10	330		
45	Dibenz(a,h)anthracene	SW8270C	10	330		33000
46	Dibenzofuran	SW8270C	10	330		
47	Diethylphthalate	SW8270C	10	330		
48	Dimethylphthalate	SW8270C	10	330		
49	Fluoranthene	SW8270C	10	330		100000
50	Fluorene	SW8270C	10	330		30000
51	Hexachlorobenzene	SW8270C	10	330		330
52	Hexachlorobutadiene	SW8270C	10	330	0.5	
53	Hexachlorocyclopentadiene	SW8270C	10	330	5	
54	Hexachloroethane	SW8270C	10	330	5	
55	Indeno(1,2,3-cd)pyrene	SW8270C	10	330		500 or SB
56	Isophorone	SW8270C	10	330		
57	N-Nitroso-di-n-propylamine	SW8270C	10	330		
58	N-Nitrosodiphenylamine	SW8270C	10	330		
59	Naphthalene	SW8270C	10	330		12000
60	Nitrobenzene	SW8270C	10	330	0.4	
61	Pentachlorophenol	SW8270C	25	330	1	800
62	Phenanthrene	SW8270C	10	330		100000
63	Phenol	SW8270C	10	330	1	330
64	Pyrene	SW8270C	10	330		100000

PCBs						
		Estimated Quantitation Limits			State of New York Standards	
	Analysis/Compound	Method	Water (ug/L)	Soil (ug/kg)	Water (ug/L) [(a)]	Soil (ug/kg) [(b)]
1	Aroclor-1016	SW8082	1.0	33	0.09	100 (Total PCBs)
2	Aroclor-1221	SW8082	2.0	33	0.09	
3	Aroclor-1232	SW8082	1.0	33	0.09	
4	Aroclor-1242	SW8082	1.0	33	0.09	
5	Aroclor-1248	SW8082	1.0	33	0.09	
6	Aroclor-1254	SW8082	1.0	33	0.09	
7	Aroclor-1260	SW8082	1.0	33	0.09	
8	Aroclor-1262	SW8082	1.0	33	NA	
9	Aroclor-1268	SW8082	1.0	33	NA	

Metals						
		Estimated Quantitation Limits			State of New York Standards	
	Analysis/Compound	Method	Water (mg/L)	Soil (mg/kg)	Water (mg/L) [a]	Soil (mg/kg) [b]
1	Antimony	SW6020	0.001	0.1	0.003	
2	Arsenic	SW6010B	0.01	2	0.025	13 or SB
3	Barium	SW6010B	0.01	1	1	350 or SB
4	Beryllium	SW6010B	0.005	0.5	0.003	7.2
5	Cadmium	SW6010B	0.005	0.5	0.005	2.5 or SB
6	Chromium	SW6010B	0.01	1	0.05	30 or SB
7	Copper	SW6010B	0.03	2.5	0.2	50
8	Lead	SW6010B	0.01	1	0.025	63 or SB
9	Mercury	SW7470A/7471A	0.0002	0.01	0.0007	0.18 or SB
10	Nickel	SW6010B	0.04	4	0.1	30
11	Selenium	SW6010B	0.015	4	0.01	3.9 or SB
12	Silver	SW6010B	0.01	1	0.05	2
13	Thallium	SW6020	0.0002	0.01	0.0005	
14	Zinc	SW6010B	0.02	2	2	109 or SB
15	Vanadium	SW6010B	0.05	1	0.0005	
16	Cobalt	SW6010B	0.05	1		
17	Aluminum	SW6010B	0.2	20		
18	Calcium	SW6010B	5	500		
19	Iron	SW6010B	0.1	10	0.3	
20	Magnesium	SW6010B	5	500	35	
21	Manganese	SW6010B	0.015	1.5	0.3	1600 or SB
22	Potassium	SW6010B	5	500		
23	Sodium	SW6010B	5	500	20	
24	Cyanide	SW9012A	0.01	0.01	200	27

Notes:

N/A - Not Applicable

(a) - Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, NYSDEC, October 1993

(b) - Standard Guidance Value from 6 NYCRR Part 375 Soil Cleanup Objectives for Unrestricted Use, December 14, 2006

(c) - Mean outdoor air concentration from study of VOCs in air of fuel heated homes, Final NYSDOH CEH BEEI Soil Vapor Intrusion Guidance, Appendix C, October 2006 (Table C-1).

SB – Site background level.

11.0 Field quality control requirements

11.1 Data measurement quality objectives

When conducting a Brownfields site investigation, all measurements should be made so that results are reflective of the environmental media and conditions being measured. To assess if environmental monitoring measurements are of an appropriate quality, “acceptance and/or performance criteria” are typically established. Acceptance/performance criteria are commonly assessed by evaluating the Precision, Accuracy, Representativeness, Completeness, and Comparability (PARCC) of pertinent QA/QC options specified for sampling and analytical activities.

- Precision; a measure of the reproducibility of analyses under a given set or conditions.
- Accuracy; a measure of the bias that exists in a measurement system.
- Representativeness; the degree sampling data accurately and precisely depict selected characteristics.
- Completeness; the measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under “normal” conditions.
- Comparability; the degree of confidence with which one data set can be compared to another.

11.2 Sample collection precision

Sample collection precision is customarily assessed by collecting field duplicate samples. Field duplicate samples are used to evaluate errors associated with sample heterogeneity, sampling methodology and analytical procedures. The analytical results from these samples are important because they provide data to evaluate overall measurement precision.

11.3 Sample collection accuracy

To assess sample accuracy, field QC samples such as rinsate, trip, and/or field blanks, are typically incorporated into the sampling scheme. The data acquired from the analysis of blanks are useful in their ability to evaluate errors which can arise from cross-contamination. The occurrence of cross-contamination can result from the improper handling of samples by field and/or lab personnel, improper decontamination procedures, improper shipment and storage, and on-site atmospheric contaminants. Therefore, to facilitate sample collection accuracy, it is essential to maintain the frequent and thorough review of field procedures so that deficiencies can be quickly documented and corrected.

11.4 Sample collection representativeness

Representativeness is an expression of the degree to which a sample accurately and precisely represents a characteristic of a population, parameter variations at a sampling point or an environmental condition. Representativeness is a qualitative parameter that relies upon the proper design of a fitting sampling program and proper laboratory protocol. This criterion is best satisfied by making certain that sampling locations are selected properly and a sufficient number of samples are collected. Therefore, sample representativeness will be assessed by collecting field duplicates. Traditionally, field duplicates are by definition, equally representative of a given point in space and time.

11.5 Sample collection comparability

Comparability is defined as an expression of the confidence with which one data set can be compared to another.

In most instances, the proficiency of field sampling efforts will be the determining factor that affects the overall comparability of environmental measurement data. To optimize the comparability of environmental measurement data, sample collection activities should always be performed using standardized procedures whenever possible. When performing a Brownfields site investigation, these efforts will be facilitated by adhering to the quality control criteria and technical guidelines put forth in this Site-specific Brownfields SAMP.

11.6 Sample collection completeness

Completeness is defined as the measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. Data completeness is often expressed as the percentage of valid data obtained from a given measurement system. To consider data valid, it is customary to assess if a set of data satisfies all of the specified acceptance/performance criteria (accuracy measures, precision measures, etc.) to render a determination. This necessitates that the data acquired for all confirmatory analyses critical to a Brownfields site investigation sampling program be validated (100%). Therefore, by performing a full data validation effort to ensure completeness, the rationale for considering data points non-critical will not be required.

11.7 Sampling quality control requirements

11.7.1 Field QC samples

To assess field sampling and decontamination performance, two types of "blanks" will be collected and submitted to the laboratory for analyses. In addition, the precision of field sampling procedures will be assessed by collecting coded field duplicates and matrix spike/matrix spike duplicates (MS/MSDs).

The blanks will include:

- Trip Blanks - A Trip Blank will be prepared before the sample containers are sent by the laboratory. The trip blank will consist of a 40-ml VOA vial containing distilled, deionized water, which accompanies the other water sample bottles into the field and back to the laboratory. A trip blank will be included with each shipment of water samples for target compound list (TCL) volatiles analysis. The Trip Blank will be analyzed for TCL volatile organic compounds to assess any contamination from sampling and transport, and internal laboratory procedures.
- Equipment Blanks - Equipment Blanks will be taken at a minimum frequency of one for each type of equipment used each day a decontamination event is carried out. Equipment Blanks are used to determine the effectiveness of the decontamination procedures for sampling equipment. It is a sample of deionized, distilled water provided by the laboratory that has passed through a decontaminated bailer or other sampling apparatus. It is usually collected as a last step in the decontamination procedure, prior to taking an environmental sample. The field blank may be analyzed for all or some of the parameters of interest. Equipment blanks are not applicable to soil vapor samples.

The duplicates will consist of:

- Coded Field Duplicate - To determine the representativeness of the sampling methods, coded field duplicates will be collected. The samples are termed "coded" because they will be labeled in such a manner that the laboratory will not be able to determine that they are a duplicate sample. This will eliminate any possible bias that could arise. Field duplicates will be collected at a frequency of one duplicate per 20 field samples.
- Matrix Spike/Matrix Spike Duplicate (MS/MSD) - MS/MSD samples (MS/MSD for organics; MS and laboratory duplicate for inorganics) will be taken at a frequency of one pair per 20 field samples.

These samples are used to assess the effect of the sample matrix on the recovery of target compounds or target analytes. MS/MSD samples are not applicable to soil vapor samples.

Table 11-1 details the laboratory and field Quality Control Checks, the Control Limits, and the Laboratory Corrective Actions.

Table 11-1 Internal Laboratory Quality Control Checks

Parameter	QC Check	Frequencies	Control Limits	Laboratory Corrective Actions
VOCs	Method blanks	One per 12 hour analytical shift of a similar matrix (24 hours for method TO-15)	No target analytes detected above PQL for all compounds but methylene chloride, acetone, 2-butanone <5X PQL.	Check analytical system, reanalysis
	Surrogate spikes	Every sample, blank, standard	Laboratory control limits*; must be greater than 10%	Reanalysis; if still out of control limits, then report both sets of data
	MS/MSD samples	One per 20 field samples, of a similar matrix (not applicable to method TO-15)	Laboratory control limits*; must be greater than 10%	Report results
	LCS	One per 20 field samples, of a similar matrix	Laboratory control limits*; must be greater than 10%	Reanalysis; if still out, re-preparation/reanalysis of entire batch
	IS areas	Every sample, blank, standard	-50% to +100% of associated continuing calibration Standard (-60% to +140% for method TO-15)	Reanalysis; if still out of control limits, then report both sets of data
	GC/MS mass tuning	At beginning of each 12 hour analytical shift (24 hour shift for method TO-15)	Control criteria listed in method	Recalibrate instrument until control criteria are met
SVOCs	Method blanks	One per 20 field samples or each extraction batch	No target analytes detected above PQL for all compounds but phthalates <5X PQL.	Reanalysis; if still out of control limits, then re-extract entire batch
	Surrogate spikes	Every sample, blank, standard	Laboratory control limits*; must be greater than 10%	Reanalysis; if still out of control limits, then report both sets of data
	MS/MSD samples	One per 20 field samples, of a similar matrix	Laboratory control limits*; must be greater than 10%	Report results
	LCS	One per 20 field samples, of a similar matrix	Laboratory control limits*; must be greater than 10%	Reanalysis; if still out, re-preparation/reanalysis of entire batch
	IS areas	Every sample, blank, standard	-50% to +100% of associated continuing calibration standard	Reanalysis; if still out of control limits, then report both sets of data
	GC/MS mass tuning	At beginning of each 12 hour analytical shift	Control criteria listed in method	Recalibrate instrument until control criteria are met
PCBs	Method Blanks	One per 20 field samples or each extraction batch	All compounds <PQL	Reanalysis; if still out of control emits then re-extract entire batch
	Surrogate Spikes	Every sample, blank, standard	Laboratory control limits*; must be greater than 10%	Reanalysis; if still out of control limits, then report both sets of data
	MS/MSD Samples	One per 20 field samples of a similar matrix	Laboratory control limits*; must be greater than 10%	Report results
	LCS	One per 20 field samples, of a similar matrix	Laboratory control limits*; must be greater than 10%	Reanalysis; if still out, re-preparation/reanalysis of entire batch

Parameter	QC Check	Frequencies	Control Limits	Laboratory Corrective Actions
Metals	Reagent/prep blanks	One per analytical batch	No analytes above PQL	Re-preparation/reanalysis of entire prep batch
	MS samples	One per 20 field samples, per day of similar matrix	75 – 125% recovery.	Check LCS, flag results
	Duplicate samples	One per 20 field samples, per day of similar matrix	20% RPD aqueous; 50% RPD solid.	Check analytical system, flag results
	LCS	One per 20 field samples, per day of similar matrix	80 – 120% recovery.	Reanalysis; if still out, re-preparation/reanalysis of entire batch
	Interference check (Method 6010B)	Beginning of each analytical run	± 20%	Evaluate; reanalysis if necessary
Cyanide	Reagent/prep blanks	One per analytical batch	No analytes above PQL	Re-preparation/reanalysis of entire prep batch
	MS samples	One per 20 field samples, per day of similar matrix	75 – 125% recovery.	Check LCS, flag results
	Duplicate samples	One per 20 field samples, per day of similar matrix	20% RPD aqueous; 50% RPD solid.	Check analytical system, flag results
	LCS	One per 20 field samples, per day of similar matrix	80 – 120% recovery.	Reanalysis; if still out, re-preparation/reanalysis of entire batch

NA = Not Applicable
PCB = Polychlorinated Biphenyls
QC = Quality Control
RPD = Relative Percent Difference
MS/MSD = Matrix Spike/Matrix Spike Duplicate
GC/MS - Gas Chromatograph/Mass Spectrometer
IS = Internal Standard
TPH = Total Petroleum Hydrocarbons
RL = Reporting Limit
LCS = Laboratory Control Sample
[1]Project Specific Criteria
*Laboratory control limits are developed by the laboratory and are statistically derived values based on historical data. A copy of the laboratory's current limits is presented in Attachment W.

12.0 Data management and documentation

12.1 Data reporting

It is essential to the success of any Brownfields site investigation that a data flow or reporting scheme be developed. For any such scheme to be effective, it must address the complete scope of measurement results generated from all facets of an environmental monitoring project including the collection of raw data through the storage of validated results. In addition, it must also completely cover the step-wise procedures for entering data onto various reporting forms, as well as into computer systems. These procedures should always cover routine data transfer and entry validation checks to ensure these processes are complete. To assist in these efforts, pre-printed forms (for example, field data sheets) should always be utilized for transcribing data whenever possible; examples of field data sheets are included in Attachment Q.

12.2 Data formatting

When conducting a Brownfields site investigation there must always be adequate documentation available to enable the summation of all pertinent measurement data. This is necessary to assist in the interpretation of the data while ensuring that it is both scientifically valid and legally defensible. As a result, it is integral that all records be legible, complete, and properly organized. In some instances, it may be appropriate to utilize a document control system. Therefore, when planning a Brownfields site investigation project, one must consider the type of record to be maintained, and the process for how these records will be stored. The following sections discuss types of records, record maintenance, and record storage for various aspects of the project (field data, laboratory data, etc.).

12.3 Field data reporting

All real-time measurements and observations must always be recorded in project log books, field data records, or in similar types of record keeping books. Field measurements may include pH, temperature, specific conductance, turbidity, water flow, soil gas readings, and possibly FID/PID measurements. All measurement data collected by performing in-situ analyses must always be recorded directly and legibly in field logbooks, with all entries being signed and dated. If entries must be changed, it is essential that these changes be made in such a manner that none of the original entries become obscured. Likewise, the reason for making a change should be specified with the correction and explanation being signed and dated at the time the revision was made.

Examples of field data sheets are included as Attachment Q. Field notebooks will be standard, water-resistant field books with numbered pages, and will be labeled with the project name and project number. Original field records will be maintained by the field team, reviewed by the senior Project Geologist, and hard copies stored in project files. In addition, and electronic scans will be made of all field data sheets, logs, and field notes. Electronic copies will be clearly named and stored in project-specific shared network drives within ENSR. Communications and other miscellaneous relevant material will be stored electronically within ENSR. Reports, laboratory data, figures, and tables will be stored electronically and in hard copy in project files. ENSR will submit electronic copies of all documents contained within the RI/RA Report.

12.4 Laboratory data reporting

Whenever laboratory data are acquired, an analytical report should always be prepared to summarize the results of each environmental sample analyzed in accordance with this Site-specific Brownfields SAMP. The laboratory analytical report will comply with NYSDEC Analytical Service Protocol Exhibit B Reporting and Deliverables Requirement presented in Attachment X.

The analytical report will contain information regarding the analytical methods or procedures employed, sample results, QA/QC results, chain of custody documentation, laboratory correspondence, and all accompanying raw data (i.e., all data necessary for calculating percent recoveries be presented along with the analytical results). The laboratory-provided Laboratory Quality Manual (Attachment T) details the laboratory's process for reviewing, maintaining, and filing of internal laboratory documents.

12.5 Data management and documentation requirements

Data collected during the field investigation will be reduced and reviewed by the laboratory QA personnel, and a report on the findings will be tabulated in a standard format. The criteria used to identify and quantify the analytes will be those specified for the applicable methods in the USEPA SW-846 and subsequent updates and in the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. The data package provided by the laboratory will contain all items specified in the USEPA SW-846 appropriate for the analyses to be performed, and be reported in standard format.

The completed copies of the Chain-of-custody records (both external and internal) accompanying each sample from time of initial bottle preparation to completion of analysis shall be attached to the analytical reports.

12.5.1 Data reporting

Two hard copies of the analytical data packages and one electronic copy will be provided by the laboratory to the Project Manager. The laboratory analytical report will comply with NYSDEC Analytical Service Protocol Exhibit B Reporting and Deliverables Requirement presented in Attachment X. The laboratory-provided data will be compared to appropriate guidance values (see Section 14.0), and used to prepare the Site Investigation/Remedial Alternatives report, as described in Section 14.0. If the City or County has proposed a redevelopment plan for the site at the time of report preparation, the proposed end use will be used to determine the cleanup objectives used. Otherwise, the cleanup objectives will be determined based on current use of the surrounding areas, under the assumption that the proposed redevelopment would be integrated with the surrounding community.

12.5.2 Data validation

ENSR will utilize an internal chemist to perform the data validation for this Site-specific Brownfields SAMP. A Data Usability Summary Report (DUSR) will be prepared at the conclusion of the project. The purpose of the DUSR is to determine whether or not the data, as provided by the laboratory, meets project specific DQOs prior to, or instead of, formal data validation. The data package review leading up to production of the DUSR provides a thorough evaluation of analytical data and provides answers to the following questions:

- Is the data package complete as defined under the requirements for the NYSDEC ASP Category B or USEPA CLP deliverables?
- Have all holding times been met?
- Do all the QC data: blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, replicate analyses, laboratory controls and sample data fall within the protocol required limits and specifications?
- Have all the data been generated using established and agreed upon analytical protocols?
- Does an evaluation of the raw data confirm the results provided in the data summary sheets and quality control verification forms?
- Have the correct data qualifiers been used?

The data usability summary report (DUSR) will be prepared in accordance with the New York State Department of Environmental Conservation, Division of Environmental Remediation's, *Guidance for the Development of Data Usability Summary Reports,* (NYSDEC, August 2001).

12.6 Fixed laboratory data deliverable requirements

Two copies of the analytical data packages will be provided by the laboratory to the Project Manager. All laboratory data will comply with NYSDEC Analytical Service Protocol Exhibit B Reporting and Deliverables Requirement presented in Attachment X.

13.0 Assessment and response actions

13.1 Quality assurance requirements

The data collection scheme put forward in this Site-specific Brownfields SAMP encourages the design of a monitoring network that blends in-situ field analytical screening techniques with confirmatory fixed laboratory analyses. Therefore, to ensure data are of an appropriate quality, the following protocols apply whenever duplicate samples are collected to confirm field screening and/or laboratory analyses with limited analytical deliverables:

- When applicable, rinse and trip blanks will be collected and analyzed with all environmental samples.
- QA/QC samples will be collected and as described in Section 11 of this SAMP.
- Protocols for analytical methods, sample containers, data deliverables, preservatives, chain-of-custody forms, matrix spike sample volumes, and shipping requirements are derived from the U.S.EPA Sampler's Guide to the Contract Laboratory Program.

13.2 Definitive Data Requirements

When conducting a Brownfields site investigation, definitive data should always be acquired using rigorous analytical protocols, such as conventional USEPA reference methods. This involves securing the acquisition of data which are media-specific to confirm target analyte identities and concentrations. Conventional analytical methods are known to produce tangible raw data (chromatograms, spectra, digital values, etc.) in the form of paper printouts and/or computer-generated electronic files. In most instances, definitive data can be generated at the site with a field analytical screening technique or at an off-site fixed laboratory by employing the necessary QA/QC protocols. But regardless of what type of determination is utilized, for data to be definitive, an assessment of analytical or total measurement error must be determined. Therefore, the following criteria should always be implemented when performing a site-specific Brownfields investigation:

- Definitive data QA/QC elements.
- Sample documentation (location, date and time collected, batch, etc.).
- Chain of custody for samples analyzed by an off-site laboratory.
- Sampling design approach (systematic, simple or stratified random, judgmental, etc.).
- Initial and continuing calibration.
- Determination and documentation of instrument and method detection limits.
- Analyte(s) identification.
- Analyte(s) quantification.
- QC blanks (trip, method, rinsate).
- Matrix spike recoveries.

13.3 Analytical error

Performing an estimate of analytical error is the process of determining a measure of overall precision for a particular analytical method. To render a determination of analytical error, an appropriate number of duplicate aliquots are taken from at least one thoroughly homogenized sample. These duplicate sample aliquots are then analyzed with standard laboratory QC parameters to calculate and compare method performance criteria (variance, mean, and coefficient of variation).

13.4 Total measurement error

The determination of total measurement error is an estimate of the overall precision of an environmental data acquisition system, from sample collection through analysis. To render a determination of total measurement error, an appropriate number of samples are independently collected from the same location. These collocated samples are then analyzed with standard laboratory QC parameters to calculate and assess measurement error goals (variance, mean, and coefficient of variation). Measurement error goals are acceptance/performance criteria typically established for the purpose of evaluating data quality. To ascertain a thorough assessment of total measurement error, this process should be undertaken for each environmental matrix under investigation and/or repeated for a given media at more than one location.

13.5 Assessment and response actions

13.5.1 Introduction

Quality assurance audits may be performed by the project quality assurance group under the direction and approval of the project Quality Assurance Officer (QAO) (see Section 1.2). These audits will be implemented to evaluate the capability and performance of project and subcontractor personnel, items, activities, and documentation of the measurement system(s). Functioning as an independent body and reporting directly to corporate quality assurance management, the QAO may plan, schedule, and approve system and performance audits based upon procedures customized to the project requirements. At times, the QAO may request additional personnel with specific expertise from company and/or project groups to assist in conducting performance audits. However, these personnel will not have responsibility for the project work associated with the performance audit. These audits are not anticipated to occur during the duration of this project; However, if conditions adverse to quality are detected, or if the Project Manager requests, audits may occur.

13.5.2 System audits

System audits may be performed by the QAO or designated auditors, and encompass a qualitative evaluation of measurement system components to ascertain their appropriate selection and application. In addition, field and laboratory quality control procedures and associated documentation may be system audited.

13.5.3 Performance audits

The laboratory may be required to conduct an analysis of Performance Evaluation (PE) samples or provide proof that Performance Evaluation samples submitted by USEPA or a state agency have been analyzed within the past twelve months.

13.5.4 Formal audits

Formal audits refer to any system or performance audit that is documented and implemented by the QA group. These audits encompass documented activities performed by qualified lead auditors to a written procedure or checklists to objectively verify that quality assurance requirements have been developed, documented, and instituted in accordance with contractual and project criteria. Formal audits may be performed on project and subcontractor work at various locations.

Audit reports will be written by auditors who have performed the site audit after gathering and evaluating all data. Items, activities, and documents determined by lead auditors to be in noncompliance shall be identified at exit interviews conducted with the involved management. Noncompliances will be logged, and documented through audit findings which are attached to and are a part of the integral audit report. These audit finding forms are directed to management to satisfactorily resolve the noncompliance in a specified and timely manner.

The Project Manager has overall responsibility to ensure that all corrective actions necessary to resolve audit findings are acted upon promptly and satisfactorily. Audit reports must be submitted to the Project Manager within fifteen days of completion of the audit. Serious deficiencies will be reported to the Project Manager within 24 hours. All audit checklists, audit reports, audit findings, and acceptable resolutions are approved by the QAO prior to issue. Verification of acceptable resolutions may be determined by re-audit or documented surveillance of the item or activity. Upon verification acceptance, the QAO will close out the audit report and findings.

14.0 Project reports

14.1 Quality assurance reporting

When conducting a Brownfields site investigation, it is essential to establish mechanisms for providing periodic reports on measurement system performance and data quality to management. These reports should always provide an assessment of measurement data in terms of PARCC, performance audit results, systems audit results, and significant QA problems along with any recommended solutions. In addition, it is prudent that these reports be prepared to include a separate QA section for the purpose of summarizing pertinent information on environmental measurement data quality.

14.2 Roles and responsibilities

To ensure the successful outcome of any Brownfields site investigation project, it is integral for the environmental professional responsible for leading a municipality's remedial efforts to maintain close contact with the U.S.EPA Remedial Project Manager. This is necessary to ensure that pertinent information regarding the technical and financial progress of a site-specific Brownfields investigation is fully understood by all the parties that are involved. Customarily, this communication will begin upon the award of a U.S.EPA Brownfields pilot project grant. This will then necessitate the initiation of QA activities such as the development of project planning documentation.

14.3 Trip reports

To provide a detailed accounting of what occurred during a particular sampling mobilization, trip reports are to be prepared for each site-specific Brownfields investigation. Traditionally, trip reports are to be completed within two weeks of the last day of each sampling mobilization. For the effective use of trip reports, it is important that they provide information in a timely manner by noting major events, dates, and personnel on-site (including affiliations). To facilitate these efforts, trip reports should be assembled as follows:

- Background.
- Observations and Activities.
- Conclusions and Recommendations (optional).
- Future Activities.

14.4 Project report requirements

A Remedial Investigation Report (RI Report) will be prepared summarizing the information generated during implementation of this Work Plan, including tank closures. The report will be prepared in accordance with 6 NYCRR Part 375.4, the NYSDEC Environmental Restoration Projects Procedures Handbook and Draft DER-10 Technical Guidance for Site Investigation and Remediation.

The report will also include the following information and data pertaining to the Site:

- Boring /test pit/field sampling logs.
- Analytical data tables presenting the analytical results for the soil, groundwater, and soil vapor samples including comparisons to appropriate standards, criteria, and guidance (e.g., 6 NYCRR Subpart 375-6 Remedial Program Soil Cleanup Objectives and NYSDEC Groundwater Standards; New York State does not have any standards, criteria, or guidance values for concentrations of volatile chemicals in subsurface vapors).

- A narrative that summarizes the results of the investigation including a discussion of the physical and analytical results.
- A qualitative human health exposure assessment.
- A fish and wildlife resources impact analysis.
- A graphical conceptual model of the Site in part or in whole.
- Figures showing isoconcentrations of GW contamination.
- Spider diagrams (small boxes showing contaminant concentrations with arrows pointing to each sample location) showing the concentrations of contaminants of concern.
- Color (or other shading technique) figures showing soil (or soil vapor) contamination concentrations.

In addition, the analytical data for characterization soil samples, groundwater samples will be reviewed and a Data Usability Summary Report (DUSR) will be prepared. The DUSR will be incorporated as an appendix to the Remedial Investigation Report.

15.0 Verification of sampling procedures

15.1 Performance and system audits

When conducting a Brownfields site investigation it is integral to perform internal, as well as, external performance and systems audits. These audits are undertaken to evaluate the capability and performance of the total measurement system comprising a Brownfields environmental monitoring network. These oversight activities are useful in ensuring that field activities are providing samples reflective of the site and its conditions.

To evaluate the accuracy of the total measurement system or component thereof, performance audits are usually undertaken periodically to assess data collection efforts. In regard to field sampling operations, this oversight function is performed to critique in-situ monitoring efforts and sample collection activities. However, for performance audits to be effective, they should be scheduled in accordance with the applicable field operations warranting oversight. Alternately, a systems audit focuses on evaluating the principal components of a measurement system to determine proper selection and use. In regard to field sampling operations, this oversight activity is performed to critique the quality control procedures which are to be employed. Systems audits of this nature are to be performed periodically, prior to or shortly after, field operations commence until the project is completed.

15.2 Verification of sampling procedures

Reviews of the sampling activities will be conducted by the Site Supervisor or their designated substitute. The intent of these reviews will be to verify that all established procedures that are documented in this Site-specific Brownfields SAMP are followed. Reviews will be conducted at the beginning and at the midpoint of site activities. Each review will include an examination of field sampling records, field instrument operating records, sample collection frequencies and techniques, maintenance of QA procedures, and chain-of-custody documentation. The reviews will be documented in a field notebook dedicated to this purpose for easy reference during data validation. If corrective action is required, a follow-up review will be performed to document the corrective action taken. The follow-up review will also be recorded in the field notebook.

16.0 Data verification and validation

16.1 Data validation

To ensure that the measurement data acquired when performing a Brownfields site investigation are of an appropriate quality, it is important to specify and follow procedures for validating all pertinent environmental monitoring results. Data validation is regarded as a systematic process for reviewing a body of results against a set of established criteria to provide a specified level of assurance concerning validity. It requires a systematic and uniform evaluation to be performed on the data to identify those results with questionable quantitative value.

The approach for performing data validation should always be independent of the data production effort, and objective in its application. In most instances, the criteria for validating data will include conducting checks for internal consistency, reviews for transmittal errors, and/or audits for verifying laboratory capability. This will typically involve interpreting the results of external performance audits such as split sample, duplicate sample (field and laboratory), spiked sample, and initial calibration determinations. In conjunction, the assessment of detection limit studies, intra-laboratory comparisons, inter-laboratory comparisons, tests for normality, tests for outliers, and data base entry checks may also be undertaken.

16.2 Data verification and validation requirements

Section 12.0 discusses project-specific data validation. Additional steps to verify data quality will be:

- Each data package received from the selected laboratory will be reviewed upon receipt for completeness, correctness, and contractual compliance.
- The QC package received from the laboratory will be reviewed to verify that it includes all required elements.
- The data will be reviewed to verify that the requirements of the site assessment have been met, including assessing any exceedances of relevant standards, criteria, and guidance.
- ENSR will subcontract with a selected data validator to perform data validation for the SAMP.
- All field screening and laboratory data will be tabulated and located on a site map to verify that the results are consistent and reasonable.
- ENSR will verify that a minimum of 90 percent of the laboratory analyzed samples were validated and deemed acceptable by the laboratory.

16.3 Fixed-laboratory confirmatory data verification and validation requirements

ENSR will utilize an internal Senior Project Chemist to perform the data validation for this Site-specific Brownfields SAMP. The data usability summary report (DUSR) will be prepared in accordance with the New York State Department of Environmental Conservation, Division of Environmental Remediation's, *Guidance for the Development of Data Usability Summary Reports,* (NYSDEC, August 2001).

All laboratory data will comply with NYSDEC Analytical Service Protocol Exhibit B Reporting and Deliverables Requirement presented in Attachment X.

17.0 Data usability

17.1 Data quality assessment

When performing a Brownfields site investigation, it is essential to correlate validated measurement data for reconciliation with the acceptance/performance criteria specified for the project. This will involve rendering a determination to ascertain whether measurement data are of the right type, quality, and quantity required to support environmental decision making efforts. To perform this activity, scientific and statistical procedures must be employed to provide an assessment. The technique for determining if validated measurement results are adequate for their intended use is known as the Data Quality Assessment (DQA) process. The DQA process can provide information to enable a decision maker to draw conclusions about the strength of evidence depicted by a set of collected measurement data. To assist in these efforts, an outline of the formal DQA process is described in the *U.S.EPA Guidance for Data Quality Assessment: Practical Methods for Data Analysis.*

17.2 Data quality assessment process

The DQA process is both a scientific and statistical evaluation technique which consists of the following five steps:

- Review project acceptance/performance criteria and sampling design.
- Conduct a preliminary data review.
- Select a statistical test (i.e., Shaprio-Wilk W test, Student's t-Test, etc.).
- Verify the assumptions of the selected statistical test.
- Draw conclusions from the data.

Even if the formal DQA process is not followed in its entirety, a systematic assessment of measurement data quality should always be performed when conducting a Brownfields site investigation. This systematic process will involve carrying out the following data assessments:

- Validating all pertinent measurement data for scientific anomalies.
- Correlating all pertinent measurement data to the PARCC parameters designated for the project.
- Identifying measurement data trends and outliers.

In doing so, one can assimilate an abstract estimation of data "worth" to provide Brownfields stakeholders with a rationale for making proper decisions.

17.3 Data usability/reconciliation requirements

All of the field screening results and laboratory data will be included in the final Remedial Investigation Report. Any questions on the usability of the data that come to light in the data review will be described in the report. The conclusions and recommendations made in the report will be qualified if there are uncertainties about the validity of the sampling results. All laboratory data will be compared to the relevant standards, criteria, and guidance. The report will include a discussion of the Data Usability Summary Report generated during data validation.

18.0 References

USEPA, 1986. SW-846 "Test Method for Evaluating Solid Waste," dated November 1986. U.S. Environmental Protection Agency, Washington, D.C.

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USEPA, 1999. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second edition, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Cincinnati, OH, January 1992.

NYSDOH, 2006 Guidance for Evaluating Soil Vapor Intrusion in the State of New York, New York State Department of Health, Center for Environmental Health, Bureau of Environmental Exposure Investigation, October 2006

Attachment A

Contract Laboratory Program Guidance for Field Samplers

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EPA 540-R-07-06

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Office of Superfund Remediation and Technology Innovation



Contract Laboratory Program Guidance for Field Samplers

Disclaimer: The final version of the document replaces any prior versions of the document in their entirety.

Foreword

The intent of the Contract Laboratory Program (CLP) Guidance for Field Samplers is to replace the CLP Samplers Guide. This guidance document is designed to provide users with general information regarding environmental sample collection for the United States Environmental Protection Agency's (USEPA) Contract Laboratory Program (CLP). This document provides minimum CLP requirements, an explanation of the general sampling process sequence of events, and any related information. The appendices contain useful reference information and checklists to aid in planning and documenting sampling activities.

CLP users also are encouraged to review the Introduction to the Contract Laboratory Program document that contains a general overview of the CLP, how it works, and how to access the program. The CLP requires samplers to use the functionality provided by the Field Operations Records Management System (FORMS) II Lite™ software, which is the preferred means of creating CLP sample documentation. For guidance in using the software to record and submit sampling data, users should reference the FORMS II Lite User's Guide.

Both the Introduction to the Contract Laboratory Program and the Contract Laboratory Program Guidance for Field Samplers can be downloaded from the CLP Web site at the following address:

http://www.epa.gov/superfund/programs/clp/guidance.htm

The FORMS II Lite User's Guide can be downloaded from the CLP Web site at the following address:

http://dyncsdao1.fedcsc.com/itg/forms2lite/doc.html

For more information regarding the CLP or this guide, please contact Elizabeth Holman via email at Holman.Elizabeth@epa.gov or via telephone at (703) 603-8761.

Key Information

Text in blue and underlined indicates an external link to information outside of this document.

The images below are located throughout the document to draw attention to important information and each are labeled accordingly:



Important



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ASB	Analytical Services Branch
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
CLP PO	CLP Project Officer
CRQL	Contract Required Quantitation Limit
CVAA	Cold Vapor Atomic Absorption
DOT	Department of Transportation
DQO	Data Quality Objective
dbf	Database File
ET	Eastern Time
FORMS II Lite™	Field Operations Records Management System II Lite
FSP	Field Sampling Plan
HCN	Hydrocyanic acid
IATA	International Air Transport Association
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
MS	Matrix Spike
MSD	Matrix Spike Duplicate
$NAHSO_4$	Sodium Bisulfate
NPL	National Priorities List
OSC	On-scene/on-site Coordinator
OSHA	Occupational Safety and Health Administration
OSRTI	Office of Superfund Remediation and Technology Innovation
OSWER	Office of Solid Waste and Emergency Response
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation
PM	Program Manager
ppb	Parts-Per-Billion
ppt	Parts-Per-Trillion
PRP	Potentially Responsible Party
PT	Proficiency Testing
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QASPER	Quality Assurance Sampling Plan for Environmental Response
QATS	Quality Assurance Technical Support
QC	Quality Control
RAS	Routine Analytical Services
RPM	Remedial Project Manager
RSCC	Regional Sample Control Center Coordinator
RSM	Regional Site Manager
SAM	Site Assessment Manager
SAP	Sampling Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SDG	Sample Delivery Group
SMC	System Monitoring Compound
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SVOA	Semivolatile Organic Analyte
TR/COC	Traffic Report/Chain of Custody
txt	Text File
UN	United Nations
USEPA	United States Environmental Protection Agency
VOA	Volatile Organic Analyte
XML	eXtensible Markup Language

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1.0 INTRODUCTION

1.1 About this Guide

This document describes the important organizational roles and responsibilities for those who plan and conduct environmental sample collection projects for analysis through the Superfund's Contract Laboratory Program (CLP). This chapter introduces the structure and purpose of this document. Chapter 2, *Pre-field Activities*, addresses pre-field planning activities that the sampling team could complete prior to the actual sampling event. Chapter 3, *In-field Activities*, addresses those activities that need to be completed during the sampling event.

Appendix A describes the functions within a sampling project which are taken from the Quality Assurance Project Plan requirements. Appendix B and Appendix C contain the sample collection guidelines for Volatile Organic Analytes (VOAs) in soil and in water. Appendix D recommends sampling techniques. Appendix E contains checklists to help the sampler ensure that all necessary steps are completed.



A project and site-specific Quality Assurance Project Plan (QAPP) providing Regional guidance will override guidance given within this document.

1.2 Overview of the CLP

The CLP is a national program of commercial laboratories under contract to support the USEPA's nationwide effort to clean up designated hazardous waste sites by supporting its Superfund program. The Superfund program was originally established under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 and presently exists under the Superfund Amendments and Reauthorization Act (SARA) of 1986.

The CLP uses state-of-the-art technology to provide users with analytical services. The program provides data of known and documented quality to support USEPA enforcement activities or other user needs. To achieve this goal, the CLP has established strict Quality Control (QC) procedures and detailed documentation requirements. Current CLP users include the USEPA Regions, States and Tribal governments, and other Federal agencies. CLP users also are encouraged to review the *Introduction to the Contract Laboratory Program* document that contains a general overview of the CLP, how it works, and how to access the program.

1.2.1 Key Players Within the CLP

In coordinating Superfund sampling efforts, the Analytical Services Branch (ASB) is supported by the Sample Management Office (SMO) contractor, the Regional CLP Project Officers (CLP POs), the Regional Sample Control Center Coordinators (RSCCs), and the Regional Site Managers (RSMs), including Site Assessment Managers (SAMs), On-scene/On-site Coordinators (OSCs), and Remedial Project Managers (RPMs). Samplers may work directly with the RSCC and/or RSM (or equivalent), and/or an OSC from the Field Support Section during a sampling event. See Table 1-1 for a brief description of the functions performed by key participants (functions may vary by Region).

Table 1-1. Participants in the CLP Sampling Process

Participants	Responsibilities
Analytical Services Branch	USEPA ASB directs the CLP from within the Office of Superfund Remediation and Technology Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER). ASB responsibilities include: • Development of the Statements of Work (SOWs) that define required analytical methods (including QC, detection/quantitation limits, and holding times) for the analytical services procured under the CLP; • Development and implementation of policies and budgets for Superfund analytical operations; • Development of information management policies and products for analytical data; • Management of SMO and Quality Assurance Technical Support (QATS) contracts; • National administration, evaluation, and management of the CLP; and • Direction of CLP Quality Assurance (QA) activities in coordination with overall OSWER QA activities. To obtain the most current ASB contact list, refer to the following Web site: http://www.epa.gov/superfund/programs/clp/contacts.htm#ASB
CLP Sample Management Office	The contractor-operated SMO provides necessary management, operations, and administrative support to the CLP. SMO receives Regional analytical requests, coordinates and schedules sample analyses, and tracks sample shipments. SMO also receives and checks data for completeness and compliance, processes laboratory invoices, and maintains a repository of sampling records and program data.
CLP Contract Laboratories	The contractor-operated laboratories within CLP provide necessary analytical services for the isolation, detection, and quantitation of the CLP's target compounds and analytes.
Regional CLP Project Officer	The CLP PO monitors the technical performance of the contract laboratories in each Region. The CLP PO works closely with ASB Program Managers (PMs) to identify and resolve laboratory technical issues, and leads laboratory on-site evaluations. To obtain the most current CLP PO contact list, refer to the following Web site: http://www.epa.gov/superfund/programs/clp/polist.htm
Regional Sample Control Center Coordinator	In most Regions, the RSCC coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. The RSCC works with SMO to schedule sample shipments to laboratories. In addition, the RSCC's activities may include: informing SMO of sample shipment, cancellations, special instructions, and sampling issues. To obtain the most current RSCC contact list, refer to the following Web site: http://www.epa.gov/superfund/programs/clp/rscclist.htm
Regional Site Manager	The RSM Coordinates the development of acceptance or performance criteria and oversees project-specific contractors, state officials, or private parties conducting site sampling efforts. The RSM could be the SAM, the OSC, or the Remedial Project Manager (RPM).
Field Support Section	The Field Support Section consists of personnel such as the OSC, SAM, and RPM. In most Regions, the Field Support Section develops Standard Operating Procedures (SOPs) for field sampling and related procedures, and assists sampling teams in following those SOPs. The sampling team determines what type(s) of CLP services will be required for a particular sampling event. The Field Support Section reviews Sampling Analysis Plans (SAPs) prepared by sampling teams and oversees sampling teams in the field. The Field Support Section may also prepare their own SAPs, perform sampling activities in the field, and analyze and report the results of their sampling events to the RSM.

1.3 Overview of the Sampling Process

At–a-Glance:
Overview of the Sampling Process

- ✓ Procedures must be consistent.
- ✓ Analytical data must be accurate and defensible.
- ✓ Procedures must meet minimum requirements.

Once USEPA has determined that physical, chemical, and/or biological testing of a site is necessary, samples of material from the site area must be collected. The type of material that must be collected and the analytical method to be used depends upon the physical location of the site, detection level(s), site history (previous sampling), and known or unknown conditions and contaminants. The sampling process includes carefully planned and consistently applied procedures that produce accurate and legally defensible data. The sampling team should consider the procedures and plans presented in this guide as minimum sampling process guidelines to maintain sample integrity and identity. Samples should be collected according to the approved project and site-specific QAPP and SAP. This document does not define specific sampling procedures because specific sampling protocols depend on individual site conditions, Regional requirements, and acceptance and performance criteria. Since Regions may have their own specific requirements for individual sampling programs, they are responsible for generating Region-specific sampling SOPs.

1.3.1 Procedures Must be Consistent

The purpose of sampling is to collect representative portions from a suspected contaminated site. Sample collection is critical to determining the presence, type, concentration, and extent of environmental contamination by hazardous substances, thus it is a crucial part of every sampling and environmental testing effort. Sampling procedures must be consistently written and followed to mitigate risk of error and the expense of re-sampling.

Failure to follow proper sampling and shipping procedures could result in samples that are contaminated, broken, mislabeled, lost during shipping, or unusable because of a missed holding time. If procedures are inconsistently or improperly followed, any resultant analytical data may be inaccurate and may not be defensible in a court of law.



If re-sampling is needed due to improper sampling, the sampling team may incur the cost.

1.3.2 Analytical Data Must be Accurate and Defensible

The data gathered during sampling activities helps to accurately characterize contaminated waste sites so that the impact on human health and the environment can be properly evaluated. Acquiring accurate and defensible data that will be accepted in a court of law is the CLP's primary objective; therefore, the sampler must collect samples according to strict sampling procedures, plans, and guidelines. USEPA and many other Federal agencies use data resulting from analytical testing of soil/sediment/aqueous samples to:

- Determine if a site is contaminated with organic and/or inorganic compounds;
- Identify pollution sources and Potentially Responsible Parties (PRPs);
- Validate remedial design methodologies;
- Assess response and remedial priorities;
- Assess risk to human health and the environment;
- Determine appropriate cleanup actions; and
- Determine cleanup achievements.

1.3.3 Sampling Procedures and Guidelines Must Meet Minimum Requirements

It is imperative that samplers be aware of the minimum CLP and Regional requirements that directly impact and define how a sampling event will take place. It is important to note that the procedures and guidelines set forth in this document are considered minimum CLP requirements. Samplers should reference the following sections within this document that specifically address important requirements that must be met for a successful sampling event:

- Section 1.4.1 CLP Documentation Requirements;
- Section 2.4.1 Request Scheduling of Analysis, SMO-assigned Case Numbers, CLP Sample Numbers, and Laboratory Contact Information;
- Section 2.7 Comply with Transportation and Shipping Requirements;
- Section 2.8 Provide Shipment Notification;
- Section 3.1 Collect Samples; and
- Section 3.2 Complete Documentation.

1.4 Overview of Sampling Documentation Requirements

The sampler must properly document samples collected for analysis in order to uniquely identify each sample and ensure adequate chain-of-custody procedures. When collecting samples, the sampler should always keep in mind that any samples collected may be used in future litigation. This is especially important when samples are from privately owned property. If sampling on privately owned property, samplers should also provide the property owner with a receipt for samples collected and removed from that owner's property. Samplers may also be required by a Region to use a sample label, sample tag, or field operations records documenting information such as daily activities, equipment and materials used, personnel involved, site security, etc. These types of documentation help ensure proper sample identification and provide additional chain-of-custody records.

The documentation required by a Region for a sampling event is outlined in project plans such as the QAPP, SAP, and Field Sampling Plan (FSP).

At–a-Glance:
Overview of the Sampling Document Requirements

- ✓ Must use FORMS II Lite to create sample documentation. Analytical data must be accurate and defensible.
- ✓ CLP documentation requirements:
  - CLP Sample Number
  - SMO-assigned Case Number
  - Traffic Report/Chain of Custody (TR/COC) Record
  - Sample Labels
  - Sample Tags
  - Custody Seals
  - Field Operation Records



Under no circumstances should the site name appear on any documentation that is sent to the laboratory (for the CLP).

1.4.1 CLP Documentation Requirements

Samplers must:

1) Record the CLP Sample Number on each sample bottle;
2) Complete the Traffic Report/Chain of Custody (TR/COC) Record using the FORMS II Lite software, making sure to indicate on the TR/COC Record if the samples require the use of a Modified Analysis;
3) Complete and attach sample labels;
4) Complete and attach sample tags to meet Regional requirements;
5) Complete and attach custody seals to meet Regional requirements; and
6) Complete field operations records, as necessary.

Please contact your RSCC (see Table 1-1) for information regarding CLP Sample Numbers, SMO-assigned Case Numbers, TR/COC Records, and chain-of-custody seals for sampling events.

For information regarding using FORMS II Lite to create and complete a TR/COC Record, refer to the following Web site:

http://www.epa.gov/superfund/programs/clp/f2lite.htm

1.4.1.1 CLP Sample Number

A CLP Sample Number is unique per sampling location and is used to identify and track samples throughout the sampling and analytical processes and is recorded on many types of sampling documentation (e.g., TR/COC Records, sample labels, and sample tags). CLP Sample Numbers are provided to samplers by their RSCC or SMO.

Samplers must contact their RSCC (or their designee) to obtain CLP Sample Numbers for their sampling event. Samplers must correctly assign the CLP Sample Numbers to the appropriate sample bottle or container. Please refer to Section 3.2.1 for more detailed information regarding the use of CLP Sample Numbers.



If the sampler has any questions regarding the assignment of CLP Sample Numbers, they should contact their RSCC.

1.4.1.2 SMO-assigned Case Number

SMO-assigned Case Numbers are used to track groups of samples throughout the sampling and analytical processes and are recorded on many types of sampling documentation (e.g., TR/COC Records, sample labels, and sample tags). Samplers must correctly assign the SMO-assigned Case Number to the appropriate sample bottle or container. To obtain a SMO-assigned Case Number, samplers must contact their RSCC (or their designee).

1.4.1.3 Laboratory Assignment

Samplers are responsible for shipping samples to the appropriate SMO-assigned laboratory for analysis. Samplers must contact their RSCC (or their designee) to obtain their laboratory assignment or they may be provided by SMO.

1.4.1.4 TR/COC Record

The TR/COC Record is used as physical evidence of sample custody and functions as a permanent record of each sample collected.

Per CLP documentation requirements, each cooler must contain a TR/COC Record that lists all the samples contained therein.

In an effort to automate sample documentation in the field, ASB has developed a stand-alone, Windows-based software application that samplers can use to automatically create and generate sample documentation. The FORMS II Lite software allows users to enter information prior to and during sampling events. It allows users to multi-task and electronically create, edit, and print documentation associated with sampling activities. Users can customize data entry screens throughout the entire documentation process. Users can also customize the format and content of sample labels based on specific requirements.

The program simplifies and accelerates the tedious manual sample documentation process by reducing the generation of handwritten documents by almost 70%. The FORMS II Lite software enables samplers to:

- Increment CLP Sample Numbers or manually assign their own unique, project-specific non-CLP Sample Numbers;
- Input the SMO-assigned Case Number into the appropriate field;
- Create sample labels, sample tags, TR/COC Records, Sample Weight forms, and receipts for samples taken from a site;
- Track samples from the field to the laboratory;

- Electronically capture sample information into databases; and
- Export electronic data as a database File (.dbf), Text (.txt), or eXtensible Markup Language (.xml) file.

USEPA requires samplers to use the FORMS II Lite software for all CLP sampling efforts. For assistance with obtaining or using the FORMS II Lite software, please contact the FORMS II Lite Help Desk at 703-818-4200 from 9:00 AM - 5:00 PM Eastern Time (ET). For additional information regarding FORMS II Lite use and training, please refer to the following Web site:

http://www.epa.gov/superfund/programs/clp/f2lite.htm

1.4.1.5 Chain-of-Custody Seals

A chain-of-custody seal is any adhesive label or tape that can be used to seal a sample bottle, container, plastic bag, or shipping cooler such that if it is opened or tampered with, the seal will be broken. Custody seals must be placed on each sample bottle, container, or bag (as appropriate) and each shipping cooler or container. The custody seal is an excellent means of maintaining a record of chain-of-custody, as well as guarding against possible sample contamination or tampering during shipping.

1.4.1.6 Sample Labels

A sample label is a sticker attached to a sample bottle or container that contains a sample. Sample labels are affixed to each sample container as samples are collected in the field or affixed prior to going in the field. A sample label must contain, at a minimum, a CLP Sample Number so that they can be associated with, and listed on, the associated TR/COC Record. The sample label may also include the required analysis/fraction and preservative used (to eliminate confusion at the laboratory). Samplers should refer to their project plans for Region-specific sample label requirements.

1.4.1.7 Sample Tags

A sample tag identifies a sample bottle or container that contains a sample. The tag also provides specific analytical direction and proof that a sample existed. To support the use of sample data in potential enforcement actions, samples with other than in situ measurements (e.g., pH, temperature, conductivity) can be identified with a sample tag. A CLP Sample Number and SMO-assigned Case Number must be recorded on a sample tag to indicate that the sample container comprises the whole sample in the case where there is just one container of sample, or part of the indicated sample in the case of multiple containers of sample. Samplers should refer to their project plans for Region-specific sample tag requirements.

1.4.1.8 Field Operation Records

Samplers should maintain complete, accurate, and legible field operations records as they perform a sampling activity. The following records are included: Field Logbooks; Corrective Action Reports; Sampling Trip Reports; supplemental standardized forms; logs; and records such as maps or photographs that document each step of the work performed in the field. Samplers should refer to their project plans for Region-specific field operations record requirements. These records are very important tools because they are considered part of the official project file when legal issues arise.

1.4.1.9 Weight Logs

A sample weight log identifies the tared, sample and final weights per bottle for VOA samples. In order to support Method 5035 for VOAs, samplers should enter tared and final weights per bottle in the CLP Sample Weight Log.

2.0 PRE-FIELD ACTIVITIES

This chapter provides instructions for completing the suggested pre-field activities that samplers could complete prior to performing sampling activities. These important pre-field activities will save time and help the sampler to better prepare for the sampling event. Samplers should be aware of issues routinely arise during the sampling process so that samplers can avoid making the same mistakes or having the same problems that could adversely affect their sampling event. Samplers are also expected to review all pertinent project plans and meet both CLP and Regional requirements that directly impact the structure and purpose of a sampling event.

At-a-Glance:
Pre-field Activities

- ✓ Prepare for and communicate during a sampling event.
- ✓ Review project plans containing Regional requirements.
- ✓ Plan to meet documentation requirements.
- ✓ Obtain any necessary permits, licenses, and clearances.
- ✓ Identify and obtain sampling materials.
- ✓ Comply with transportation and shipping requirements.
- ✓ Provide shipment notification.
- ✓ Perform Readiness Review/Run-through.

The project plans provide information such as the types and numbers of samples to be collected, the analytical methods to be used based on the desired level of quantitation, and the necessary equipment and supplies. The plans also describe the sampling method which may require different specific sample volumes/masses, containers, preservation, shipping, and handling to maintain the integrity of the samples without degradation or contamination.

In addition to reviewing project plans, samplers should determine if the sampling site is privately or publicly owned and obtain the necessary permission to access the sampling site. If the site is privately owned, samplers should make sure to have receipts for available samples to provide to the owner for all samples collected and removed from their property. Samplers must also prepare to identify and obtain sampling materials, prepare to meet documentation requirements by obtaining and learning to use the required software, comply with transportation and shipping requirements, and perform a readiness review/dry run of the sampling process.

2.1 Prepare for a Sampling Event

Samplers must prepare to meet CLP and Regional requirements for a sampling event, appropriately use the CLP Sample Number and SMO-assigned Case Number, complete the TR/COC Record using the FORMS II Lite software, and complete and attach the custody seal(s). It is very important that the sampler include the correct CLP Sample Number on each sample. It is also imperative that the TR/COC Record be accurately completed and submitted with the sample(s). Finally, the sampler must accurately and legibly complete and attach a custody seal to each sample container, or plastic sample bag (as appropriate), and each shipping cooler or container.

However, meeting the sampling requirements requires more than just the proper application of a CLP Sample Number on each sample, completion of the TR/COC Record, and use of a custody seal. The actual collection of samples, packaging, and shipping of those samples are equally important to a successful sampling event.

For example, if a sampler collects an insufficient volume of a sample, the laboratory may not be able to perform the requested analysis. Insufficient sample volumes may also result in a laboratory being unable to perform laboratory quality control, such as Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Duplicate sample analysis. Additionally, if the laboratory receives a sample that is either unpreserved or the sample pH is outside of the required range, the sample cannot be properly analyzed.

Unfortunately, improper shipping and labeling processes and procedures often result in:

- Samples being shipped to the wrong laboratory;
- Broken or empty samples being received at the laboratory; and
- Custody seals or sealant tape that is missing or broken on sample bottles, containers, plastic bags, or shipping coolers shipped to the laboratories.

The importance of completing the paperwork associated with a sampling event cannot be overemphasized. Samplers must make a conscientious effort to accurately complete the TR/COC Record since this is the main document used to derive vital information about a particular sample. The person completing a TR/COC Record

must be careful to avoid errors such as the appropriate sample(s) not being listed, or the wrong samples being listed. In an effort to eliminate such errors and the confusion that can be associated with handwritten TR/COC Records, samplers must use the FORMS II Lite software to complete the TR/COC Record and other associated sampling documentation.

It is extremely important that QC samples, including field sample duplicates, field samples for Matrix Spike and Matrix Spike Duplicate analyses, and Proficiency Testing (PT) samples, also known as Performance Evaluation (PE) samples, be designated and labeled per Regional guidance by samplers in the field. Mislabeling of QC samples can result in improper and/or inaccurate analysis of a sample at the laboratory.

2.2 Communicate During a Sampling Event

Communication is a key element in planning, administrating, and conducting a sampling event. It is extremely important that all parties involved in a sampling event be in contact throughout the sampling process. The procedures and recommendations outlined in this guide are based on more than 20 years of experience. It has been demonstrated that approximately 50% of all sampling efforts have been negatively affected by incorrect sampling procedures and poor communication among participants.

The key elements of communication for a sampling event include the relationship between the RSCC, SMO, the samplers in the field, and the laboratories who will be accepting the samples. For instance, the samplers must contact the RSCC to start the process for setting up a sampling event. The RSCC will in turn contact SMO who will schedule the sampling event, establish laboratory availability, and arrange for the laboratory to accept projected samples. SMO will then communicate the laboratory assignment to the Region and possibly the sampler.



The sampler should contact the RSCC (per Regional guidelines) and allow enough time for the RSCC to contact SMO at least a week prior to the sampling event.

SMO provides SMO-assigned Case and CLP Sample Numbers in time for the sampling event. SMO also schedules a laboratory and makes sure the laboratory will not have any capacity problems. Communication is also important because if there is a change in the sampling event due to a cancellation or an increase or decrease in the number of samples that will be sent to the laboratory, the sampler can contact the RSCC who can work with SMO to remedy potential capacity, availability, or overbooking problems.

2.3 Review Project Plans Containing Regional Requirements

In addition to meeting CLP requirements, the sample collection process must fulfill numerous Regional requirements. These requirements are determined by a variety of factors that affect how samples should be collected for an individual sampling event. These factors include:

- The type of samples being collected (organic/inorganic, water, soil/sediment, etc.);
- The method by which the samples will be analyzed;
- The acceptance or performance criteria (i.e., Data Quality Objectives [DQOs]); and
- The type of data needed.

The QAPP for each sampling project is written to meet requirements outlined in the documents *EPA Requirements for Quality Assurance Project Plans* (QA/R-5), *EPA Guidance on Quality Assurance Project Plans* (G-5), and Regional QAPP preparation documents. The QAPP is prepared in advance of field activities and is used by samplers to develop any subsequent plans such as the Sampling SAP or the FSP. Samplers should review the QAPP and any subsequent project plans for information outlining the basic components of a sampling activity. QAPP and project plans should be finalized and approved by appropriate Regional QA personnel, the OSC, SAM, or the RPM before providing them to the sampling team. This should be done prior to the start of field activities. Appendix A explains the functions within a sampling project (as these functions relate to a sampling event) and the elements of that function as described in a typical QAPP. Copies of all project plans and relevant SOPs should be maintained in the field for the duration of the sampling project.

2.4 Plan to Meet Documentation Requirements

At-a-Glance:
Plan to meet documentation requirements.

- ✓ Request SMO-assigned Case and CLP Sample Numbers.
- ✓ Prepare sample cooler return documentation.
- ✓ Prepare to use the FORMS II Lite software.

Sampling events require a variety of accurate and complete documentation. Samplers should review their project plans to determine the types of documentation that must be completed for a sampling project and to ensure that the appropriate documentation will be on-hand in the field. The CLP documentation requirements include the CLP Sample Number, the SMO-assigned Case Number, the TR/COC Record, sample labels, sample tags, custody seals, and field operations records (as necessary). Samplers need to request SMO-assigned Case and CLP Sample Numbers for each sampling event prior to starting field activities. Samplers also need to make sure that the correct TR/COC Records (Organic TR/COC Record for organic analysis or Inorganic TR/COC Record for inorganic analysis) are being used within the FORMS II Lite software. Finally, samplers should be prepared to complete the appropriate shipping cooler return documentation.

Since samplers are required to use the FORMS II Lite software to prepare and submit sampling project documentation and maintain sample chain-of-custody, software users must be familiar with all emergency back up procedures that should be followed in the event of a system failure. Samplers must have access to FORMS II Lite-generated TR/COC Records at sampling events. If problems are experienced while using the FORMS II Lite software, please contact the FORMS II Lite Help Desk at 703-818-4200 from 9:00 AM - 5:00 PM ET.

In the event of a system crash, samplers must have backup hardcopies of FORMS II Lite TR/COC Records. For information regarding emergency backup procedures, please refer to the following Web site:

http://www.epa.gov/superfund/programs/clp/trcoc.htm

2.4.1 Request Scheduling of Analysis, SMO-assigned Case Numbers, CLP Sample Numbers, and Laboratory Contact Information

SMO-assigned Case Numbers are assigned based on a request for CLP Routine Analytical Services (RAS), which is processed though the RSCC (or his/her designee). The sampler must request the RSCC to schedule CLP RAS analysis. The CLP does have the capacity to schedule sampling on an emergency basis, however the sampler must contact the RSCC (or his/her designee) to obtain details regarding how to handle such a situation. When scheduling a sampling event that will last for more than one week, it is recommended that the sampler contact the RSCC (or his/her designee) on a weekly basis to provide updates. This contact between the sampler, the RSCC (or his/her designee), and SMO is very important because it will ensure better availability of laboratory capacity.

In addition to SMO-assigned Case and CLP Sample Numbers, samplers should make sure to have accurate laboratory contact information, such as:

- Laboratory name;
- Laboratory address;
- Contact name; and
- Laboratory phone number.

This information is used for both TR/COC Records and chain-of-custody documentation and shipping paperwork such as address labels and airbills.

The SMO-assigned Case Number is used to track groups of samples throughout the sampling and analytical processes. Samplers must correctly indicate the assigned Case Number on the appropriate sample bottle or container.



The RSCC (or his/her designee) provides the CLP Case Numbers and Sample Numbers for each sampling event to samplers. Once the CLP Sample Numbers have been provided to the sampler, the sampler can use FORMS II Lite to print them onto sample labels.

The following characters are not to be used in generating CLP Sample Numbers and should never appear on any paperwork submitted to the laboratory: I, O, U, and V.

A CLP *Sample Number* is defined **as** a number that is unique per sampling location and identifies each CLP sample (see Section 1.4.1.1). Since samples must be identified per analytical program (either organic or inorganic), there are two types of TR/COC Records and two letter codes to denote organic vs. inorganic analysis.

A CLP *sample* is defined as one discrete portion of material to be analyzed that is contained at one concentration level, from one station location for each individual or set of analytical fractions -- provided the fractions are all requested for the same CLP analytical service (i.e., organic or inorganic), and identified by a unique Sample Number.



When samples are collected from several station locations to form a composite sample, the composite sample should be assigned either a number from one of the station locations used during collection, or a unique number that represents the composite sample for tracking purposes. The numbering scheme used internally at a sampling event for identifying composite samples should also be documented appropriately (e.g., in the field logs).

Organic CLP Sample Numbers begin with the Regional letter code, followed by four letters and/or numbers. Inorganic CLP Sample Numbers begin with "M", followed by the Regional letter code and then four letters and/or numbers. See Table 2-1 for Region and letter codes for each sample type (i.e., organic or inorganic).

Table 2-1. CLP Sample Number Letter Codes

Region	Letter Code	
	Organic	Inorganic
1	A	MA
2	B	MB
3	C	MC
4	D	MD
5	E	ME
6	F	MF
7	G	MG
8	H	MH
9	Y	MY
10	J	MJ

According to CLP guidelines, each individual inorganic water sample may be analyzed for total metals or dissolved metals, but not both. Therefore, water samples collected for total metal and dissolved metal analyses from the same sampling location must be assigned separate (unique) CLP Sample Numbers. A sampler can use the same CLP Sample Number for an inorganic soil or water sample collected for total metals, mercury and cyanide analyses.

Organic soil and water samples may be collected for analysis under the SOM01 SOW to detect:

- Aroclors;
- Semivolatile Organic Analytes (SVOAs);
- Pesticides;
- Volatile Organic Analytes (VOAs); and/or
- Trace Volatile Analytes

Inorganic soil and water samples may be collected for analysis for cyanide, and for metals using Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) and Cold Vapor Atomic Absorption (CVAA), under the ILM05.X SOW.

Inorganic water only samples may be collected for analysis for cyanide, and for metals using Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) and CVAA, under the ILM05 SOW.

2.4.2 Prepare Sample Cooler Return Documentation

CLP laboratories must routinely return sample shipping coolers to the appropriate sampling office within 14 calendar days following receipt of shipment from the sampler. For sample coolers to be returned, the

sampler must complete the appropriate cooler documentation and work with Regions and government agencies to provide a cost-effective mechanism for laboratories to return the empty coolers to the appropriate sampling office. The sampling cooler return documentation can be prepared in advance and provided to samplers before field activities begin. **The sampler (not the CLP laboratory) is responsible for paying for return of the cooler and should also include shipping airbills bearing the sampler's account number, as well as a return address to allow for cooler return.**

To maintain consistency among cooler transportation programs, samplers should:

- Minimize the use of multiple transportation carriers to avoid confusion;
- Use multiple-copy labels so the laboratory and the sampling team can each retain a copy for their records;
- Prepare labels in advance so that the laboratory can simply affix a completed shipping label on the cooler;
- Include third-party billing information (i.e., their shipping account number) on labels so the laboratory will not be billed by the transportation carrier;
- Confirm that the laboratory knows which transportation carrier to use; and
- Include the SMO-assigned Case Number on return information.

2.5 Obtain Municipal Permits, Licenses, and Clearances

Before starting a sampling event, samplers must make sure to obtain the proper municipal permits, accesses to the property, and any government clearances, if required. The sampler must also contact any appropriate utility companies to ascertain where any underground pipes, cables, etc., may be located.

At-a-Glance:
Obtain permits, licenses, and clearances.

- ✓ Request access to County, State, Tribal, military, and/or Federal property.
- ✓ Contact private property owner(s).
- ✓ Contact utility companies.

2.5.1 Request Access to County, State, Tribal, Military, and/or Federal Property

Proper access to perform sampling activities is important not only for legal reasons, but also to eliminate delays in work and possible refusal to allow sampling to take place. It is crucial that the appropriate permits, licenses, and clearances be secured to obtain access for sampling activities that will be performed on County, State, Tribal, military, and/or Federal property. The sampler must contact the appropriate government offices or personnel well in advance to determine what kinds of approval are required. Pre-approval may be required for specific types of sample collection such as drilling or excavation. For example, drilling on a military base requires pre-approval. Base security may require clearances for all members of the sampling team, including subcontractors. This process may take two or more days.

If arrangements are not made in advance, the team may not be allowed to enter the site until their clearances are processed and the team has been approved to drill. As a result, the sampling schedule is delayed, costing extra time and money.

2.5.2 Contact Private Property Owners

The sampler must obtain written permission from the private property owner(s) before sampling on their property, even if verbal permission has been granted. It is recommended that samplers obtain verbal permission prior to their arrival at the sampling location, but written permission can be obtained on the day of sampling. If a property owner refuses to grant access to their property, it may be necessary for sampling participants to contact the appropriate authorities for assistance.

2.5.3 Contact Utility Companies

The sampler should contact local utility companies (e.g., power, phone, gas, cable, sanitation, etc.) at least one week prior to the sampling event to have underground cables, lines, and pipes flagged and marked. This is required by law. A national one-call directory can be found at:

http://www.digsafely.com/contacts.htm.

This will eliminate potential safety hazards and service disruption. For example, soil sampling in a residential area may require digging below the soil's surface. It is very important to know where utility lines and pipes are located so that samplers do not hit live electrical wires or rupture gas lines. Samplers should follow Regional or other appropriate program procedures for the procurement of such services. The utility service(s) disruption dates should be confirmed at least two days prior to sampling activities.



Pre-payment of survey fees to local utility companies may be required.

2.6 Identify and Obtain Sampling Materials

Samplers must make sure to be prepared for a sampling project with the appropriate sampling materials (equipment, supplies, sample containers, packing materials, and shipping materials). The equipment and supplies must be properly cleaned, calibrated, and tested as necessary to meet the needs of the sampling project.

At-a-Glance:
Identify and obtain sampling materials.

- ✓ Procure appropriate equipment and supplies.
- ✓ Procure sample containers.
- ✓ Procure shipping supplies.

2.6.1 Procure Appropriate Equipment and Supplies

Each sampling event requires the procurement of equipment and materials to collect, document, identify, pack, and ship samples. The proper field sampling equipment is vital to a successful sample collection. Regional or other samplers should obtain, and arrange in advance, all of the equipment and supplies required for each sampling event. Samplers should review the project plans to verify that the proper equipment is being used for sample collection.

At a minimum, the following materials are generally required during a sampling event:

- Sample storage containers;
- Packing material;
- Sample containers;
- Shipping containers;
- Access to the FORMS II Lite software for creating sample labels, stickers, tags, and TR/COC Records;
- Custody seals; and
- Sampling equipment such as bowls, augers, pumps, etc.

Sampling events may also require specific items such as:

- Cooler temperature blanks;
- Trip blanks for VOA analysis;
- Preservation supplies (e.g., ice or acid); and
- Specially prepared sample vials (e.g., for SW-846 Method 5035A).

2.6.2 Procure Sample Containers

The analytical protocol(s) to be used for sample analysis often requires the use of a particular type of sample container. The type of container also may depend on the sample matrix and analysis. It is recommended that samplers use borosilicate glass containers, which are inert to most materials, when sampling for pesticides and/or other organics. Conventional polyethylene is recommended when sampling for metals because of the lower cost and absorption rate of metal ions.

Using the wrong container may result in breakage, gathering of an insufficient volume needed to perform sample analysis, or the container material may interfere with the analysis. Therefore, samplers should identify and use the correct sample containers for each sampling event.

Containers procured for a sampling event are usually pre-cleaned and shipped ready-for-use from the manufacturer to the sampling site. Regardless of the type of container used, samplers must ensure that the containers have been analyzed or certified clean to levels below concern for the project. These containers must meet the USEPA container type specifications listed in Table 2-2.

Table 2-2. Container Type Specifications

Reference Number	Container Type	Specifications	
		Closure	Septum
1	40 mL amber glass vial, 24 mm neck finish.	Polypropylene or phenolic, open-top screw-cap, 15 cm opening, 24-400 size.	24 mm disc of 0.005 in. Polytetrafluoroethylene (PTFE) bonded to 0.120 in. silicone for total a thickness of 0.125 in.
2	1 L high density polyethylene, cylinder-round bottle, 28 mm neck finish.	Polyethylene cap, ribbed, 28-410 size; F217 polyethylene liner.	N/A
3	8 oz short, wide mouth, straight-sided, glass jar, 70 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner.	N/A
4	4 oz (120 mL) tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.	Polypropylene or phenolic cap, 48-400 size; 0.015 in. PTFE liner.	N/A
5	1 L amber round glass bottle, 33 mm pour-out neck finish.	Polypropylene or phenolic cap, 33-430 size; 0.015 in. PTFE liner.	N/A
6	500 mL high density polyethylene, cylinder-round bottle, 28 mm neck finish.	Polypropylene cap, ribbed, 28-410 size; F217 polyethylene liner.	N/A
7	Coring tool used as a transport device (e.g., 5 g Sampler).	Has built-in closing mechanism.	N/A
8	250 mL high density polyethylene, cylinder-round bottle, 28 mm neck finish.		N/A

The information contained in this table is also cross-referenced in the sample collection parameters discussed in Chapter 3. The container Reference Numbers are used in Tables 3-2 and 3-3 under the Containers column. For example, samples collected for low-level soil VOA analysis using SW-846 Method 5035A may require the sampler to use pre-prepared, tared closed-system purge-and-trap vials with a preservative (refer to Appendix B).



Have extra containers readily available for each sampling event in case of breakage, loss, or contamination.

2.6.3 Procure Shipping Supplies

Samples should be correctly packaged into the appropriate shipping containers to reduce the risk of breakage or leakage, and the shipping containers should be appropriately prepared for shipment. Before heading into the field, samplers should refer to the appropriate project plans to determine the types of samples that will be taken during the sampling project so that samplers will have the proper packaging materials at the site for all pertinent samples container types and sample matrices. Samplers should also make sure to obtain the appropriate shipping paperwork (e.g., shipping forms required by the delivery service).

2.7 Comply with Transportation and Shipping Requirements

Samplers are expected to review the applicable project plans to be aware of all State, Federal, Department of Transportation (DOT), and International Air Transport Association (IATA) regulations governing environmental and hazardous sample packaging. The person who ships the samples is responsible for being in compliance with applicable packaging, labeling, and shipping requirements.



Samplers should request and receive sample permits for outside the continental United States, prior to shipping.

Additional information can be obtained on Hazardous Materials Safety Program regulations from the DOT's Research and Special Programs Administration. Federal transportation regulations can be found in 49-CFR Parts 100-185, are available on the Internet at:

http://www.myregs.com/dotrspa/

2.8 Provide Shipment Notification

Some Regions may require that samplers notify their RSCC (or his/her designee) when samples are shipped. Some Regions allow samplers to contact SMO directly to provide shipment notification. It is recommended that samplers contact the RSCC of sample origin to verify if such notification is necessary. If samplers are shipping samples after 5:00 PM ET, samplers must notify the RSCC (or designee) or SMO by 8:00 AM ET on the following business day.



For Saturday delivery at the laboratory, samplers MUST contact the RSCC (or designee) or SMO so that SMO will receive the delivery information by 3:00 PM ET on the Friday prior to delivery.

2.9 Perform Readiness Review/Dry Run

A readiness review/dry run is a test run of the proposed sampling event. This is a recommended practice since it gives samplers a chance to check all plans, documentation software (i.e., FORMS II Lite), and equipment lists for accuracy and completeness prior to sampling activities. It also provides an opportunity to consult with sampling team members to make sure all the elements are in place and everyone understands their tasking before actually going out to the field. Sampling project managers should provide the test or dry run dates and schedules to samplers so that samplers can prepare accordingly.

3.0 IN-FIELD ACTIVITIES

This chapter addresses the in-field activities a sampler will focus on during a sampling event such as: determining the type of samples to be collected; collecting the samples; meeting volume, preservation, and holding time requirements; completing documentation; and packing and shipping samples.

When performing a sampling event, the sampler is expected to follow prescribed sampling techniques. The sampler should also be aware of any special sampling considerations, contamination issues, and sample compositing and mixing methods that could affect their sampling efforts. Please refer to Appendix D for more detailed information.

At-a-Glance:
In-field Activities

- ✓ Collecting samples
- ✓ Completing documentation
- ✓ Sampling considerations
- ✓ Procuring shipping supplies



Appropriate Regional guidance and procedures should be consulted for detailed sample collection, preservation, handling and storing, equipment decontamination, and QA/QC procedures.

3.1 Collect Samples

CLP RAS are generally used to analyze samples from Superfund sites. The matrices can be water, soil, or sediment. In some instances, a mixed-matrix sample may be collected which contains either a supernate (for a sediment/soil sample) or a precipitate (for a water sample). In this event, samplers should consult their management plans and/or discuss the required procedures with the RSM or their designee.

A CLP sample consists of all sample aliquots (portions):

- for each individual or set of analytical fractions;
- from one station location;
- for one sample matrix;
- at one concentration level;
- for one laboratory; and
- for one analytical program;

provided that the fractions are all requested from the same CLP analytical service.

In general, it is recommended that two individual samples be collected by separating the aqueous layer from the solid/precipitate layer at the point of collection. They may be assigned two different sample IDs (e.g., Sample IDs ABC124 and ABC125 for Sample ID ABC123), along with a note in the field sample log or tracking system that the sample IDs are derived or related to the same sample ID, to ensure correct follow-up upon receipt of results from the laboratory. Alternatively, they may be assigned the same sample ID, along with a notation of each individual sub-sample or fraction (e.g., Sample IDs ABC123-1 and ABC123-2 or Sample ID ABC123 Fraction 1 and Sample ID ABC123 Fraction 2 for Sample ID ABC123).

3.1.1 Determine Types of Samples to be Collected

Samplers may be required to take several types of samples or sample aliquots during a sampling event. They should refer to their project plans to determine the types of samples or aliquots to be taken, the volumes needed of each sample or aliquot, and the preservation needed for each sample. For an explanation of the various sample types and the requirements for collecting and submitting each particular type, refer to Table 3-1.

Table 3-1. QC Sample Types and CLP Submission Requirements

Sample Type	Purpose	Collection[1]	CLP Sample Number
Field Duplicate	To check reproducibility of laboratory and field procedures. To indicate non-homogeneity.	Collect from areas that are known or suspected to be contaminated. Collect one sample per week or 10% (Regions may vary) of all field samples per matrix, whichever is greater.	Assign two separate (unique) CLP Sample Numbers (i.e., one number to the field sample and one to the duplicate). Submit blind to the laboratory.
Field Blanks	To check cross-contamination during sample collection, preservation, and shipment, as well as in the laboratory. Also to check sample containers and preservatives.	Collect for each group of samples of similar matrix per day of sampling. Organics - Use water (demonstrated to be free of the contaminants of concern). Inorganics - Use metal-free (deionized or distilled) water.	Assign separate CLP Sample Numbers to the field blanks.
Trip Blank (Volatile Organic Analysis Only)	To check contamination of VOA samples during handling, storage, and shipment from field to laboratory.	Prior to going into the field, prepare and seal one sample per shipment per matrix using water demonstrated to be free of the contaminants of concern (deionized water is appropriate). Place this sample in the cooler used to ship VOA samples.	Assign separate CLP Sample Numbers to the trip blanks.
Equipment Blank or Rinsate Blank	To check field decontamination procedures.	Collect when sampling equipment is decontaminated and reused in the field or when a sample collection vessel (bailer or beaker) will be used. Use blank water (water demonstrated to be organic-free, deionized or distilled for inorganics) to rinse water into the sample containers.	Assign separate CLP Sample Numbers to the equipment blanks.
Matrix Spike (MS) and Duplicate (MSD)[2] (Organic Analysis Only)	To check accuracy and precision of organic analyses in specific sample matrices.	Collect from areas that are known or suspected to be contaminated. For smaller sampling events (i.e., 20 samples or less), MS/MSD additional volume should be collected in the first round of sampling and included in the first shipment of samples to the laboratory. Collect double or triple volume[3] for aqueous samples and soil VOA samples designated for MS/MSD analyses. Additional sample volume is not required for soil samples requiring SVOA, Pesticide, and/or Aroclor analysis. See Appendix B for VOA collection volumes.	Assign the same CLP Sample Number to the field sample and the extra volume for MS/MSD. Identify the sample designated for MS/MSD on the TR/COC Record.
Matrix Spike (MS) and Duplicate (MSD) (Inorganic Analysis Only)	To check accuracy and precision of inorganic analyses in specific sample matrices.	Collect from areas that are known or suspected to be contaminated. For smaller sampling events (i.e., 20 samples or less), Matrix Spike and Duplicates should be collected in the first round of sampling and included in the first shipment of samples to the laboratory. Additional sample volume may be required for inorganic analysis.[4]	Assign the same CLP Sample Number to the field sample and extra volume (if collected). Identify the sample(s) designated for Matrix Spike and Duplicates on the TR/COC Record.
PE Samples	Specially-prepared QC samples used to evaluate a laboratory's analytical proficiency.	The PE samples contain analytes with concentrations unknown to the laboratory. Designated Regional or authorized personnel (depending on Regional policy) arrange for Case-specific CLP PE samples to be prepared and shipped by the QATS contractor. The PE samples can be shipped to the site, or shipped per Regional direction. QATS provides the appropriate preparation instructions and chain-of-custody materials.	Samplers have no direct interaction with the PE sampling process, but should be aware that such samples do exist within the CLP sampling process. Samplers must, however, order PE samples and ship them to the laboratory if required by the Region.

[1] Consult Regional or Project Manager Guidance for field QC sample frequencies; laboratory QC sample frequencies are generally fixed in the laboratory subcontracts or specified in analytical methods. Current frequency for MS/MSD (organic) and MS/duplicate (inorganic) for the CLP is one sample per twenty field sample of similar matrix.

[2] Samples sent under the Organic SOW (SOM01) do not require an MS or MSD for Trace VOA, VOA and BNA fractions, but the Region may opt to send them at their discretion.

[3] Example of double volume: An aqueous sample for SVOA analysis would require the field sampler to collect at least 2 L of field sample and at least 1 L each for the MS and MSD samples for a total volume of 4 L. If Pesticide or Aroclor MS/MSD analyses are required for the same sample, an additional 4 L must be collected. Double volume is the MINIMUM allowable volume for samples designated for MS/MSD analysis. Triple volume may be sent for MS/MSD samples to allow for sufficient volume for these analyses in the event sample volume is lost as a result of samples breaking, leaking, or laboratory accidents.

[4] Double volume may be sent for inorganic aqueous MS and MSD samples to allow for sufficient volume for these analyses in the event sample volume is lost as a result of samples breaking, leaking or laboratory accidents.

3.1.1.1 Collect Field QC Samples

Samplers can collect field QC samples and laboratory QC samples to verify that sample quality is maintained during a sampling project.

Field QC samples are designed to assess variability of the media being sampled and to detect contamination and sampling error in the field. The types of field QC samples that are generally collected include field duplicates and field blanks (such as equipment, trip, or rinse blanks). Generally, field duplicate samples should remain "blind" to the laboratory (i.e., they should have separate CLP Sample Numbers).

3.1.1.2 Collect Laboratory QC Samples

A laboratory QC sample is an additional analysis of a field sample, as required by the laboratory's contract. There are three types of laboratory QC samples:

- MS [for organic and inorganic samples];
- MSD [for organic samples only]; and
- Duplicates [for inorganic samples only].



Samplers should obtain Regional guidance regarding the collection of MS and MSD samples (especially for organics analyses).

Samplers should select one sample per matrix per 20 samples as a "laboratory QC" sample. Designated organic laboratory QC samples should be noted on the Organic TR/COC Record. Designated inorganic laboratory QC samples should be noted on the Inorganic TR/COC Record. The laboratory QC sample must not be designated only in the "Field QC Qualifier" column on either the Organic or Inorganic TR/COC Records. Make sure that the laboratory QC sample is included in TR/COC Record samples to be used for the Laboratory QC field.

The sampler should select a field sample as the laboratory QC sample. If the sampler does not select a field sample as the laboratory QC sample, then it is possible that the laboratory could select the field blank (e.g., an equipment or rinsate blank) sample to meet contractual QC requirements. The use of field blanks for laboratory MS/MSD/Duplicate analysis reduces the usability of the data to assess data quality.



In the event of multiple sample shipments during a sampling event, it is recommended that the sampler submit laboratory QC samples in the first sample shipment.

3.1.2 Meet Volume, Preservation, and Holding Time Requirements

Samplers should refer to their project plans to obtain the specific sample volumes to be collected, the preservation needed for those samples, and the technical holding times under which they must submit samples to the scheduled CLP laboratory. Sample collection parameters (to include sample volumes, preservatives, and technical holding times) for organic collection and analysis are listed in Tables 3-2 and 3-3. Sample collection parameters for inorganic analysis and collection are listed in Table 3-4.

3.1.2.1 Collect Sample Volume

Collecting sufficient sample volume is critical. There must be sufficient physical sample volume for the analysis of all required parameters and completion of all QC determinations. The type of analytical procedure(s) to be performed will often dictate the sample volume to collect. For example, each water sample collected for VOA analysis by CLP SOW SOM01 or ILM05 requires a minimum of three vials, each filled completely to a 40 mL capacity. See Appendix C for information regarding the collection of VOAs in water. It is extremely important that samplers refer to their specific project plans to identify and collect the correct sample volume during each sampling event.

When sampling for VOAs in soils, samplers must use SW-846 Method 5035A guidelines included in Appendix B.

3.1.2.2 Preserve Samples

Degradation of some contaminants may occur naturally (e.g., VOAs). The sampler must chemically preserve some water samples for certain analytes before shipping them to the laboratory. The sampler should preserve and immediately cool all samples to 4°C (±2°C) upon collection and samples should remain cooled until the time of analysis (do not freeze water samples). Preservation techniques vary among Regions so the sampler should obtain Region-specific instructions and review the appropriate project plans and SOPs. See Appendix C for information regarding the collection of VOAs in water.

3.1.2.3 Ship within Holding Times

Samplers should ship samples to scheduled CLP laboratories as soon as possible after collection. Daily shipment of samples to CLP laboratories is preferred whenever possible. If samples cannot be shipped on a daily basis, they must be properly preserved and maintained to meet CLP-specified temperatures, holding times, and custody requirements.

The technical holding times are the maximum time allowed between a sample collection and the completion of the sample extraction and/or analysis. In contrast, contractual holding times are the maximum lengths of time that the CLP laboratory can hold the sample prior to extraction and/or analysis. These contractual holding times are described in the appropriate CLP SOW. Contractual holding times are shorter than the technical holding times to allow for sample packing and shipping.



If samplers are shipping samples after 5:00 PM ET, they must notify the RSCC (or designee) or SMO by 8:00 AM ET on the following business day. When making a Saturday delivery, samplers shall contact the RSCC (or designee) or SMO by 3:00 PM ET on the Friday prior to delivery.

Table 3-2. Sample Collection Requirements for CLP SOW SOM01 (VOAs)

Matrix	Container Type	Sample Type	Minimum Number of Containers Needed				Minimum Volume/ Mass	Important Notes	Preservative	Technical Holding Time
			with Water	Dry	% Moisture	TOTAL				
Water	See Table 2-2, Reference Number 1.	Samples Only	-	-	-	**3**	Fill to capacity	Containers/vials must be filled to capacity with no headspace or air bubbles. Refer to Appendix C for samples requiring QC analyses.	Preserve to a pH of 2 with HCl and cool to 4°C (±2ºC) immediately after collection. DO NOT FREEZE water samples.	14 days
		Samples with SIM	-	-	-	**4**				
		Samples with MS/MSD	-	-	-	**6**				
		Samples with SIM and MS/MSD	-	-	-	**8**				
Soil/ Sediment	**OPTION 1** Closed-system Vials See Table 2-2, Reference Number 1.	Samples Only	-	3	1	**4**	5g	Place samples on side prior to being frozen. Refer to Appendix B for samples requiring QC analyses.	Frozen (-7°C to -15°C) or iced to 4° (±2°C).	14 days
		Samples with MS/MSD	-	9	1	**10**				48 hours
	OPTION 2 Closed-system containing Vials Water See Table 2-2, Reference Number 1.	Samples Only	2	1	1	**4**	5g	Containers/vials must be filled to capacity with no headspace or air bubbles. Place samples on side prior to being frozen. Refer to Appendix B for samples requiring QC analyses.	Frozen (-7°C to -15°C) or iced to 4° (±2°C). DO NOT FREEZE water samples.	14 days
		Samples with MS/MSD	6	1	5	**12**				48 hours
	OPTION 3 See Table 2-2, Reference Number 7.	Samples Only	-	3	1	**4**	5g	Refer to Appendix B for samples requiring QC analysis.	Frozen (-7°C to -15°C) or iced to 4°C (±2°C).	48 hours
		Samples with MS/MSD	-	9	1	**10**				48 hours

Notes

[1] Minimum volume/mass to be collected in order to ensure sample analysis can be performed.

[2] Check Regional guidance regarding use of acid as a preservative of samples that may contain carbonates, residual chlorine, and other oxidants.

[3] This technical holding time is calculated from the time of sample collection to sample extraction. Sample extracts are to be analyzed within 40 days of extraction. It is recommended that samplers ship samples to the laboratory on the same day that they are collected, or as soon as possible thereafter.

[4] Check Regional guidance regarding use of acid preservatives when testing for carbonates, residual chlorine, and other oxidants.

Table 3-3. Sample Collection Requirements for CLP SOW SOM01 (SVOAs, Pesticides and Aroclors)

Analysis	Matrix	Containers	Minimum Volume/ Mass	Important Notes	Preservative	Technical Holding Time
Semivolatile Analytes	Water	See Table 2-2, Reference Number 5.	2L	If amber containers are not available, the samples should be protected from light.	Cool all samples to 4°C (±2°C) immediately after collection. DO NOT FREEZE water samples.	7 days
	Soil/ Sediment	See Table 2-2, Reference Numbers 3 and 4.	Fill to capacity		Cool all samples to 4°C (±2°C) immediately after collection.	14 days
Pesticides/ Aroclors	Water	See Table 2-2, Reference Number 5.	2L	If amber containers are not available, the samples should be protected from light.	Cool all samples to 4°C (±2°C) immediately after collection. DO NOT FREEZE water samples.	7 days
	Soil/ Sediment	See Table 2-2, Reference Numbers 3 and 4.	Fill to capacity		Cool all samples to 4°C (±2°C) immediately after collection.	14 days

Notes

[1] Minimum volume/mass to be collected in order to ensure sample analysis can be performed.
[2] Check Regional guidance regarding use of acid as a preservative of samples that may contain carbonates, residual chlorine, and other oxidants.
[3] This technical holding time is calculated from the time of sample collection to sample extraction. Sample extracts are to be analyzed within 40 days of extraction. It is recommended that samplers ship samples to the laboratory on the same day that they are collected, or as soon as possible thereafter.
[4] Check Regional guidance regarding use of acid preservatives when testing for carbonates, residual chlorine, and other oxidants.

Table 3-4. Sample Collection Requirements for CLP SOW ILM05

Analysis	Matrix	Containers	Minimum Volume/ Mass[1]	Important Notes	Preservative	Technical Holding Time[4]
Metals/ICP-AES and/or Mercury by CVAA	Water	See Table 2-2, Reference Number 2.	1L	If collecting for both ICP-AES AND ICP-MS methods, a separate 1L volume of sample must be collected for each method per sample location.	Acidify to pH < 2 with HNO_3 and cool to 4°C (±2°C) immediately after collection.[2] DO NOT FREEZE water samples.	6 months for all metals except Mercury (28 days)
	Soil/ Sediment	See Table 2-2, Reference Number 3.	Fill to capacity		Cool to 4°C (±2°C) immediately after collection.	6 months
Cyanide/ Spectrophotometric Determination[3]	Water	See Table 2-2, Reference Number 2.	1L		To neutralize residual chlorine, immediately upon collection, add 0.6 g ascorbic acid for each liter of sample collected. Add NaOH until pH >12 and cool to 4°C (±2°C) immediately after collection.[5] DO NOT FREEZE water samples.	14 days
	Soil/ Sediment	See Table 2-2, Reference Number 3.	Fill to capacity		Cool to 4°C (±2°C) immediately after collection.	14 days

Notes

1 Minimum volume/mass to be collected in order to ensure sample analysis can be performed.

2 Check Regional guidance regarding use of acid as a preservative of samples that may contain carbonates, residual chlorine, and other oxidants.

3 Samplers must test for sulfide and oxidizing agents (e.g., chlorine) in aqueous samples in the field upon collection. Please refer to the SAP and Appendix C for guidance. Sulfides adversely affect the analytical procedure. The following can be done to test for and neutralize sulfides. Place a drop of the sample on lead acetate test paper to detect the presence of sulfides. If sulfides are present, treat 25 mL more of the sample than that required for the cyanide determination with powdered cadmium carbonate or lead carbonate. Yellow cadmium sulfide or black lead sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complication or occlusion of cyanide on the precipitated material. Sulfide removal should be performed in the field, if practical, prior to pH adjustment with NaOH.

4 This technical holding time is calculated from the time of sample collection to sample extraction. Sample extracts are to be analyzed within 40 days of extraction. It is recommended that samplers ship samples to the laboratory on the same day that they are collected, or as soon as possible thereafter.

3.2 Complete Documentation

Samplers must complete all documentation, including the recording of the CLP Sample Number on the sample container or bottle, sample labels, and chain-of-custody seals (as appropriate), the completion of the TR/COC Record, and the completion of field operations records (as necessary).

Samplers should use the FORMS II Lite software to create and print sample labels and the TR/COC Record. Samplers can create and print out two copies of a sample label and attach one to the sample container or bottle, and place the other on the sample tag that may be attached to the sample container or bottle.

Samplers are expected to review their project plans to determine what documentation they are expected to include during a sampling event. It is highly recommended that samplers provide documentation, even if the Region does not require it.

Note: Under no circumstances should the site name appear on any documentation being sent to the laboratory.

An example of a packaged sample is shown in Figure 3-1. A description of each type of documentation and instructions for accurate completion are included in the following sections.

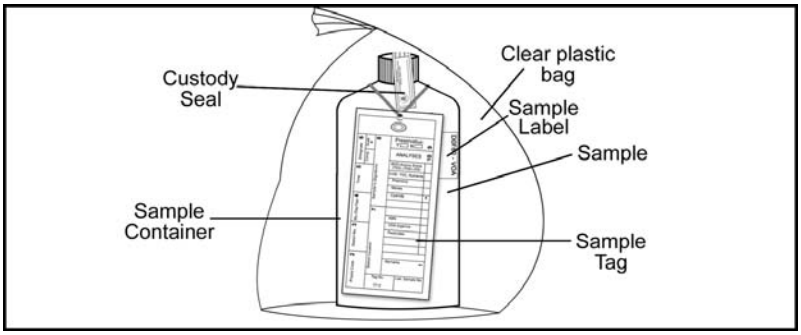


Figure 3-1. Packaged Sample with Identification and Chain-of-Custody Documentation (Excluding TR/COC Record)

3.2.1 Identify a Sample with a CLP Sample Number and SMO-assigned Case Number

The CLP Sample Number and SMO-assigned Case Number **must** be recorded on each sample taken during a sampling event (see Section 1.4.1.1). Samplers can record these numbers on the sample bottle or container using permanent ink. The numbers must also be recorded on the sample tag, if required.

Note: Dissolved metal samples and total metal samples taken from the same sampling location cannot have the same CLP Sample Number because two different sets of data will be generated.

3.2.2 Complete TR/COC Records

A Traffic Report is used as physical evidence of sample custody and as a permanent record for each sample collected. A chain-of-custody record documents the exchange and transportation of samples from the field to the laboratory.

The ASB requires samplers to use the FORMS II Lite software to create documentation for all CLP sampling efforts. For assistance with obtaining or using the FORMS II Lite software, please contact the FORMS II Lite Help Desk at 703-818-4200 from 9:00 AM - 5:00 PM ET.

To meet CLP sample documentation and chain-of-custody requirements, the sampler must attach a separate TR/COC Record to each cooler they ship. The TR/COC Record must document each sample within the cooler. Samples shipped in other coolers should not be documented. This practice maintains the chain-of-custody for all samples in case of incorrect shipment.

If more than one TR/COC Record is used for the samples within one cooler, all of the records must have complete header information and original signatures. Samplers are responsible for the care and custody of samples from the time of collection to the time of shipment to the laboratories for analysis. A sample is considered under custody if:

- It is in possession or in view after being in possession;
- It was in possession and then secured or sealed to prevent tampering; or
- It was in possession when placed in a secured area.

Each time the custody of samples is turned over to another person, the TR/COC Record must be signed off by the former custodian and accepted by the new custodian. Samplers are, therefore, responsible for properly completing any forms or other Region-required documentation used to establish the chain-of-custody for each sample during a sampling event.

3.2.2.1 Complete a TR/COC Record Using the FORMS II Lite Software

Once the sampler inputs sample collection information into FORMS II Lite, a TR/COC Record will be generated electronically. The software automatically displays only the information to be entered by the sampler. FORMS II Lite then generates a laboratory and a Regional copy of the TR/COC Record (see Figures 3-2 through 3-5). The sampler can print out multiple copies of the TR/COC Record as necessary. The sampler must sign and submit original copies of the TR/COC Record as appropriate.

An electronic TR/COC Record created using the FORMS II Lite software contains basic header information; however, the sampler can also include some additional detailed information. For example, not only is the sample matrix listed on the electronic TR/COC Record, but the name of the sampler taking the sample can also be entered. Samplers should note that certain information will not appear on the electronic TR/COC Record (e.g., matrix and preservative descriptions).

3.2.2.2 Indicate Modified Analysis on FORMS II Lite TR/COC Records

When completing a TR/COC Record using FORMS II Lite, the sampler should identify any samples that will be analyzed using a CLP Modified Analysis. Samplers should indicate use of a Modified Analysis by creating a new analysis within the FORMS II Lite Wizard or through the FORMS II Lite Reference Tables. This newly-created analysis should contain the Modification Reference Number within the name assigned to the analysis. For example, if a Region submits a Modified Analysis for an additional analyte, and SMO assigns the Modification Reference Number 1301.0, the FORMS II Lite analysis could be named "VOA by M.A. 1301.0". The associated abbreviation for this analysis could be "VOA M.A.". If you have any questions regarding identification of Modified Analysis using FORMS II Lite, please contact the FORMS II Lite Help Desk at 703-818-4200 from 9:00 AM - 5:00 PM ET.

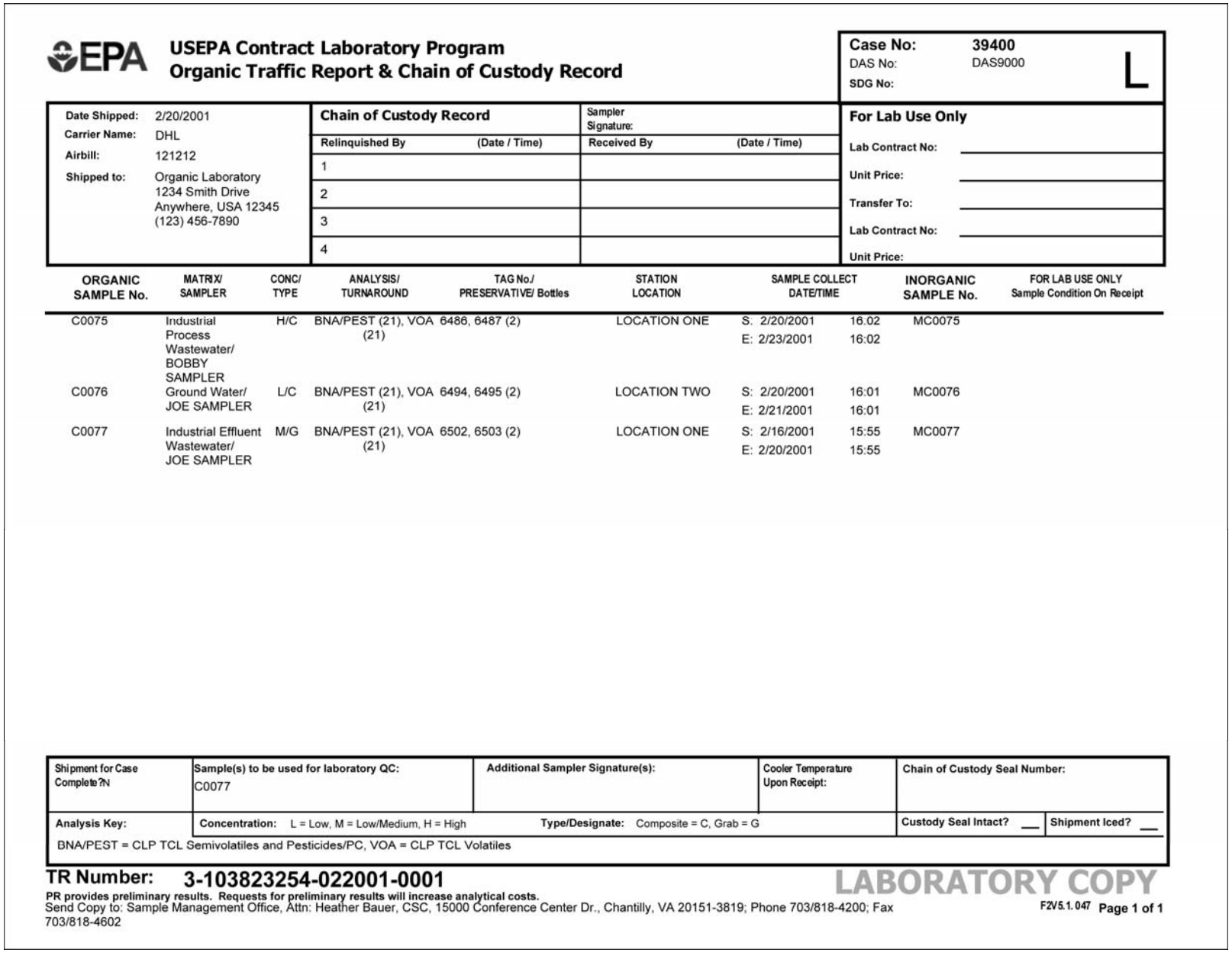
3.2.2.3 Make Manual Edits to Printed FORMS II Lite TR/COC Records

If a FORMS II Lite TR/COC Record has been printed and deletions or edits need to be made by the sampler, the following procedures must be followed:

- If making a deletion, manually cross out the information to be disregarded from the TR/COC Record, initial and date the deletion.
- If making an addition, enter the new information and initialsign and date the newly added information.



All modifications made on a printed TR/COC Record must be initialed and dated.



USEPA Contract Laboratory Program
Organic Traffic Report & Chain of Custody Record

Case No: 39400
DAS No: DAS9000
SDG No:
L

Date Shipped:	2/20/2001
Carrier Name:	DHL
Airbill:	121212
Shipped to:	Organic Laboratory 1234 Smith Drive Anywhere, USA 12345 (123) 456-7890

Chain of Custody Record

Relinquished By	(Date / Time)	Received By	(Date / Time)
1			
2			
3			
4			

Sampler Signature:

For Lab Use Only
Lab Contract No:
Unit Price:
Transfer To:
Lab Contract No:
Unit Price:

ORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME		INORGANIC SAMPLE No.	FOR LAB USE ONLY Sample Condition On Receipt
C0075	Industrial Process Wastewater/ BOBBY SAMPLER	H/C	BNA/PEST (21), VOA (21)	6486, 6487 (2)	LOCATION ONE	S: 2/20/2001 E: 2/23/2001	16:02 16:02	MC0075	
C0076	Ground Water/ JOE SAMPLER	L/C	BNA/PEST (21), VOA (21)	6494, 6495 (2)	LOCATION TWO	S: 2/20/2001 E: 2/21/2001	16:01 16:01	MC0076	
C0077	Industrial Effluent Wastewater/ JOE SAMPLER	M/G	BNA/PEST (21), VOA (21)	6502, 6503 (2)	LOCATION ONE	S: 2/16/2001 E: 2/20/2001	15:55 15:55	MC0077	

Shipment for Case Complete?N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Cooler Temperature Upon Receipt:	Chain of Custody Seal Number:	
	C0077				
Analysis Key:	**Concentration:** L = Low, M = Low/Medium, H = High	**Type/Designate:** Composite = C, Grab = G		**Custody Seal Intact?** ___	**Shipment Iced?** ___

BNA/PEST = CLP TCL Semivolatiles and Pesticides/PC, VOA = CLP TCL Volatiles

TR Number: 3-103823254-022001-0001

LABORATORY COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

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Figure 3-2. Organic Traffic Report & Chain of Custody Record (Laboratory Copy)

USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record

Case No: 39400
DAS No: DAS9000
SDG No:
L

Date Shipped:	2/20/2001
Carrier Name:	DHL
Airbill:	121212
Shipped to:	Inorganic Laboratory 1234 Smith Drive Anywhere, USA 12345 (123) 456-7890

Chain of Custody Record

Relinquished By	(Date / Time)	Received By	(Date / Time)
1			
2			
3			
4			

Sampler Signature:

For Lab Use Only
Lab Contract No:
Unit Price:
Transfer To:
Lab Contract No:
Unit Price:

INORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME		ORGANIC SAMPLE No.	FOR LAB USE ONLY Sample Condition On Receipt
MC0075	Industrial Process Wastewater/ BOBBY SAMPLER	H/C	Al (21), Ba (21), Ca (21), Cr (21), TM/CN (21)	6481, 6482, 6483, 6484, 6485 (5)	LOCATION ONE	S: 2/20/2001 E: 2/23/2001	16:02 16:02	C0075	
MC0076	Ground Water/ JOE SAMPLER	L/C	Al (21), Ba (21), Ca (21), Cr (21), TM/CN (21)	6489, 6490, 6491, 6492, 6493 (5)	LOCATION TWO	S: 2/20/2001 E: 2/21/2001	16:01 16:01	C0076	
MC0077	Industrial Effluent Wastewater/ JOE SAMPLER	M/G	Al (21), Ba (21), Ca (21), Cr (21), TM/CN (21)	6497, 6498, 6499, 6500, 6501 (5)	LOCATION ONE	S: 2/16/2001 E: 2/20/2001	15:55 15:55	C0077	

Shipment for Case Complete?N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Cooler Temperature Upon Receipt:	Chain of Custody Seal Number:	
	MC0077				
Analysis Key:	**Concentration:** L = Low, M = Low/Medium, H = High	**Type/Designate:** Composite = C, Grab = G		**Custody Seal Intact?** ___	**Shipment Iced?** ___

Al = Aluminum, Ba = Barium, Ca = Calcium, Cr = Chromium, TM/CN = CLP TAL Total Metals and Cyanide

TR Number: 3-103823254-022001-0003

LABORATORY COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

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Figure 3-3. Inorganic Traffic Report & Chain of Custody Record (Laboratory Copy)

USEPA Contract Laboratory Program
Organic Traffic Report & Chain of Custody Record

Case No: 39400 R
DAS No: DAS9000

Region:	3	Date Shipped:	2/20/2001	Chain of Custody Record	Sampler Signature:
Project Code:	QW-123	Carrier Name:	DHL	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	ACCT000	Airbill:	121212	1	
CERCLIS ID:		Shipped to:	Organic Laboratory 1234 Smith Drive Anywhere, USA 12345 (123) 456-7890	2	
Spill ID:	ID3			3	
Site Name/State:	REAL SITE, UT			4	
Project Leader:	DAN SAMPLER				
Action:	Other				
Sampling Co:	SMITH CO.				

ORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	INORGANIC SAMPLE No.	QC Type
C0075	Industrial Process Wastewater/ BOBBY SAMPLER	H/C	BNA/PEST (21), VOA (21)	6486, 6487 (2)	LOCATION ONE	S: 2/20/2001 16:02 E: 2/23/2001 16:02	MC0075	--
C0076	Ground Water/ JOE SAMPLER	L/C	BNA/PEST (21), VOA (21)	6494, 6495 (2)	LOCATION TWO	S: 2/20/2001 16:01 E: 2/21/2001 16:01	MC0076	Spike
C0077	Industrial Effluent Wastewater/ JOE SAMPLER	M/G	BNA/PEST (21), VOA (21)	6502, 6503 (2)	LOCATION ONE	S: 2/16/2001 15:55 E: 2/20/2001 15:55	MC0077	--

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC: C0077	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____
BNA/PEST = CLP TCL Semivolatiles and Pesticides/PC, VOA = CLP TCL Volatiles			

TR Number: 3-103823254-022001-0001

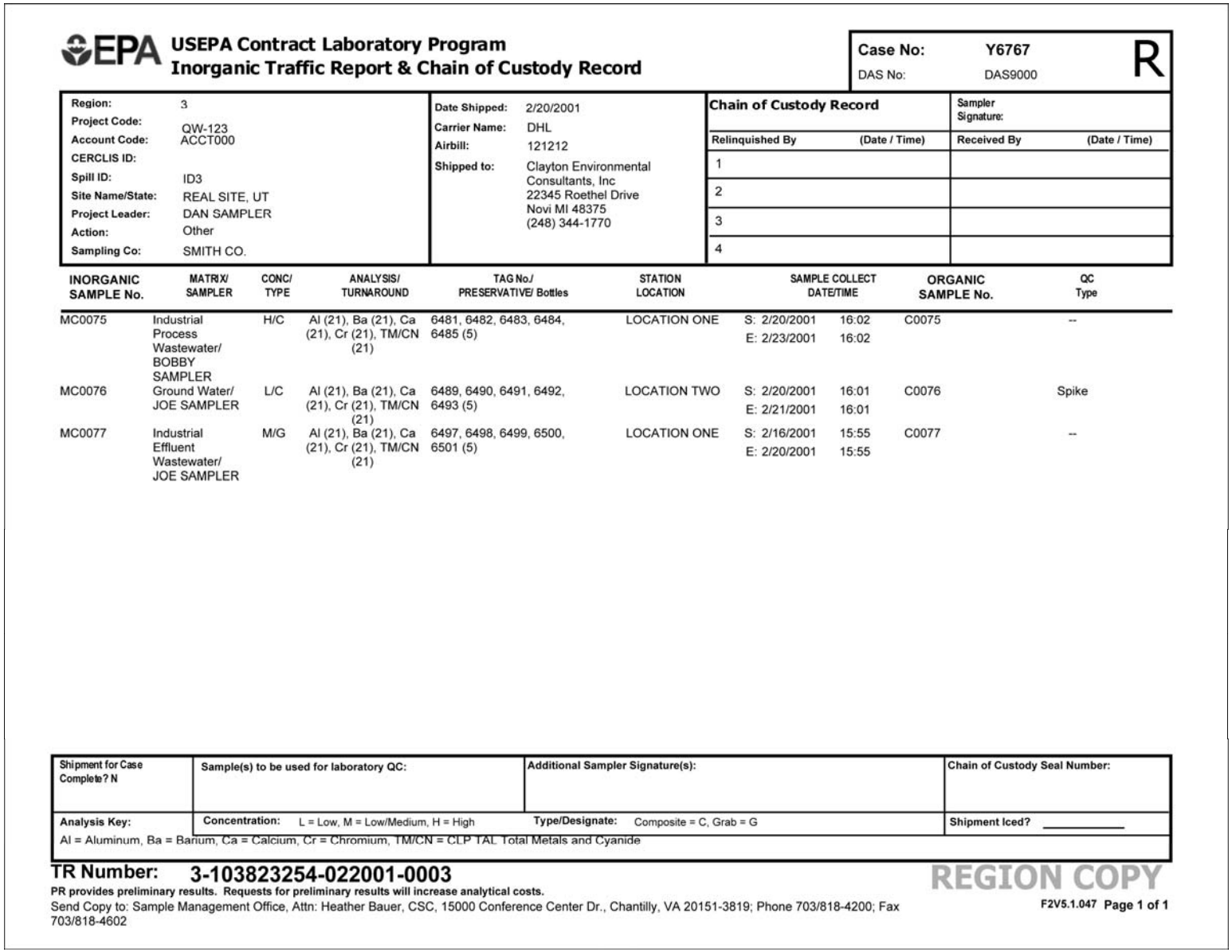
REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

F2V5.1.047 Page 1 of 1

Figure 3-4. Organic Traffic Report & Chain of Custody Record (Region Copy)



EPA **USEPA Contract Laboratory Program**
Inorganic Traffic Report & Chain of Custody Record

Case No: Y6767 **R**
DAS No: DAS9000

Region:	3	**Date Shipped:**	2/20/2001
Project Code:	QW-123	**Carrier Name:**	DHL
Account Code:	ACCT000	**Airbill:**	121212
CERCLIS ID:		**Shipped to:**	Clayton Environmental Consultants, Inc 22345 Roethel Drive Novi MI 48375 (248) 344-1770
Spill ID:	ID3		
Site Name/State:	REAL SITE, UT		
Project Leader:	DAN SAMPLER		
Action:	Other		
Sampling Co:	SMITH CO.		

Chain of Custody Record

Relinquished By	(Date / Time)	Received By	(Date / Time)
1			
2			
3			
4			

Sampler Signature:

INORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	QC Type
MC0075	Industrial Process Wastewater/ BOBBY SAMPLER	H/C	Al (21), Ba (21), Ca (21), Cr (21), TM/CN (21)	6481, 6482, 6483, 6484, 6485 (5)	LOCATION ONE	S: 2/20/2001 16:02 E: 2/23/2001 16:02	C0075	--
MC0076	Ground Water/ JOE SAMPLER	L/C	Al (21), Ba (21), Ca (21), Cr (21), TM/CN (21)	6489, 6490, 6491, 6492, 6493 (5)	LOCATION TWO	S: 2/20/2001 16:01 E: 2/21/2001 16:01	C0076	Spike
MC0077	Industrial Effluent Wastewater/ JOE SAMPLER	M/G	Al (21), Ba (21), Ca (21), Cr (21), TM/CN (21)	6497, 6498, 6499, 6500, 6501 (5)	LOCATION ONE	S: 2/16/2001 15:55 E: 2/20/2001 15:55	C0077	--

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____
Al = Aluminum, Ba = Barium, Ca = Calcium, Cr = Chromium, TM/CN = CLP TAL Total Metals and Cyanide			

TR Number: 3-103823254-022001-0003

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

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Figure 3-5. Inorganic Traffic Report & Chain of Custody Record (Region Copy)

3.2.3 Complete and Attach Custody Seals

Custody seals are usually pre-printed stickers that are signed (or initialed) and dated by the sampler after sample collection and placed on sample bottles or containers and/or shipping coolers or containers (see Figure 3-6). The custody seals document who sealed the sample container and verifies that the sample has not been tampered with. The seals must be placed such that they will break if the sample bottle or container or the shipping cooler or container is tampered with or opened after leaving custody of samplers. Custody seals can also be used to maintain custody of other items such as envelopes containing videotapes of the sample collection process.



Custody seals should never be placed directly onto a coring tool used as a transport device (e.g., 5 g Sampler) or tared, 40 mL closed-system vials. The seals must be placed on the bag for the coring tool used as a transport device, or on the bag used to enclose the vials. Refer to Appendix B for details.

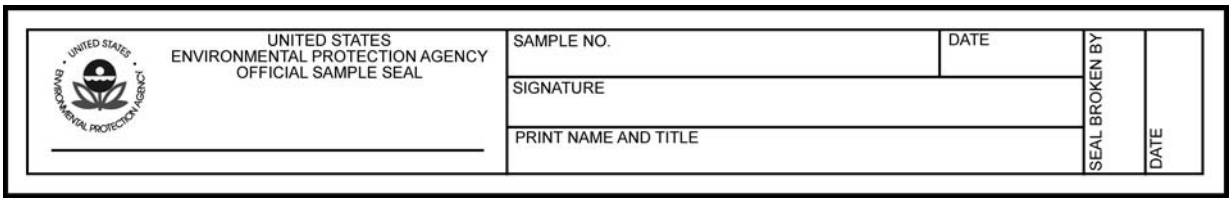


Figure 3-6. Custody Seal

Instructions for completing and attaching a custody seal are included in Table 3-5.

Table 3-5. Completing and Attaching a Custody Seal

Step	Action	Important Notes
1	Record the CLP Sample Number.	The space for the CLP Sample Number does not need to be completed on custody seals being placed on the opening of a cooler, only on those being placed on the opening of sample bottles or containers.
2	Record the month, day, and year of sample collection.	
3	Sign the seal in the Signature field.	
4	Print your name and title in the Print Name and Title field.	
5	Place the custody seal over the edge of the sample bottle or container such that it will break if tampered with.	Custody seals can be placed directly on any sample container except for coring tools used as a transport device (e.g., 5 g Samplers) and tared VOA bottles. If packing coring tools used as a transport device or tared VOA bottles, place them in a clear plastic bag and place the custody seal on the outside of the bag.
6	If possible, cover the custody seal with clear plastic tape to protect it.	Take special care to not place the protective tape over the seal in such a way that it can be removed and then re-attached without signs of tampering.

The use and type of custody seals can vary by Region or collecting organization. Samplers should obtain the appropriate custody seals and specific instructions for correctly attaching them from the RSCC.

3.2.4 Complete and Attach Sample Labels

Samplers affix sample labels to each sample container. A sample label must contain the associated CLP Sample Number (either written or pre-printed), SMO-assigned Case Number, and the preservative used. It must also denote the analysis/fraction. Samplers may also include additional information such as the station location or the date/time of collection. Samplers should use FORMS II Lite to create and print sample labels. The sampler can print two labels and attach one to the sample container or bottle, and place the other label on the sample tag that should also be attached to the sample container or bottle. The

labels should then be covered with clear packaging tape to protect the label and maintain legibility. If handwriting a sample label, the sampler should complete the label information using waterproof ink, place the label on the outside of the sample bottle or container, then cover the label with clear packaging tape to protect the label and maintain legibility (see Figure 3-1).



Do not attach labels to tared VOA sample vials. A label should already be pre-attached to the tared vial.

3.2.5 Complete and Attach Sample Tags

To support use of sample data in potential enforcement actions, sample characteristics other than on-site measurements (e.g., pH, temperature, conductivity) can be identified with a sample tag. Typically, site-specific information is written on the tags using waterproof ink. The use and type of sample tags may vary by Region. For each sampling event, samplers should receive the required sample tags and type of information to include from the RSCC. The sampler can use FORMS II Lite to create and print out multiple sample labels, one of which can be attached to the sample tag and then covered with clear packaging tape to protect the label and maintain legibility. If FORMS II Lite-created sample labels are not available, a detailed set of instructions for completing and attaching a handwritten sample tag are included in Table 3-6.



The use and type of sample tags may vary among Regions.

Table 3-6. Completing and Attaching a Handwritten Sample Tag

Step	Action	Important Notes
1	Under the "Remarks" heading, record the CLP Sample Number and SMO-assigned Case Number.	Make sure to record the correct CLP Sample Number and SMO-assigned Case Number in a legible manner.
2	Record the project code (e.g., Contract Number, Work Assignment Number, Interagency Agreement Number, etc.) assigned by USEPA.	
3	Enter the station number assigned by the sampling team coordinator.	
4	Record the month, day, and year of sample collection.	
5	Enter the military time of sample collection (e.g., 13:01 for 1:01 PM).	
6	Identify the designate and place an "X" in either the "Comp." or "Grab" box if the sample is either a composite or grab sample.	
7	Record the station location.	
8	Sign the sample tag in the Signature area.	
9	Place an "X" in the box next to Yes or No to indicate if a preservative was added to the sample.	
10	Under "Analyses", place an "X" in the box next to the parameters for which the sample is to be analyzed.	
11	Leave the box for "Laboratory Sample Number" blank.	
12	It is recommended that the sample tag be attached to the neck of the sample bottle or container using regular string, stretch string, or wire (see Figure 3-1).	Do **NOT** use wire to attach a sample tag to a metals sample.

An example of a completed sample tag is included in Figure 3-7 below:

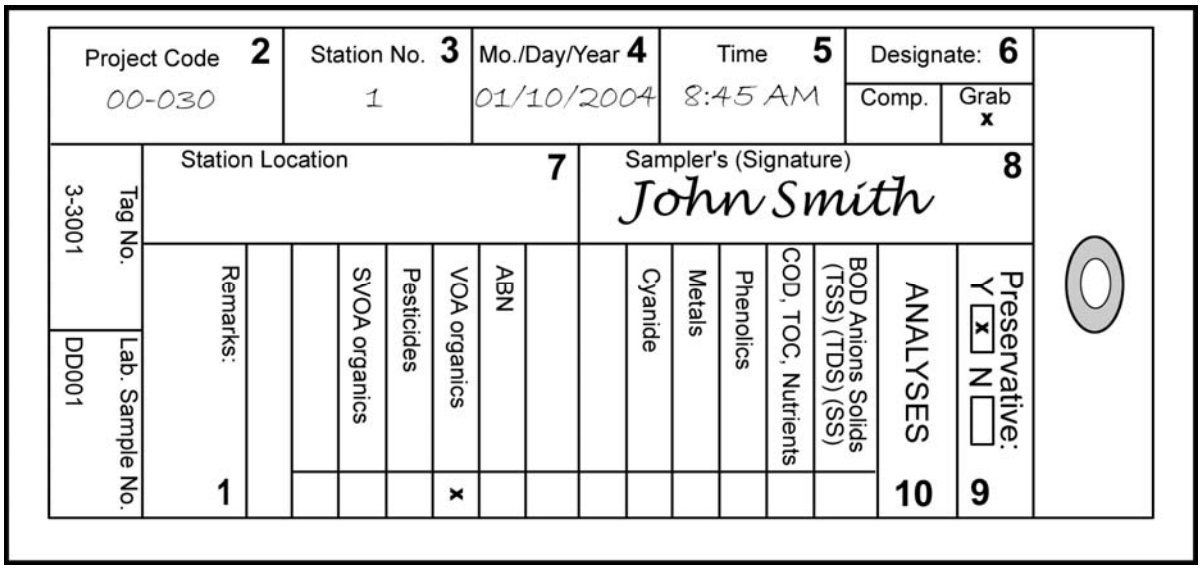


Figure 3-7. Completed Sample Tag

3.3 Provide Sample Receipt

After samples have been taken from private property, the sampler should prepare a receipt for these samples and provide this receipt to the property owner. This is especially important when sampling on private property since these samples could be used during future litigation and the receipt will verify that the owner granted approval for the removal of the samples from the property. An example of a sample receipt created using FORMS II Lite is shown in Figure 3-8.

EPA Region 3
U.S. ENVIRONMENTAL PROTECTION AGENCY

RECEIPT FOR SAMPLES

PROJECT NO.	PROJECT NAME	NAME & LOCATION OF FACILITY/SITE
QW-123		EXAMPLE SITE
SAMPLERS: (SIGNATURES)		

STATION NO.	LOCATION/DESCRIPTION	DATE	TIME	Comp/Grab	NO. OF EPA CONTAINERS	SPLIT SAMPLE Y OR N	EPA SAMPLE TAG NO.'S
STATION ONE	LOCATION ONE	2/20/2001	15:55	G	11	Yes	112, 113, 114, 115, 116, 117, 118, 119, 120, 121, 122
STATION ONE	LOCATION TWO	2/20/2001	16:01	C	11	Yes	123, 124, 125, 126, 127, 128, 129, 130, 131, 132, 133
STATION TWO	LOCATION ONE	2/20/2001	16:02	C	11	Yes	134, 135, 136, 137, 138, 139, 140, 141, 142, 143, 144

SPLIT SAMPLES TRANSFERRED BY: (PRINT)	DATE	SPLIT SAMPLES RECEIVED BY ☐ OR DECLINED BY ☐ (PRINT)	DATE/TIME
(SIGN)	TIME	(SIGN)	TELEPHONE
		TITLE	

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Figure 3-8. Sample Receipt Created Using the FORMS II Lite Software

3.4 Pack and Ship Samples

Once the samples have been collected, it is very important that the sampler properly package the samples for shipment and ensure that the samples are sent to the appropriate laboratory as quickly as possible. Prompt and proper packaging of samples will:

- Protect the integrity of samples from changes in composition or concentration caused by bacterial growth or degradation from increased temperatures;
- Reduce the chance of leaking or breaking of sample containers that would result in loss of sample volume, loss of sample integrity, and exposure of personnel to toxic substances; and
- Help ensure compliance with shipping regulations.

3.4.1 Sample Containers

Once samples are collected, they must be stored in conditions that maintain sample integrity. All samples should be placed in shipping containers or other suitable containers with ice to reduce the temperature as soon as possible after collection. Ideally, all samples should be shipped the day of collection for overnight delivery to the laboratory. If samples cannot be shipped on the day of collection, the sample temperature should be maintained at 4°C (±2°C) until they are shipped to the laboratory.

One CLP RAS sample may be contained in several bottles and vials. For example, one soil sample may consist of all containers needed for three of the analytical fractions available under this service (i.e., SVOA fraction, Pesticide fraction, and Aroclor fraction), even though the fractions are collected in separate containers. Therefore, the analysis to be performed and the matrix type will determine the type of container(s) that will be used, as well as the volume that must be collected for that particular sample fraction.

3.4.2 Inventory of Samples and Documentation

Prior to shipment, samplers should conduct an inventory of the contents of the shipping cooler or container against the corresponding TR/COC Record when packing for shipment to laboratories. An inventory will ensure that the proper number of containers have been collected for each analysis of the samples, that the required PE and QC samples and cooler temperature blanks are included, and the correct Sample Numbers and fractions have been assigned to each sample.

3.4.3 Shipping Regulations

Sample shipping personnel are legally responsible for ensuring that the sample shipment will comply with all applicable shipping regulations. For example, hazardous material samples must be packaged, labeled, and shipped in compliance with all IATA Dangerous Goods regulations or DOT regulations and USEPA guidelines. Refer to Appendix B for detailed shipping guidelines when using SW-846 Method 5035A to preserve and ship samples.

3.4.4 Sample Packaging for Shipment

Samplers are responsible for the proper packaging of samples for shipment. To ensure that samples are appropriately packaged (e.g., to avoid breakage and/or contamination) the sampler should consult their respective project plans to determine the proper packing and shipping procedures. The sampler must determine the sample type, pack the shipping containers correctly, include necessary paperwork, label and seal shipping containers or coolers, and ship the samples.

3.4.4.1 Determine the Sample Type and Container

Samplers should know what kinds of samples they are handling to ensure proper packaging. Samplers should refer to their appropriate project plans to determine which type of sample container should be used for each type of sample being taken during the sampling event.



Please follow Regional guidance with reference to samples containing dioxin or radioactive waste.

3.4.4.2 Pack Shipping Containers

It is imperative that samples are correctly and carefully packed in shipping containers to prevent the sample containers from breaking or leaking. Samplers must prepare and pack a shipping cooler or container according to the instructions outlined in Table 3-7.

Table 3-7. Packing Samples for Shipment

Step	Action	Important Notes
1	Seal all drain holes in the shipping container, both inside and out, to prevent leakage in the event of sample breakage.	
2	Check all lids/caps to make sure the samples are tightly sealed and will not leak.	
3	Seal samples within a clear plastic bag.	Custody seals can be placed directly on any sample container except for coring tools used as a transport device (e.g., 5 g Samplers) and tared VOA bottles. If packing coring tools used as a transport device or tared VOA bottles, place them in a clear plastic bag and place the custody seal on the outside of the bag.
4	Fully chill samples to 4°C (±2°C) prior to placement within suitable packing materials.	
5	Prior to placing samples within the shipping cooler, it is recommended that samplers line shipping containers with non-combustible, absorbent packing material.	
6	Place samples in CLEAN, sealed, watertight shipping containers (metal or hard plastic coolers).	
7	Conduct an inventory of the contents of the shipping cooler/container against the corresponding TR/COC Record.	
8	Cover samples in double-bagged ice to prevent water damage to packing materials.	Do NOT pour loose ice directly into the sample cooler. The ice is used to maintain the temperature of the samples within the shipping cooler.
9	It is recommended a temperature blank be included within each cooler being shipped.	The temperature blank is generally a 40 L vial filled with water and labeled “temperature blank” but does not have a Sample Number.
10	Ensure that the site name or other site-identifying information does not appear on any documentation being sent to the laboratory.	The laboratory should not receive any site-identifying information.

3.4.4.3 Include Necessary Paperwork

Samplers must properly place the necessary paperwork in the shipping cooler. All paperwork must be placed in a plastic bag or pouch and then secured to the underside of the shipping cooler lids (see Figure 3-9).

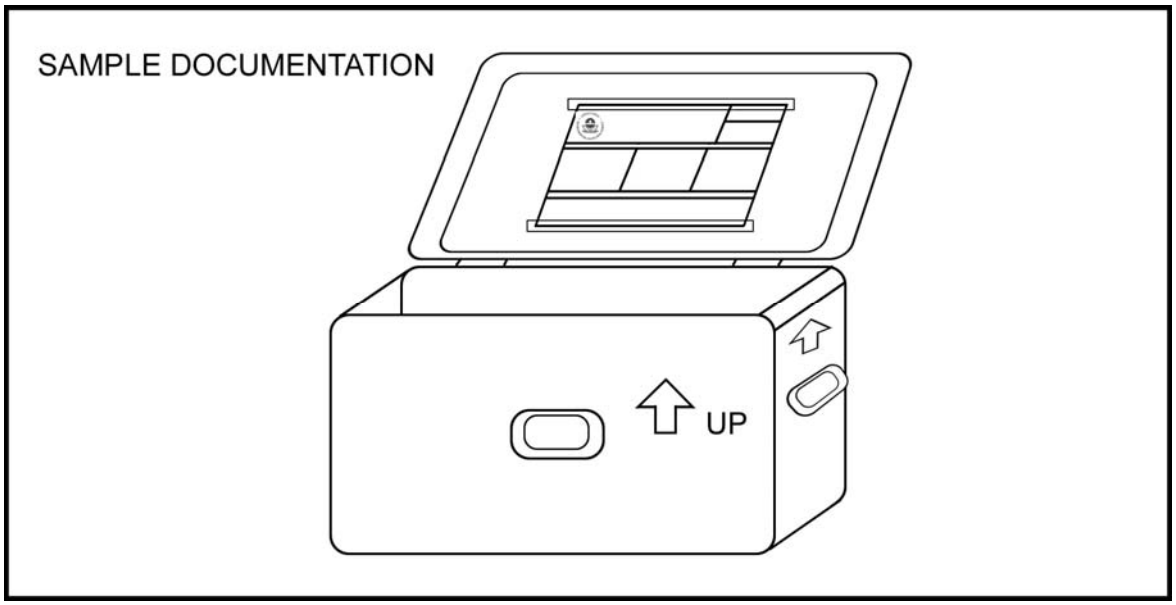


Figure 3-9. Sample Cooler with Attached TR/COC Record and Cooler Return Documentation

Necessary paperwork includes TR/COC Records and sample weight logs (see Figure 3-10), if required (for VOA samples). Samplers should contact their RSCC (or designee) for specific paperwork requirements.

USEPA Contract Laboratory Program
Sample Weight Log

Shipped to: AAA Testing Laboratory
1700 Mill Avenue
Houston TX 77099
(281) 983-1234

Case No. 39563
DAS No. DAS34
Date Shipped: 9/29/2003

Sample No.	Matrix	Analysis	Preservative	Bottle/ Tag Number	Tared Weight (g)	Final Weight (g)	Sample Weight (g)	Laboratory Weight	Traffic Report No.
C0036	Subsurface Soil (>12")	CLP TCL Volatiles	Ice Only	199548	32.80	37.20	4.40		3-103018225-092903-0001
C0036	Subsurface Soil (>12")	CLP TCL Volatiles	Ice Only	199547	32.10	38.30	6.20		3-103018225-092903-0001
C0036	Subsurface Soil (>12")	CLP TCL Volatiles	Ice Only	199549	31.20	38.60	7.40		3-103018225-092903-0001
C0037	Surface Soil (0"-12")	CLP TCL Volatiles	Ice Only	199552	32.00	36.90	4.90		3-103018225-092903-0001
C0037	Surface Soil (0"-12")	CLP TCL Volatiles	Ice Only	199551	32.40	37.10	4.70		3-103018225-092903-0001
C0037	Surface Soil (0"-12")	CLP TCL Volatiles	Ice Only	199550	31.90	35.90	4.00		3-103018225-092903-0001

Completed By:	Date:

All weights are measured in grams

Figure 3-10. Sample Weight Log

3.4.4.4 Return Sample Shipping Coolers

CLP laboratories must routinely return sample shipping coolers within 14 calendar days following shipment receipt. Therefore, the sampler should also include cooler return instructions with each shipment. The sampler (not the CLP laboratory) is responsible for paying for return of the cooler and should also include shipping airbills bearing the sampler's account number, as well as a return address to allow for cooler return.

3.4.4.5 Label and Seal Sample Shipping Coolers

After samples are packaged within shipping coolers, samplers must carefully secure the top and bottom of the coolers with tape, place return address labels clearly on the outside of the cooler, and attach the required chain-of-custody seals (see Figure 3-11).

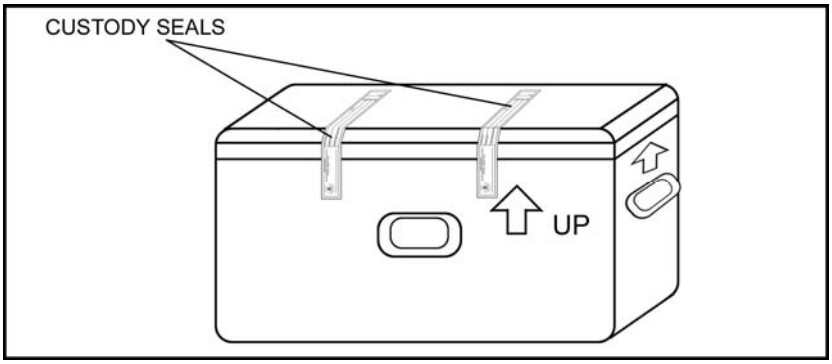


Figure 3-11. Shipping Cooler with Custody Seals

If more than one cooler is being delivered to a laboratory, samplers should mark each cooler as "1 of 2", "2 of 2", etc. In addition, samplers must accurately complete and attach shipping airbill paperwork for shipment of the samples to the laboratory. An airbill, addressed to the Sample Custodian of the receiving laboratory, should be completed for each cooler shipped. Samplers should receive the correct name, address, and telephone number of the laboratory to which they must ship samples from the RSCC or SMO. To avoid delays in analytical testing, samplers should make sure they are sending the correct types of samples to the correct laboratory when collecting samples for multiple types of analysis. For example, inorganic samples may be shipped to one laboratory for analysis, while organic samples may need to be shipped to another laboratory.

3.4.4.6 Ship Samples

The sampling contractor should ensure that samplers know the shipping company's name, address, and telephone number. In addition, they should be aware of the shipping company's hours of operation, shipping schedule, and pick-up/drop-off requirements.

Overnight Delivery

It is imperative that samples be sent via overnight delivery. Delays caused by longer shipment times may cause technical holding times to expire, which in turn may destroy sample integrity or require the recollection of samples for analysis.

Saturday Delivery

For shipping samples for Saturday delivery, the sampler MUST contact the RSCC (or their designee) or SMO so that SMO will receive the delivery information by 3:00 PM ET on the Friday prior to delivery.

3.4.5 Shipment Notification

When samples are shipped to CLP Laboratories, samplers **must immediately** report all sample shipments to the RSCC (or their designee) or to SMO. **Under no circumstances should the sampler contact the laboratory directly.** If samplers are shipping samples after 5:00 PM ET, they must notify the RSCC (or

designee) or SMO by 8:00 AM ET on the following business day. Samplers should receive the name and phone number of the appropriate SMO coordinator to contact from the Region/RSCC.

Samplers must provide the following information to the RSCC (or their designee) or to SMO:

- Name and phone number at which they can easily be reached (preferably closest on-site phone number if still in the field);
- SMO-assigned Case Number (see Section 2.4.1);
- Number, concentration, matrix and analysis of samples being shipped;
- Name of laboratory (or laboratories) to which the samples were shipped;
- Airbill number(s);
- Date of shipment;
- Case status (i.e., whether or not the Case is complete);
- Problems encountered, special comments, or any unanticipated issues;
- When to expect the next anticipated shipment; and
- An electronic export of the TR/COC Record (must be sent as soon as possible after sample shipment). For information regarding electronic export of TR/COC Records, refer to the following Web site:

http://www.epa.gov/superfund/programs/clp/f2lsubmit.htm



For Saturday delivery, samplers MUST contact the RSCC (or their designee) or SMO so that SMO will receive the delivery information by 3:00 PM ET on the Friday prior to delivery.

Samplers should be aware if their Region requires them to notify the RSCC (or designee) and/or SMO of sample shipment.

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Appendix A: Functions within a Sampling Project

The following table describes Quality Assurance Project Plan (QAPP) requirements taken from *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R-5).

Functions Within a Sampling Project	Elements of that Function
Project Management	
Project/Task Organization	Identifies the individuals or organizations participating in the project and defines their specific roles and responsibilities.
Problem Definition/Background	States the specific problem to be solved or decision to be made and includes sufficient background information to provide a historical and scientific perspective for each particular project.
Project/Task Description	Describes the work to be performed and the schedule for implementation to include: • Measurements to be made during the course of the project; • Applicable technical, regulatory, or program-specific quality standards, criteria, or objectives; • Any special personnel and equipment requirements; assessment tools needed; and • A work schedule and any required project and quality records, including types of reports needed.
Quality Objectives and Criteria	Describes the project quality objectives and measurement performance criteria.
Special Training/Certification	Ensures that any specialized training for non-routine field sampling techniques, field analyses, laboratory analyses, or data validation should be specified.
Documents and Records	• Itemizes the information and records that must be included in the data report package and specifies the desired reporting format for hard copy and electronic forms, when used. • Identifies any other records and/or documents applicable to the project such as audit reports, interim progress reports, and final reports that will be produced. • Specifies or references all applicable requirements for the final disposition of records and documents, including location and length of retention period.
Data Generation and Acquisition	
Sampling Process Design (Experimental Design)	• Describes the experimental design or data collection design for the project. • Classifies all measurements as critical or non-critical.
Sampling Methods	• Describes the procedures for collecting samples and identifies sampling methods and equipment. Includes any implementation requirements, support facilities, sample preservation requirements, and materials needed. • Describes the process for preparing and decontaminating sampling equipment to include the disposal of decontamination by-products, selection and preparation of sample containers, sample volumes, preservation methods, and maximum holding times for sampling, preparation, and/or analysis. • Describes specific performance requirements for the method. • Addresses what to do when a failure in sampling occurs, who is responsible for corrective action, and how the effectiveness of the corrective action shall be determined and documented
Sample Handling and Custody	• Describes the requirements and provisions for sample handling and custody in the field, laboratory, and transport, taking into account the nature of the samples, the maximum allowable sample holding times before extraction and analysis, and the available shipping options and schedules. • Includes examples of sample labels, custody forms, and sample custody logs.

Analytical Methods	• Identifies the analytical methods and equipment required, including sub-sampling or extraction methods, waste disposal requirements (if any), and specific method performance requirements. • Identifies analytical methods by number, date, and regulatory citation (as appropriate). If a method allows the user to select from various options, the method citations should state exactly which options are being selected. • Addresses what to do when a failure in the analytical system occurs, who is responsible for corrective action, and how the effectiveness of the corrective action shall be determined and documented. • Specifies the laboratory turnaround time needed, if important to the project schedule. • Specifies whether a field sampling and/or laboratory analysis Case Narrative is required to provide a complete description of any difficulties encountered during sampling or analysis.
Quality Control (QC)	• Identifies required measurement QC checks for both the field and laboratory. • States the frequency of analysis for each type of QC check, and the spike compounds sources and levels. • States or references the required control limits for each QC check and corrective action required when control limits are exceeded and how the effectiveness of the corrective action shall be determined and documented. • Describes or references the procedures to be used to calculate each of the QC statistics.
Instrument/Equipment Testing, Inspection, and Maintenance	• Describes how inspections and acceptance testing of environmental sampling and measurement systems and their components will be performed and documented. Identifies and discusses the procedure by which final acceptance will be performed by independent personnel. • Describes how deficiencies are to be resolved and when re-inspection will be performed. • Describes or references how periodic preventative and corrective maintenance of measurement or test equipment shall be performed. • Identifies the equipment and/or system requiring periodic maintenance. • Discusses how the availability of spare parts identified in the operating guidance and/or design specifications of the systems will be assured and maintained.
Instrument/Equipment Calibration and Frequency	• Identifies all tools, gauges, instruments, and other sampling, measuring, and test equipment used for data collection activities affecting quality that must be controlled, and at specific times, calibrated to maintain performance within specified limits. • Identifies the certified equipment and/or standards used for calibration. • Describes or references how calibration will be conducted using certified equipment and/or standards with known valid relationships to nationally recognized performance standards. If no such standards exist, documents the basis for calibration. • Indicates how records of calibration shall be maintained and traced to the instrument.
Inspection/Acceptance of Supplies and Consumables	• Describes how and by whom supplies and consumables shall be inspected and accepted for use in the project. • States acceptance criteria for such supplies and consumables.
Non-direct Measurements	• Identifies any types of data needed for project implementation or decision-making that are obtained from non-measurement sources (e.g., computer databases, programs, literature files, historical databases). • Describes the intended use of data. • Defines the acceptance criteria for the use of such data in the project. • Specifies any limitations on the use of the data.
Data Management	• Describes the project data management scheme, tracing the data path from generation in the field or laboratory to their final use or storage. • Describes or references the standard record-keeping procedures, document control system, and the approach used for data storage and retrieval on electronic media.

Appendix B: CLP Sample Collection Guidelines for VOAs in Soil by SW-846 Method 5035A

A. Preferred Options for the Contract Laboratory Program (CLP) are Options 1, 2, and 3:



Soil samples must be placed on their sides prior to being frozen.

Option 1.

Closed-system Vials:

Container - tared or preweighed 40 mL VOA Vials containing a magnetic stir bar.

Collect 5 g of soil per vial (iced or frozen in the field).

Regular Samples	3 Vials - Dry (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace) 4 Total Vials
Regular Samples Requiring QC Analysis	9 Vials - Dry (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace) 10 Total Vials

Option 2.

Closed-system Vials Containing Water:

Container - tared or pre-weighed 40 mL VOA vials containing a magnetic stir bar and 5 mL water.

Collect 5 g of soil per vial (iced or frozen in the field).

Regular Samples	2 Vials with water added (5 g soil and 5 mL water per vial) 1 Vial - Dry (5 g soil in vial) 1 Vial - Dry (filled with soil, no headspace) 4 Total Vials (2 with water and 2 dry)
Regular Samples Requiring QC Analysis	6 Vials with water added (5 g soil and 5 mL water per vial) 5 Vials - Dry (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace) 12 Total Vials (6 with water and 6 dry)

Option 3.

Coring Tool used as a Transport Device

Container - 5 g Samplers or equivalent.



All Samplers should be iced or frozen in the field and bagged individually.

Regular Samples	3 Samplers (5 g soil per Sampler) 1 Vial - Dry (filled with soil, no headspace) 4 Total (3 Samplers and 1 Vial)
Regular Samples Requiring QC Analysis	9 Samplers (5 g soil per Sampler) 1 Vial - Dry (filled with soil, no headspace) 10 Total (11 Samplers and 1 Vial)

B. Options 4, 5, and 6 are NOT preferred options for the CLP:

Option 4.

Closed-system Vials:

Container - tared or preweighed 40 mL VOA Vials containing a magnetic stir bar and preservative.

Collect 5 g of soil per vial and add Sodium bisulfate ($NaHSO_4$) preservative (5 mL water + 1 g $NaHSO_4$) - iced or frozen in the field.

Caution: This option is NOT a Preferred Option for the CLP because:

NaHSO4 preservation creates low pH conditions that will cause the destruction of certain CLP target analytes (e.g., vinyl chloride, trichloroethene, trichlorofluoromethane, cis- and trans-1,3-dichloropropene). Projects requiring the quantitation of these analytes should consider alternative sample preservation methods. NaHSO4 also cannot be used on carbonaceous soils. Check the soil before using this method of collection! Soil can be checked by placing a test sample in a clean vial, then adding several drops of NaHSO4 solution. If the soil bubbles, use Option 4b and note this issue on the TR/COC Record.

Option 4a. Samples preserved in the field

Regular Samples	2 Vials with $NaHSO_4$ preservative added (5g soil per vial) 1 Vial without $NaHSO_4$ preservative added (5g soil per vial) 1 Vial - Dry (filled with soil, no headspace) 4 Total Vials (2 with $NaHSO_4$ preservative and 2 without)
Regular Samples Requiring QC Analyses	4 Vials with $NaHSO_4$ preservative added (5g soil per vial) 5 Vials without $NaHSO_4$ preservative added (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace) 10 Total Vials (4 with $NaHSO_4$ and 6 without)

Option 4b. Samples are preserved by the laboratory (No $NaHSO_4$ preservative is added to these samples in the field).

Regular Samples	3 Vials - Dry (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace) 4 Total Vials
Regular Samples Requiring QC Analyses	9 Vials - Dry (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace) 10 Total Vials

Option 5.

Methanol Preservation (medium-level analysis only):

Container - tared or pre-weighed 40 mL VOA vials containing 5-10 mL methanol.

Collect 5 g of soil per vial (iced in the field).

Caution: This is NOT a preferred option for the CLP because:

Samples preserved with methanol can only be analyzed by the medium-level method. Low-level Contract Required Quantitation Limit (CRQLs) cannot be achieved when samples are preserved this way.

Additional problems associated with use of methanol as a preservative in the field include:

- Possible contamination of the methanol by sampling-related activities (e.g., absorption of diesel fumes from sampling equipment);
- Leakage of methanol from the sample vials during shipping, resulting in loss of VOAs prior to analysis.

Regular Samples	2 Vials (5 g soil and 5-10 mL methanol per vial) 1 Vial - Dry (filled with soil, no headspace) 3 Total Vials (2 with methanol and 1 dry)
Regular Samples Requiring QC Analysis	6 Vials (5 g soil and 5-10 mL methanol per vial) 1 Vial -Dry (filled with soil, no headspace) 7 Total Vials (6 with methanol and 1 dry)



If shipping samples containing methanol as a preservative, a shipping label must be used to indicate methanol. This label must also contain the United Nations (UN) identification number for methanol (UN 1230), and indicate Limited Quantity.

Option 6.

Glass Containers filled with sample - No Headspace:

Container - 4 oz Glass Jars.

Glass container filled with soil with no headspace and iced.

Caution: This is NOT a preferred option for the CLP because:

Samples collected in this manner lose most of their volatile analytes prior to analysis when the sample containers are opened and sub-sampled in the laboratory. This option is only available due to Regional requirements.

Regular Samples	2 Glass Jars (4 oz) filled with sample, no headspace 1 Vial - Dry (filled with soil, no headspace) 3 Total Containers
Regular Samples Requiring QC Analysis	2 Glass Jars (4 oz) filled with sample, no headspace 1 Vial - Dry (filled with soil, no headspace) 3 Total Containers

C. Caution:

1. Extreme care must be taken to ensure that frozen samples do not break during shipment.
2. Before adding soil to pre-weighed vials containing a stir bar, weigh the vials to confirm the tared weight. If the weight varies by more than 0.1 g, record the new weight on the label and the sample documentation. Do NOT add labels to these vials once the tared weight has been determined/confirmed.

D. Dry Samples:

All options include taking a sample in a dry 40 mL VOA vial (or a 4 oz wide mouth jar) with no headspace. No additional water, $NaHSO_4$, or methanol is added to this sample. This sample is taken to determine moisture content; therefore, it does not need to be tared or have a stir bar.

E. Iced or Frozen Samples:

1. Iced means cooled to 4°C (±2°C) immediately after collection.
2. Frozen means cooled to between -7°C and -15°C immediately after collection.

F. Sample Delivery:

CLP strongly recommends that all samples reach the laboratory by COB the next day after sample collection.

G. Notes:

1. For Option 4, samples can be preserved with $NaHSO_4$ either:
   - In the field; or
   - In the laboratory upon receipt. In this case, the sampler should put the following information in the Preservation Column of the TR/COC Record - "To be preserved at lab with $NaHSO_4$". This Regional Request should also be communicated to SMO so that the laboratory can be notified.
2. Regional QAPPs may require the use of Option 5. Please note that this option is for medium-level analysis ONLY.
3. If water, methanol, or $NaHSO_4$ preservative is added to the vials in the field, a field blank containing the appropriate liquid used in the vials should be sent to the laboratory for analysis.

H. Number of Containers Rationale:

The rationale for the number of containers (vials or samplers) required for the field sample and the required laboratory QC for each option is given as follows:

Option 1.

Rationale for Regular Vials:	1 vial for low-level analysis (water purge) 1 vial for backup low-level analysis 1 vial for medium-level analysis (methanol extraction)
Rationale for QC Vials:	2 vials for MS and MSD low-level analysis 2 vials for MS and MSD medium-level analysis 2 vials for backup (MS and MSD) low-level or medium-level analysis

Option 2.

Rationale for Regular Vials:	1 vial for low-level analysis (water purge) 1 vial for back up low-level analysis 1 vial dry for medium-level analysis (methanol extraction)
Rationale for QC Vials:	2 vials for MS and MSD low-level analysis 2 vials for MS and MSD medium-level analysis 2 vials for backup (MS and MSD) low-level or medium-level analysis
Medium-level: Analysis	Methanol will be added in the laboratory

Option 3.

Rationale for Regular Samples:	1 sampler for low-level analysis (water purge) 1 sampler for back up low-level analysis 1 sampler for medium-level analysis (methanol extraction)
Rationale for QC Samples:	2 samplers for MS and MSD low-level analysis 2 samplers for backup MS and MSD low-level analysis 2 samplers for MS and MSD medium-level analysis 2 samplers for backup MS and MSD medium-level analysis

Option 4a ($NaHSO_4$ added in the field).

Rationale for Regular Vials:	1 vial with water for low-level analysis (water purge) 1 vial with water for backup low-level analysis 1 vial dry for medium-level analysis (methanol extraction)
Rationale for QC Vials:	2 vials with water for MS and MSD low-level analysis 2 vials dry for MS and MSD medium-level analysis 2 vials for backup (MS and MSD) low-level or medium-level analysis

Option 4b ($NaHSO_4$ added in the laboratory).

Rationale for Regular Vials:	1 vial for low-level analysis (water purge) 1 vial for backup low-level analysis 1 vial for medium-level analysis (methanol extraction)
Rationale for QC Vials:	2 vials for MS and MSD low-level analysis 2 vials for MS and MSD medium-level analysis 2 vials for backup (MS and MSD) low-level or medium-level analysis

Option 5.

Rationale for Regular Samples:	1 vial for regular medium-level analysis 1 vial for back up medium-level analysis
Rationale for QC Samples:	2 samples for MS and MSD 2 samples for backup MS and MSD

Option 6.

In this option, all Regular and QC samples for both low-level and medium analysis are taken as subsamples from the same container.

Rationale for Regular Analysis	1 glass jar for low-level analysis and medium-level analysis **1 glass jar for backup low-level analysis and medium-level analysis**
Rationale for QC Analysis:	1 glass jar for low-level analysis and medium-level analysis **1 glass jar for backup low-level analysis and medium-level analysis**

Appendix C: General CLP Sample Collection Guidelines VOAs in Water



Regional guidance and/or specific Project Plan requirements will supersede the guidelines listed below.

Collect the following:

- At least two 40 mL glass containers with polytetrafluoroethylene (PTFE)-lined septa and open top screw-caps that are filled to capacity with no air bubbles, preserved to a pH of 2 with HCl, and cooled to 4°C (±2°C) immediately after collection. DO NOT FREEZE THE SAMPLES.
- If Selected Ion Monitoring (SIM) analysis is requested, at least two additional 40 mL glass containers with PTFE-lined septa and open top screw-caps that are filled to capacity with no air bubbles, preserved to a pH of 2 with HCl, and cooled to 4°C (±2°C) immediately after collection.

Test for Carbonates, Residual Chlorine, Oxidants, and Sulfides:

- It is very important that samplers obtain Regional guidance when testing and ameliorating for:
  - Carbonates;
  - Residual chlorine (e.g., municipal waters or industrial waste waters that are treated with chlorine prior to use or discharge); or
  - Oxidants.
- VOA samples containing carbonates react with the acid preservative causing effervescence (due to formation of carbon dioxide), which can cause loss of volatile analytes.
- Residual chlorine present in VOA samples can continue to react with dissolved organic matter. This continuous reaction may lead to inaccurate quantitation of certain analytes present in the sample at the time of collection.
- Residual chlorine and oxidants present in VOA samples can cause degradation of certain volatile analytes (e.g., styrene).

Perform the following for *Pre-Preserved* Vials:

1. Pour the sample slowly down the edge of the sample vial to avoid excess aeration or agitation of the sample during filling.
2. Fill the vial completely so that a reverse (convex) meniscus is present and ensure that there are no air bubbles present (either in the body or especially at the top of the vial).
3. Place the septum on the vial so that the PTFE side is in contact with the sample, and then firmly tighten the cap.
4. Gently flip the vial a few times to ensure that the sample is mixed with the acid preservative.
5. While holding the vial upright, gently tap the sample to check for air bubbles (either in the body or especially at the top of the vial).
6. If air bubbles are present, discard the sample and select a new vial in which to recollect a new sample. Repeat Steps 1 - 5 above.
7. Do NOT mix or composite samples for VOAs.
8. Cool sample to a temperature of 4°C (±2°C). Samplers should begin the cooling process in the field as samples are being collected. Double-bagged ice should be used. DO NOT FREEZE WATER SAMPLES.
9. Immediately transfer the vial to the sample shuttle (device that contains a "set" of VOA vials) once it has been collected. Do NOT allow ice to touch the vials.

Perform the Following for *Empty* Vials:

1. Rinse the vial with sample water prior to actual sample collection and preservation.



Regions vary in their approach to pre-rinsing and/or re-using sample vials (e.g., some Regions do not recommend pre-rinsing and/or re-use of pre-cleaned containers using sample water). Be sure to follow Regional guidance.

2. Add 1-2 mL of acid preservative to the vial. Check to ensure that the sample you are collecting requires a preservative (follow Regional guidance).
3. Pour the sample slowly down the edge of the sample vial to avoid excess aeration and agitation of the sample.
4. Fill the vial completely so that a reverse (convex) meniscus is present and ensure that there are no air bubbles present (either in the body or especially at the top of the vial).
5. Place the septum on the vial so that the PTFE side is in contact with the sample, and then firmly tighten the cap.
6. Gently flip the vial a few times to ensure that the sample is mixed with the acid preservative.
7. While holding the vial upright, gently tap the vial to check for air bubbles (either in the body or especially at the top of the vial).
8. If air bubbles are present, discard the sample and recollect a new sample using the same sample vial. Repeat Steps 1 - 7 above.
9. Check the recollected sample for air bubbles. If air bubbles are present, additional sample water may be added to the vial to eliminate air bubbles. If there are air bubbles after three consecutive attempts to eliminate air bubbles by the addition of sample water, the entire sample and sample vial should be discarded and a new sample collected.
10. Do NOT mix or composite samples for VOAs.
11. Cool sample to a temperature of 4°C (±2°C). Samplers should begin the cooling process in the field as samples are being collected. Double-bagged ice should be used. DO NOT FREEZE WATER SAMPLES.
12. Immediately transfer the vial to the sample shuttle (device which contains a "set" of VOA vials) once it has been collected. Do NOT allow ice to touch the vials.

Things to Remember:

- Samples must be shipped as soon as possible, preferably on the same day as sample collection to avoid exceeding sample holding times. If overnight transit is not possible, samples should be maintained at 2 - 4°C until they are shipped to the laboratory.
- If samples are not preserved (a requirement for certain analytes), the technical holding time is shortened to 7 days.

Appendix D: Sampling Techniques and Considerations

During a sampling event, the sampler is expected to follow prescribed sampling techniques. The sampler should also be aware of any special sampling considerations, contaminant issues, and sample compositing and mixing methods that could affect their sampling efforts.



Regional guidance will take precedence over any of the techniques and considerations listed below.

D.1 General Sampling Techniques

Information regarding surface water, sediment, soil, and groundwater sampling can be found in many documents including, but not limited to, the following sources:

- Compendium of ERT Surface Water and Sediment Sampling Procedures, EPA/540/P-91/005;
- Compendium of ERT Soil Sampling and Surface Geophysics Procedures, EPA/540/P-91/006;
- Compendium of ERT Groundwater Sampling Procedures, EPA/540/P-91/007;
- Quality Assurance Sampling Plan for Environmental Response (QASPER) software, Version 4.1, ERT; and
- *Requirements for the Preparation of Sampling and Analysis Plans*; United States Army Corps of Engineers, February 1, 2001, EM 200-1-3.

When working with potentially hazardous materials, samplers should follow USEPA and OSHA requirements, specific health and safety procedures, and DOT requirements.

D.2 Special Sampling Considerations

Samplers should refer to Regionally-developed SOPs to obtain specific procedures for properly collecting and preserving samples in the field. For additional guidance regarding sampling for VOAs in soil and water, see Appendices B and C. Samplers should obtain Regional guidance when testing and ameliorating for:

- Carbonates in VOA soil and water;
- Residual chlorine in VOA soil and water, or cyanide water;
- Oxidants in VOA soil and water; or
- Sulfides in cyanide.

D.3 Contaminant Sampling

Certain compounds can be detected in the parts-per-billion (ppb) and/or parts-per-trillion (ppt) range. Extreme care MUST be taken to prevent cross-contamination of these samples. The following precautions should be taken when trace contaminants are a concern:

- Disposable gloves should be worn each time a different location is sampled.
- When collecting both surface water and sediments, surface water samples should be collected first. This reduces the chance of sediment dispersal into surface water, and the resulting loss of surface water sample integrity.
- Sampling should occur in a progression from the least to the most contaminated area, if this information is known to the sampling team.
- Samplers should use equipment constructed of PTFE, stainless steel, or glass that has been properly pre-cleaned for collection of samples for trace organic and/or inorganic analyses. Equipment constructed of plastic or polyvinyl chloride (PVC) should NOT be used to collect samples for trace organic compound analyses.
- Equipment constructed of stainless steel should NOT be used to collect samples for trace metals analysis.

D.4 Sample Compositing

Sample compositing is a site-specific activity that must be conducted according to the SAP. Compositing is typically used for large sites under investigation to improve the precision (i.e., lower the variance) of the estimated average contaminant concentrations. **Samples for VOA analysis should NOT be composited to minimize loss of VOAs/analytes.**

Composite samples consist of a series of discrete grab samples that are mixed together to characterize the average composition of a given material. The discrete samples are usually of equal volume, but may be weighted to reflect an increased flow or volume. Regardless, all discrete samples must be collected in an identical manner and the number of grab samples forming a composite should be consistent. There are several compositing techniques that may be required such as:

- Flow-proportioned – Collected proportional to the flow rate during the compositing period by either a time-varying/constant volume or a time-constant/varying volume method. This technique is usually associated with wastewater or storm water runoff sampling.
- Time – Composed of a varying number of discrete samples collected at equal time intervals during the compositing period. This technique is typically used to sample wastewater and streams, and in some air sampling applications.
- Areal – Collected from individual grab samples collected in an area or on a cross-sectional basis. Areal composites are comprised of equal volumes of grab samples where all grabs are collected in an identical manner. This technique is typically used for estimating average contaminant concentrations in soils or sediments. This technique is useful when contaminants are present in nugget form (i.e., TNT chunks, lead shot, etc.), thus exhibiting large differences in concentration over a small sample area.
- Vertical – Collected from individual grab samples but taken from a vertical cross section. Vertical composites are comprised of equal volumes of grab samples where all grab samples are collected in an identical manner. Examples would include vertical profiles of a soil borehole or sediment columns.
- Volume – Collected from discrete samples whose aliquot volumes are proportional to the volume of sampled material. Volume composites are usually associated with hazardous waste bulking operations where the sample represents combined or bulked waste.

When compositing solid samples (i.e., sediment, soil, or sludge) for analysis of compounds present in trace quantities, use a stainless steel or PTFE bowl and spatula.

D.5 Sample Mixing and Homogenizing

Mixing of the sample for the remaining parameters is necessary to create a representative sample media. It is extremely important that solid samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample location. Please refer to the project-specific SAP regarding instructions on removal of any extraneous materials (e.g., leaves, sticks, rocks, etc.). The mixing technique will depend on the physical characteristics of the solid material (e.g., particle size, moisture content, etc.). The mixing container should be large enough to hold the sample volume and accommodate the procedures without spilling. Both the mixing container (generally a bowl or tray) and the mixing implement should be properly decontaminated before use. Samples should be homogenized according to procedures listed in the project-specific SAP.

Samples for VOA analysis should not be mixed to minimize loss of volatile analytes.

Table D-1 provides a short procedure for mixing a soil sample with a small particle size (less than 1/4 in) and filling sample containers in the field.

Table D-1. Mixing a Sample and Filling Sample Containers

Step	Action
1	Roll the contents of the compositing container to the middle of the container and mix.
2	Quarter the sample and move to the sides of the container.
3	Mix each quarter individually, then combine and mix OPPOSITE quarters, then roll to the middle of the container.
4	Mix the sample once more, and then quarter the sample again.
5	Mix each quarter individually, then combine and mix ADJACENT corners, then roll to the middle of the container. The goal is to achieve a consistent physical appearance before sample containers are filled.
6	Flatten piled material into an oblong shape.
7	Using a flat-bottomed scoop, collect a strip of soil across the entire width of the short axis and place it into a sample container.
8	Repeat Step 7 at evenly-spaced intervals until the sample containers are filled.
9	Record the approximate quantity of each subsample in the field log book.

Appendix E: Sampling Checklists

Appendix E-1: Personnel Preparation Checklist

(Page 1 of 1)

Personnel Briefing	Yes	No	Comments:
1. Did you review sampling team responsibilities and identify individual(s) responsible for corrective actions?			
2. Did you ensure that you have met the appropriate personal safety and protection requirements?			
3. Did you identify sampling locations and receive permission to access them, as appropriate?			
4. Did you contact the appropriate utility companies PRIOR to the start of sampling?  By law, utility companies must be contacted prior to the start of digging/sampling so that any underground utilities (gas lines, water lines, electrical lines, etc.) can be marked. A list of one-call centers for each state may be found at: http://www.digsafely.com/contacts.htm.			
5. If sampling on private property, do you have sample receipts to provide to the property owner for all samples taken and removed from the property?			
6. Have you determined the number and type of samples to be collected?			
7. Did you review sample collection methods?			
8. Have you reviewed sample container requirements?			
9. Did you review decontamination requirements, procedures, and locations?			
10. Did you determine holding times and conditions?			
11. Did you determine Performance Evaluation (PE) and Quality Control (QC) sample requirements?			
12. Have you obtained shipping cooler temperature blanks, if required?			
13. Did you review sample label and tag requirements?			
14. Did you review Traffic Report/Chain of Custody (TR/COC) Record and custody seal requirements?			
15. Have you obtained the laboratory name, shipping addresses, and telephone number?			
16. Did you review cooler return instructions?			
17. Have you obtained shipping company information (name, telephone number, account number, pickup schedule)?			
18. Have you obtained shipping schedules?			
19. Did you review shipment reporting requirements and the appropriate contact names and telephone numbers for reporting?			
20. Have you included any sampler comments regarding sampling issues (e.g., low volumes, matrix, suspected concentrations based on field measurements)?			

Appendix E-2: General Sample Collection Checklist

(Page 1 of 1)

General Sample Collection	Yes	No	Comments:
1. Did you identify and mark the sampling location with buoys, flags, or stakes according to the sampling plans, maps, and grids?			
2. If the sampling location is inaccessible, did you contact the appropriate field or Regional personnel for instructions?			
3. Did you use the correct sampling equipment?			
4. Did you follow the correct decontamination procedures?			
5. Did you follow the correct collection procedures?			
6. Did you use the correct sample containers for each sample collected?			
7. Did you collect the correct volume for each sample?			
8. Did you collect the correct type of sample, including primary samples and Quality Control (QC) samples?			
9. Did you properly preserve each sample collected?			
10. Did you correctly document and label each sample with all necessary information?  Under no circumstances should the site name appear on any documentation being sent to the laboratory.			
11. If sampling on private property, did you provide a sample receipt to the owner of the property for all samples taken and removed from the property?			

Appendix E-3: Completing Field Logbook Checklist

(Page 1 of 1)

Completing Field Logbook	Yes	No	Comments:
1. Did you use waterproof ink when writing in the field logbook?			
2. Did you document sampling project information such as: • Project name, ID, and location; • Names of samplers; • Geological observations, including maps; • Atmospheric conditions; • Field measurements; and • Sampling dates, times, and locations?  Under no circumstances should the site name appear on any documentation being sent to the laboratory.			
3. Did you record sampling activity information such as: • Sampling dates and times; • Sample identifications; • Sample matrices; • Sample descriptions (e.g., odors and/or colors); • Number of samples taken; • Sampling methods/equipment; and • Description of QC samples?			
4. Did you document any and all deviations from the sampling plan?			
5. Did you document any and all difficulties in sampling and/or any unusual circumstances?			
6. Were all errors corrected by crossing a line through the error, initialing the error, dating the error, and then adding the correct information?			

Appendix E-4: Completing Handwritten Sample Labels Checklist

(Page 1 of 1)

Completing Handwritten Sample Labels	Yes	No	Comments:
1. Did the Region provide CLP Sample Numbers and SMO-assigned Case Numbers?			
2. If additional CLP Sample Numbers were needed, did you contact the appropriate Regional personnel?			
3. Were the CLP Sample Numbers and SMO-assigned Case Numbers on the labels correct? Organic CLP Sample Numbers begin with the Regional letter code, followed by letters and numbers. Inorganic CLP Sample Numbers begin with "M", followed by the Regional letter code, and then letters and numbers.  The following characters are not used in generating CLP Sample Numbers and should never appear on any paperwork send to the laboratory: I; O; U; and V. Also, the last character of a CLP Sample Number will never be a letter.			
4. Were samples uniquely numbered and designated to only one sample?  Samples collected for total metal and dissolved metal analyses must receive separate, unique, CLP Sample Numbers.			
5. Were Quality Control (QC) samples numbered accordingly?			
6. Were the specific requirements followed for total and dissolved metals analysis, QC and Performance Evaluation (PE) samples, and SW-846 Method 5035A?			
7. Were all temperature blanks labeled with "TEMPERATURE BLANK"?			
8. Was a sample label containing the CLP Sample Number, SMO-assigned Case Number, location, concentration, preservative, and the fraction/analysis, attached to each sample bottle or container as the sample was collected?  Under no circumstances should the site name appear on any documentation being sent to the laboratory.			
9. Was clear tape placed over the sample labels to protect the labels from moisture and to help the labels adhere to the sample bottle?			
10. Were all errors corrected by crossing a line through the error, initialing the error, dating the error, and then adding the correct information?			

Appendix E-5: Completing Handwritten Sample Tags & Custody Seals Checklists

(Page 1 of 1)

Completing Handwritten Sample Tags	Yes	No	Comments:
1. Was waterproof ink used on the sample tags?			
2. If Regionally required for individual sample containers, was the project code on the sample tag completed?			
3. Was the station number on the sample tag completed?			
4 Was the date filled in using the format MM/DD/YYYY?			
5. Was the time of sample collection indicated in military time format HH:MM?			
6. Was the box checked indicating composite or grab sample?			
7. Was the station location on the sample tag completed?			
8. Did you indicate whether or not the sample was preserved by checking "yes" or "no?"			
9. Was the appropriate analysis indicated on the sample tag?			
10. Were the appropriate CLP Sample Number and SMO-assigned Case Number indicated and cross-referenced with the numbers on the sample label?			
11. Did you sign the sample tags?			
12 Did you attach the sample tag to the neck of the sample bottle with striang, stretch string, or wire (recommended method)?  Do **NOT** use wire to attach a sample tag to a metal sample.			
13. Were all errors corrected by crossing a line through the error, initialing the error, dating the error, and then adding the correct information?			

Completing Custody Seals	Yes	No	Comments:
1. Did you sign and date the custody seal?			
2. Did you attach a completed custody seal to the sample bottle, container, or plastic bag, placing the seal over the cap or lid of each sample bottle or container or on the bag opening such that it will be broken if the sample bottle, container, or bag is opened or tampered with?			
3. As appropriate, did you attach the completed custody seal to the sample shipping container or cooler, placing the seal such that it will be broken if the container or cooler is opened or tampered with?			
4. Were all errors corrected by crossing a line through the error, initialing the error, dating the error, and then adding the correct information?			

Appendix E-6: Packing Sample Container Checklist

(Page 1 of 1)

Packing Sample Container	Yes	No	Comments:
1. Did you follow all State, Federal, Department of Transportation (DOT), and International Air Transportation Association (IATA) regulations governing the packaging of environmental and hazardous samples?  If samples contain methanol preservation (e.g., samples to be analyzed by SW-846 Method 5035A), refer to the packaging instructions in Appendix A.			
2. Were all CLP Sample Numbers, SMO-assigned Case Numbers, fractions/analyses, labels, tags, and custody seals attached to the correct sample containers?			
3. Was an inventory conducted of CLP Sample Numbers, SMO-assigned Case Numbers, fractions/analyses, and containers, and verified against the TR/COC Records?			
4. Were the correct number and type of Performance Evaluation (PE) and Quality Control (QC) samples collected?			
5. Were all sample containers sealed in clear plastic bags with the sample label and tag visible through the packaging?			
6. Were all soil/sediment samples known to contain dioxin securely enclosed in metal cans (e.g., paint cans) with the lids sealed?			
7. Was suitable absorbent packing material placed around the sample bottles or containers?			
8. Were the outsides of metal containers labeled properly with the CLP Sample Number, SMO-assigned Case Number, and the fraction/analysis of the sample inside?			

Appendix E-7: Packing Shipping Container Checklist

(Page 1 of 1)

Packing Shipping Container	Yes	No	Comments:
1. Were you shipping samples in a clean waterproof metal or hard plastic ice chest or cooler in good condition?			
2. Were all non-applicable labels from previous shipments removed from the container?			
3. Were all inside and outside drain plugs closed and covered with suitable tape (e.g., duct tape)?			
4. Was the inside of the cooler lined with plastic (e.g., large heavy-duty garbage bag)?			
5. Was the lined shipping cooler packed with noncombustible absorbent packing material?			
6. Were sample containers placed in the cooler in an upright position not touching one another?			
7. Was a sample shipping cooler temperature blank included in the cooler?			
8. Did the documentation in the cooler only address the samples in that cooler?			
9. Was the site name absent from all documentation?  Under no circumstances should the site name appear on any documentation being sent to the laboratory.			
10. Was there sufficient packing material around and in between the sample bottles and cans to avoid breakage during transport?			
11. If required, was double-bagged ice placed on top and around sample bottles to keep the samples cold at 4°C (± 2° C)?  Do Not Pack Loose Ice Into the Cooler!			
12. Was the top of the plastic liner fastened and secured with tape?			
13. Was a completed custody seal placed around the top of the fastened plastic liner (if required by the Region)?			
14. Were all sample documents enclosed within the cooler (e.g., TR/COC Record and cooler return instructions) in a waterproof plastic bag?			
15. Was the plastic bag, containing the documentation, taped to the underside of the cooler lid?			
16. Were cooler return instructions and airbills, if required, taped to the underside of the cooler lid?			
17. Was the return address of the cooler written with permanent ink on the underside of the cooler lid?			
18. Was tape placed around the outside of the entire cooler and over the hinges?			
19. Were the completed custody seals placed over the top edge of the cooler so the cooler cannot be opened without breaking the seals?			
20. Was the return address label attached to the top left corner of the cooler lid?			
21. Were instructional labels attached to the top of the cooler, as necessary (e.g., "This End Up," "Do Not Tamper With," or "Environmental Laboratory Samples")?			
22. If shipping hazardous samples, were the correct labels attached to the cooler (e.g., "Flammable Liquids", "Caution", or "Poison")?			
23. If shipping samples containing methanol as a preservative (e.g., samples to be analyzed by SW-846 Method 5035A), was a label used to indicate methanol, the United Nations (UN) identification number for methanol (UN 1230), and Limited Quantity?			

Appendix E-8: Shipping & Reporting CLP Samples Checklist

(Page 1 of 1)

Shipping CLP Samples	Yes	No	Comments:
1. Did you follow all State, Federal, Department of Transportation (DOT), and International Air Transportation Association (IATA) regulations governing the shipment of environmental and hazardous samples?			
2. Was a separate airbill filled out for each cooler being shipped?			
3. Was the airbill filled out completely, including correct laboratory name, address, and telephone number, identification of recipient as "Sample Custodian," and appropriate delivery option (e.g., overnight or Saturday)?			
4. Was the completed airbill attached to the top of the cooler with the correct laboratory address?			
5. If more than one cooler was being shipped to the same laboratory, were they marked as "1 of 2," "2 of 2," etc.?			
6. Were the samples being shipped "overnight" through a qualified commercial carrier?			
Reporting CLP Samples	**Yes**	**No**	**Comments:**
1. Did you contact the Contract Laboratory Program Sample Management Office (SMO) on the same day samples were shipped?			
2. If the samples were shipped after 5:00 PM Eastern Time (ET), were they reported to the RSCC (or designee) or to SMO by 8:00 AM ET the following business day?			
3. Did you notify the RSCC (or designee) or SMO so that SMO will receive the delivery information by 3:00 PM ET on Friday for sample shipments that will be delivered to the laboratory on Saturday?			
4. Did you provide the RSCC (or designee) or SMO with: • Your name, phone number, and Region number; • Case Number of the project; • Exact number of samples, matrix(ces), concentration(s), and type of analysis; • Laboratory(ies) to which the samples were shipped; • Carrier name and airbill number; • Date of shipment; • Date of next shipment; and • Any other information pertinent to the shipment?			

Appendix F: Glossary

Analyte -- The element, compound, or ion that is determined in an analytical procedure; the substance or chemical constituent of interest.

Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the United States Environmental Protection Agency's (USEPA's) Office of Superfund Remediation and Technology Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).

Aroclor -- Polychlorinated biphenyls (PCBs) or a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl and a general chemical formula of $C_{12}H_{10-x}Cl_x$. PCBs, commercially produced as complex mixtures containing multiple isomers at different degrees of chlorination, were marketed in North America under the trade name Aroclor.

Case -- A finite, usually predetermined, number of samples collected over a given time period from a particular site. Case Numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) -- Initiated in December 1980, CERCLA provided broad federal authority to respond directly to the release or possible release or hazardous substances that may endanger human health or the environment. CERCLA also established a trust fund to provide for cleanup when no responsible party could be identified; hence CERCLA is commonly referred to as "Superfund".

Contract Laboratory Program (CLP) -- A national program of commercial laboratories under contract to support the USEPA's nationwide efforts to clean up designated hazardous waste sites by providing a range of chemical analytical services to produce environmental data of known and documented quality. This program is directed by USEPA's Analytical Services Branch (ASB).

Contract Laboratory Program Project Officer (CLP PO) -- Monitors technical performance of the contract laboratories in each Region.

Contract Laboratory Program Sample Management Office (CLP SMO) -- A contractor-operated facility operated under the CLP, awarded and administered by the USEPA, which provides necessary management, operations, and administrative support to the CLP. SMO coordinates and schedules sample analyses, tracks sample shipments and analyses, receives and tracks data for completeness and compliance, and processes laboratory invoices.

Custody Seal -- An adhesive label or tape that is used to seal a sample bottle or container that maintains chain-of-custody and that will break if the sample bottle or container is opened or tampered with.

Cyanide (Total) -- Cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

Data Quality Objective (DQO) -- The requirements established to maintain the quality of the data being collected.

Data Validation -- Data validation is based on Region-defined criteria and limits, professional judgment of the data validator, and (if available) the Quality Assurance Project Plan (QAPP) and Sampling and Analysis Plan (SAP).

Equipment Blank -- A sample used to check field decontamination procedures. See Field Blank.

Field Blank -- Any blank sample that is submitted from the field. Each field blank is assigned its own unique USEPA Sample Number. A Field Blank checks for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsates, equipment blanks, etc.

Field Duplicate -- Checks reproducibility of laboratory and field procedures and indicates non-homogeneity.

Field Operations Reporting Management System (FORMS) II Lite -- A stand-alone, Windows-based software application that enables samplers to automatically create and generate sample documentation both prior to and during a sampling event.

Field QC Sample -- Used to detect for contamination or error in the field.

Field Sample -- Primary sample material taken out in the field from which other samples, such as duplicates or split samples are derived. A field sample can be prepared in the field and sent for analysis in one or multiple containers, and is identified by a unique EPA Sample Number.

Field Sampling Plan (FSP) -- Developed to outline the actual steps and requirements pertaining to a particular sampling event, and explains, in detail, each component of the event to all involved samplers.

Holding Time -- The elapsed time expressed in hours, days, or months from the date of collection of the sample until the date of its analysis.

> **Contractual** -- The lengths of time that the CLP laboratory must follow to comply with the terms of the contract, and are described in the CLP analytical services Statements of Work (SOWs).
>
> **Technical** -- The maximum lengths of time that samples may be held from time of collection to time of preparation and/or analysis and still be considered valid.

Laboratory Blank -- See Method Blank.

Laboratory Duplicate -- A sample required by the laboratory's contract to check the precision of inorganic analyses.

Laboratory QC Sample -- An additional volume of an existing sample, as required by the laboratory's contract, used to detect contamination or error in the laboratory's practices.

Matrix -- The predominant material of which a sample to be analyzed is composed.

Matrix Spike (MS) -- Sample required by the laboratory's contract to check the accuracy of organic and inorganic analyses. It is an aliquot of a sample (water or soil) that is fortified (spiked) with known quantities of a specific compound and subjected to the entire analytical procedure. See Matrix Spike Duplicate.

Matrix Spike Duplicate (MSD) -- Sample required by the laboratory's contract to check the accuracy and precision of organic analyses. It is a second aliquot of the same matrix as the Matrix Spike (MS) that is spiked to determine the precision of the method. See Matrix Spike.

Method Blank -- An analytical control consisting of all reagents, internal standards and surrogate standards [or System Monitoring Compounds (SMCs) for volatile organic analysis], that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination, also referred to as laboratory blank when defining the level of laboratory contamination.

Performance Evaluation (PE) Sample -- A sample of known composition provided by the USEPA for contractor analysis. Used by USEPA to evaluate contractor performance.

Pesticides -- Substances intended to repel, kill, or control any species designated a "pest", including weeds, insects, rodents, fungi, bacteria, and other organisms. Under the CLP, only organochlorine pesticides are analyzed (e.g., DDT, Dieldrin, Endrin, etc.).

Polychlorinated Biphenyls (PCBs) -- A group of toxic, persistent chemicals used in electrical transformers and capacitors for insulating purposes, and in gas pipeline systems as a lubricant. The sale and new use of PCBs were banned by law in 1979.

Quality Assurance (QA) -- An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

Quality Assurance Project Plan (QAPP) -- Document written to meet requirements outlined in the document *EPA Guidance for Quality Assurance Project Plans* (EPA QA/R-5). Prepared in advance of field activities and used by samplers to develop any subsequent plans such as the Sampling Analysis Plan (SAP) or the Field Sampling Plan (FSP).

Quality Control (QC) -- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality.

Regional Sample Control Center (RSCC) Coordinator -- In most Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

Regional Site Manager -- Coordinates the development of data quality objectives and oversees project-specific remedial or removal contractors, State officials, or private parties conducting site sampling efforts.

Rinse Blank -- A sample used to check decontamination procedures. Also see Field Blank.

Routine Analytical Service (RAS) -- The standard inorganic and organic analyses available through the CLP.

Sample -- A discrete portion of material to be analyzed that is contained in single or multiple containers, and identified by a unique Sample Number.

Sample Delivery Group (SDG) – A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received; or
- Each 20 field samples (excluding PE samples) within a Case; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG. Sample may be assigned to SDGs by matrix (e.g., all soil samples in one SDG, all water samples in another) at the discretion of the laboratory.

Sample Label -- An identification label attached to a sample bottle or container to identify the sample.

Sample Number -- A unique number used to identify and track a sample. This number can be recorded on a sample label or written on the sample bottle or container using indelible ink.

Sample Tag -- A tag attached to a sample that identifies the sample and maintains chain-of-custody.

Sampling Analysis Plan (SAP) -- A document that explains how samples are to be collected and analyzed for a particular sampling event.

Semivolatile Organic Analyte (SVOA) -- A compound amenable to analysis by extraction of the sample using an organic solvent.

Statement of Work (SOW) -- A document that specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

Superfund -- The program operated under the legislative authority of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and Superfund Amendments and Reauthorization Act (SARA) that funds and carries out USEPA removal and remedial activities at hazardous waste sites. These activities include establishing the National Priorities List (NPL), investigating sites for inclusion on the list, determining their priority, and conducting and/or supervising cleanup and other remedial actions.

Superfund Amendments and Reauthorization Act (SARA) -- The 1986 amendment to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

Traffic Report/Chain of Custody (TR/COC) Record -- A record that is functionally similar to a packing slip that accompanies a shipment of goods. Used as physical evidence of sample custody and functions as a permanent record for each sample collected.

Trip Blank -- A sample used to check for contamination during sample handling and shipment from field to laboratory. Also see Field Blank.

Volatile Organic Analyte (VOA) -- A compound amenable to analysis by the purge-and-trap technique. Used synonymously with the term purgeable compound.

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Attachment B

NYSDOH Guidance for Evaluating Soil Vapor Intrusion in the State of New York

FINAL

Guidance for Evaluating Soil Vapor Intrusion in the State of New York

October 2006

Prepared by:

NEW YORK STATE DEPARTMENT OF HEALTH
Center for Environmental Health
Bureau of Environmental Exposure Investigation

Soil Vapor Intrusion Guidance Release History

October 2006 — Current version
February 2005 — Public comment draft

Contact Information

Comments and questions on the guidance may be sent to the following:

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New York State Departments of Health and Environmental Conservation — Web Sites on Soil Vapor Intrusion

This guidance, policy documents, training documents, fact sheets, etc. are available to the public on the following web sites:

New York State Department of Health
http://www.health.state.ny.us/environmental/indoors/vapor_intrusion

New York State Department of Environmental Conservation
http://www.dec.state.ny.us/website/der/guidance/vapor/index.html

As new information becomes available (e.g., revisions or amendments to the guidance, new fact sheets, etc.), these web sites will be updated accordingly.

Preface

This guidance has been prepared by the New York State Department of Health (NYSDOH) in consultation with the New York State Department of Environmental Conservation (NYSDEC) — collectively referred to as "the State" throughout this document. It is intended as general guidance for parties evaluating soil vapor intrusion in the State of New York. The guidance is not a regulation, rule or requirement.

The guidance describes the State's methodology for evaluating soil vapor intrusion at a site. It reflects our experience in conducting soil vapor intrusion investigations and presents a reasonable and practical approach to identifying and addressing current and potential human exposures to contaminated subsurface vapors associated with known or suspected volatile chemical contamination. The approach presented is analogous to the approach taken when investigating contamination in other environmental media (e.g., groundwater, soil, etc.) and addressing corresponding exposure concerns.

The guidance is organized into five sections:

Section 1 introduces the concept of soil vapor intrusion, associated human exposure issues, factors affecting soil vapor intrusion, factors affecting indoor air quality, and the general approach recommended to evaluating vapor intrusion;

Section 2 provides guidance on collecting appropriate and relevant data that can be used to identify current or potential human exposures;

Section 3 discusses how the investigation data are evaluated, recommends actions based on the evaluation, and presents tools that are used when determining appropriate actions to address exposures;

Section 4 provides an overview of soil vapor intrusion mitigation methods and basic recommendations pertaining to their selection for use, installation and design, post-mitigation testing, operation, maintenance and monitoring, termination of operation, and annual certification; and

Section 5 describes outreach techniques commonly used to inform the community about soil vapor intrusion issues.

The State recommends that the guidance be considered anywhere soil vapor intrusion is evaluated in the State of New York — whether the evaluation is undertaken voluntarily by a corporation, a municipality, or private citizen, or whether it is performed under one of the State's environmental remediation programs.

PLEASE NOTE:

- While soil vapor intrusion can also occur with "naturally-occurring" subsurface gases (e.g., radon, methane and hydrogen sulfide), the document discusses soil vapor intrusion in terms of environmental contamination only.
- The guidance document addresses soil vapor intrusion. However, vapor intrusion can also occur through direct volatilization of contaminants from groundwater into indoor air. This can occur when, for example, a basement slab is in contact with contaminated groundwater, contaminated groundwater enters (floods) a basement or crawl space, or contaminated groundwater enters a sump pit drainage system. In such cases, volatile

chemicals can be transferred directly from groundwater to indoor air without the intervening contamination of soil vapor. Although exposures of this nature are not discussed in this guidance, they should be addressed on a site-specific and building-specific basis.

- Throughout the guidance references are made to specific brands of field equipment. These references are for discussion purposes only and are intended to be illustrative. They should not be interpreted as endorsements by the State of any one company or their products.

ACRONYMNS and ABBREVIATIONS

ASTM	American Society for Testing and Materials
ATSDR	Agency for Toxic Substance and Disease Registry
BASE	Building Assessment and Survey Evaluation
BTSA	[NYSDOH] Bureau of Toxic Substance Assessment
CME	Continuing Medical Education
CSEMs	Case Studies in Environmental Medicine
DUSR	Data Usability Summary Report
ELAP	Environmental Laboratory Approval Program
EPA	United States Environmental Protection Agency
GC	Gas Chromatograph
HEI	Health Effects Institute
HVAC	Heating, Ventilating and Air-conditioning
mcg/m^3	micrograms per cubic meter
MeCl	Methylene Chloride
MEK	Methyl Ethyl Ketone; 2-Butanone
MTBE	Methyl-*tert*-Butyl Ether
NAPL	Non-Aqueous Phase Liquid
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OM&M	Operation, Maintenance and Monitoring
OSHA	Occupational Safety and Health Administration
OVM	Organic Vapor Monitor
PCBs	Polychlorinated Biphenyls
PCE	Tetrachloroethene or Perchloroethylene
PID	Photoionization Detector
QA/QC	Quality Assurance/Quality Control
RIOPA	Relationship of Indoor, Outdoor, and Personal Air
SF_6	Sulfur Hexafluoride
SSD	Sub-slab Depressurization System
SIM	Selective Ion Monitoring
SMD	Sub-Membrane Depressurization
SVE	Soil Vapor Extraction
SVOCs	Semi-volatile Organic Compounds
TAL	Target Analyte List
TCA	Trichloroethane
TCDD	Tetrachlorodibenzo-*p*-Dioxin Equivalents
TCE	Trichloroethene
TCL	Target Compound List
VOCs	Volatile Organic Compounds

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Section 1: Introduction

This section introduces the concept of soil vapor intrusion, associated human exposure issues, factors affecting soil vapor intrusion, factors affecting indoor air quality, and the general approach to evaluating vapor intrusion.

1.1 Soil vapor intrusion

The phrase "soil vapor intrusion" refers to the process by which volatile chemicals migrate from a subsurface source into the indoor air of buildings. Soil vapor, also referred to as soil gas, is the air found in the pore spaces between soil particles (Figure 1.1). Primarily because of a difference between interior and exterior pressures, soil vapor can enter a building through cracks or perforations in slabs or basement floors and walls, and through openings around sump pumps or where pipes and electrical wires go through the foundation. For example, heating, ventilation or air-conditioning (HVAC) systems and/or the operation of large mechanical appliances (e.g., exhaust fans, dryers, etc.) may create a negative pressure that can draw soil vapor into the building. This intrusion is similar to how radon gas enters buildings from the subsurface.

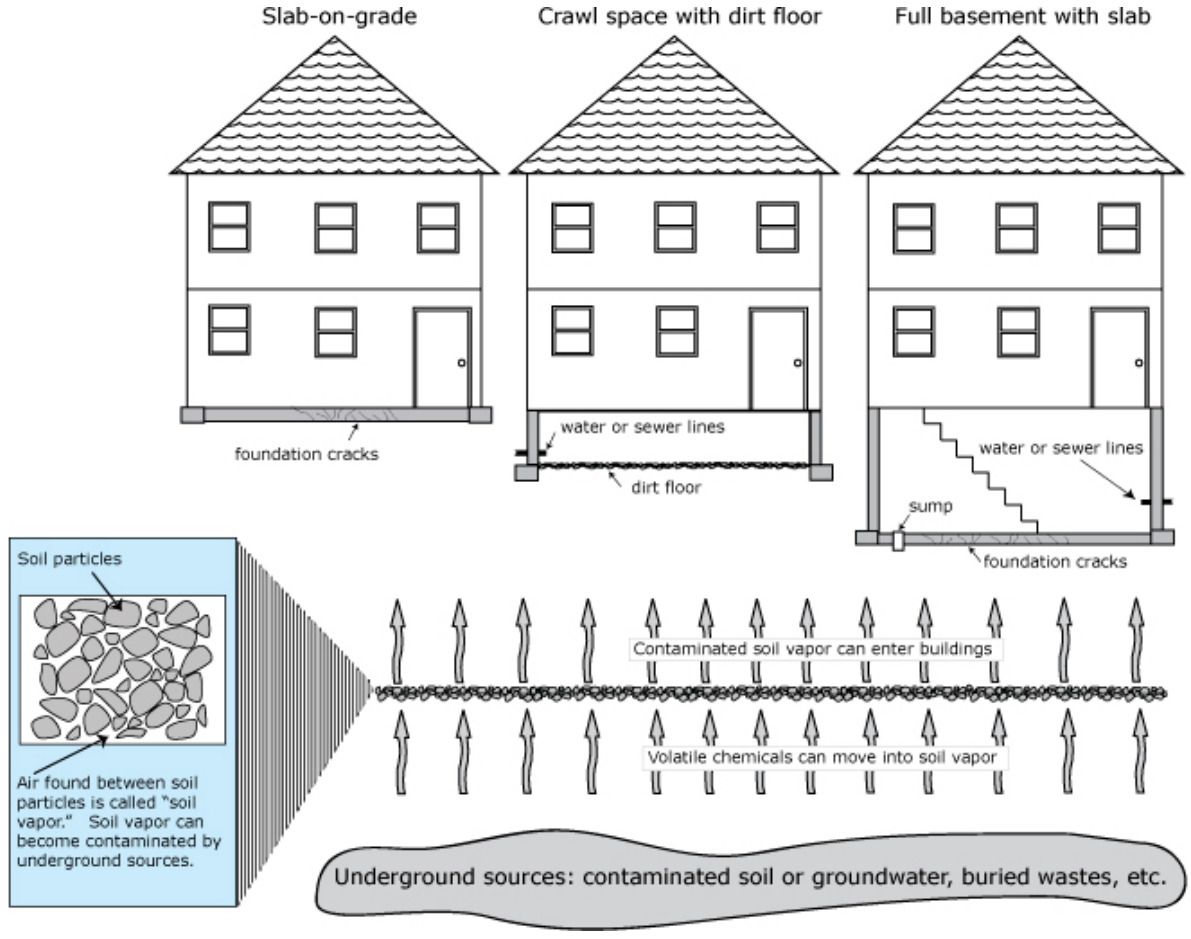


Figure 1.1
Generalized diagram of soil vapor intrusion

Soil vapor can become contaminated when chemicals evaporate from subsurface sources. Chemicals that can emit vapors are called "volatile chemicals." Volatile chemicals include volatile organic compounds (VOCs), some semi-volatile organic compounds (SVOCs), and some inorganic substances such as elemental mercury. Subsurface sources of volatile chemicals can include the following:

a. groundwater or soil that contains volatile chemicals;

b. non-aqueous phase liquid (NAPL);

c. buried wastes; and

d. underground storage tanks or drums.

If soil vapor is contaminated and enters a building, indoor air quality may be affected.

When contaminated vapors are present in the zone directly next to or under the foundation of a building, vapor intrusion is possible. Soil vapor can enter a building whether the building is old or new, or whether it is on a slab or has a crawl space or basement (Figure 1.1). However, the subsurface source of the contaminated vapor (e.g., contaminated soil or groundwater) does not need to be directly beneath a structure to contaminate the vapor immediately beneath the building's foundation (as suggested in Figure 1.1).

1.2 Soil vapor intrusion and human exposure

Humans can be exposed to contaminated soil vapor when the vapor is drawn into the building due to pressure differences [Section 1.1] and mixed with the indoor air. Inhalation is the primary route of exposure, or the manner in which the volatile chemicals, once in the indoor air, actually enter the body.

Both current and potential exposures are considered when evaluating soil vapor intrusion at sites (i.e., locations of suspected or known environmental contamination). *Current* exposures exist when vapor intrusion is documented in an occupied building. *Potential* exposures exist when volatile chemicals are present in the vapor phase beneath a building, but have not affected indoor air quality due to current site conditions. Potential exposures also exist when there is a chance that contaminated soil vapors may move beneath existing buildings not currently affected, when indoor air is affected but the building is currently unoccupied, or when there is a chance that new buildings can be built over existing subsurface vapor contamination.

Exposure to a volatile chemical due to vapor intrusion does not necessarily mean that health effects will occur. Whether or not a person experiences health effects depends on several factors, including the length of exposure (short-term or acute versus long-term or chronic), the amount of exposure (i.e., dose), the frequency of exposure, the toxicity of the volatile chemical and the individual's sensitivity to the chemical.

1.3 Factors affecting soil vapor migration and intrusion

Predicting the extent of soil vapor contamination from soil or groundwater contamination, as well as the potential for human exposure from soil vapor intrusion into buildings, is complicated by factors that can affect soil vapor migration and intrusion. For example, soil vapor contaminant plumes may not mimic groundwater contaminant plumes since different factors affect the migration pattern of each medium. In addition to the operation of HVAC systems, the operation of kitchen vents in restaurants or of elevators in office buildings may

induce pressure gradients that result in the migration of vapor-phase contaminants away from a groundwater source of vapors and toward these buildings. This is similar to when the pumping of production wells or water supply wells draws contaminated groundwater away from its natural flow path.

Factors that can affect soil vapor migration and intrusion generally fall into two categories: environmental and building factors. Examples of environmental factors are provided in Table 1.1, and examples of building factors in Table 1.2. These factors are considered when conducting an investigation of the soil vapor intrusion pathway [Section 2] and when evaluating the results [Section 3].

Table 1.1 Environmental factors that may affect soil vapor intrusion

Environmental Factor	Description
Soil conditions	Generally, dry, coarse-grained soils facilitate the migration of subsurface vapors and wet, fine-grained or highly organic soils retard migration.
Volatile chemical concentrations	The potential for vapor intrusion generally increases with increasing concentrations of volatile chemicals in groundwater or subsurface soils, as well as with the presence of NAPL.
Source location	The potential for vapor intrusion generally decreases with increasing distance between the subsurface source of vapor contamination and overlying buildings. For example, the potential for vapor intrusion associated with contaminated groundwater decreases with increasing depth to groundwater.
Groundwater conditions	Volatile chemicals dissolved in groundwater may off-gas to the vadose zone from the surface of the water table. If contaminated groundwater is overlain by clean water (upper versus lower aquifer systems or significant downward groundwater gradients), then vapor phase migration or partitioning of the volatile chemicals is unlikely. Additionally, fluctuations in the groundwater table may results in contaminant smear zones. The "smear zone" is the area of subsurface soil contamination within the range of depths where the water table fluctuates. Chemicals floating on top of the water table, such as petroleum components, can sorb onto soils within this zone as the water table fluctuates. Sorption of chemicals can influence their gaseous and aqueous phase diffusion in the subsurface, and ultimately the rate at which they migrate.
Surface confining layer	A surface confining layer (e.g., frost layer, pavement or buildings) may temporarily or permanently retard the migration of subsurface vapors to outdoor air. Confining layers can also prevent rainfall from reaching subsurface soils, creating relatively dry soils that further increase the potential for soil vapor migration.
Fractures in bedrock and/or tight clay soils	Fractures in bedrock and desiccation fractures in clay can increase the potential for vapor intrusion beyond that expected for the bulk, unfractured bedrock or clay matrix by facilitating vapor migration (in horizontal and vertical directions) and movement of contaminated groundwater along spaces between fractures.
Underground conduits	Underground conduits (e.g., sewer and utility lines, drains or tree roots, septic systems) with highly permeable bedding materials relative to native materials can serve as preferential pathways for vapor migration due to relatively low resistance to flow.
Weather conditions	Wind and barometric pressure changes and thermal differences between air and surrounding soils may induce pressure gradients that affect soil vapor intrusion.
Biodegradation processes	Depending upon environmental conditions (e.g., soil moisture, oxygen levels, pH, mineral nutrients, organic compounds, and temperature), the presence of appropriate microbial populations, and the degradability of the volatile chemical of concern, biodegradation in the subsurface may reduce the potential for vapor intrusion. For example, readily biodegradable chemicals in soil vapor may not migrate a significant distance from a source area while less degradable chemicals may travel farther.

Table 1.2 Building factors that may affect vapor intrusion

Building Factor	Description
Operation of HVAC systems, fireplaces, and mechanical equipment (e.g., clothes dryers or exhaust fans/vents)	Operation may create a pressure differential between the building or indoor air and the surrounding soil that induces or retards the migration of vapor-phase contaminants toward and into the building. Vapor intrusion can be enhanced as the air vented outside is replaced.
Heated building	When buildings are closed up and heated, a difference in temperature between the inside and outdoor air induces a stack effect, venting warm air from higher floors to the outside. Vapor intrusion can be enhanced as the air is replaced in the lower parts of the building.
Air exchange rates	The rate at which outdoor air replenishes indoor air may affect vapor migration into a building as well the indoor air quality. For example, newer construction is typically designed to limit the exchange of air with the outside environment. This may result in the accumulation of vapors within a building.
Foundation type	Earthen floors and fieldstone walls may serve as preferential pathways for vapor intrusion.
Foundation integrity	Expansion joints or cold joints, wall cracks, or block wall cavities may serve as preferential pathways for vapor intrusion.
Subsurface features that penetrate the building's foundation	Foundation perforations for subsurface features (e.g., electrical, gas, sewer or water utility pipes, sumps, and drains) may serve as a preferential pathway for vapor intrusion.

1.4 Factors affecting indoor air quality

Chemicals are a part of our everyday life. They are found in the household products we use and in items we bring into our homes. As such, chemicals are found in indoor air of homes not affected by intrusion of contaminated soil vapor. Examples of alternate sources of volatile chemicals in indoor air are given in Table 1.3. Similarly, volatile chemicals can be in the outdoor air that enters a home or place of business. Certain commercial and industrial facilities, such as gasoline stations and dry cleaners, and vehicle exhaust are examples of possible sources of volatile chemicals in outdoor air.

Commonly found concentrations of these chemicals in indoor and outdoor air are referred to as "background levels." These levels are generally determined from the results of samples collected in homes, offices and outdoor areas not known to be affected by external sources of volatile chemicals (for example, a home not known to be near a chemical spill, a hazardous waste site, a dry-cleaner, or a factory). Background sources of volatile chemicals are considered when conducting an investigation of the soil vapor intrusion pathway [Section 2] and when evaluating the results [Section 3].

Table 1.3 Alternate sources of volatile chemicals in indoor air

Source	Description
Outdoor air	Outdoor sources of pollution can affect indoor air quality due to the exchange of outdoor and indoor air in buildings through natural ventilation, mechanical ventilation or infiltration. Outdoor sources of volatile compounds include automobiles, lawn mowers, oil storage tanks, dry cleaners, gasoline stations, industrial facilities, etc.
Attached or underground garages	Volatile chemicals from sources stored in the garage (e.g., automobiles, lawn mowers, oil storage tanks, gasoline containers, etc.) can affect indoor air quality due to the exchange of air between the garage and indoor space.
Off-gassing	Volatile chemicals may off-gas from building materials (e.g., adhesives or caulk), furnishings (e.g., new carpets or furniture), recently dry-cleaned clothing, or areas (such as floors or walls) contaminated by historical use of volatile chemicals in a building. Volatile chemicals may also off-gas from contaminated groundwater that infiltrates into the basement (e.g., at a sump) or during the use of contaminated domestic well water (e.g., at a tap or in a shower).
Household products	Household products include, but are not limited to, cleaners, mothballs, cigarette smoke, paints, paint strippers and thinners, air fresheners, lubricants, glues, solvents, pesticides, fuel oil storage, and gasoline storage.
Occupant activities	For example, in non-residential settings, the use of volatile chemicals in industrial or commercial processes or in products used for building maintenance. In residential settings, the use of products containing volatile chemicals for hobbies (e.g., glues, paints, etc.) or home businesses. People working at industrial or commercial facilities where volatile chemicals are used may bring the chemicals into their home on their clothing.
Indoor emissions	These include, but are not limited to, combustion products from gas, oil and wood heating systems that are vented outside improperly, as well as emissions from industrial process equipment and operations.

1.5 General approach to evaluating soil vapor intrusion

Since no two sites are exactly alike, the approach to evaluating soil vapor intrusion is dependent upon site-specific conditions. A thorough understanding of the site, including its history of use, characteristics (e.g., geology, geography, identified environmental contamination, etc.) and potentially exposed populations, is used to develop an investigation plan. Existing information is reviewed to determine what data are available and what additional data should be collected (i.e., to guide the investigation). In addition, factors affecting soil vapor migration and intrusion [Section 1.3] and indoor air quality [Section 1.4] are also considered when both conducting an investigation [Section 2] and evaluating the results [Section 3].

This data gathering and review process should be repeated until each of the following questions can be answered:

[1] Are subsurface vapors contaminated (i.e., soil vapor as defined in Section 1.1, including vapors located immediately beneath the foundation or slab of a building)? If so, what are the nature and extent of contamination? What is/are the source(s) of the contamination?

[2] What are the current and potential exposures to contaminated subsurface vapors via soil vapor intrusion?

[3] What actions, if any, should be taken to prevent or mitigate exposures related to soil vapor intrusion and to remediate subsurface vapor contamination?

When determining what actions, if any, are appropriate to mitigate current or prevent future human exposures, all information known about a site is considered (i.e., a "whole picture" approach is taken) because each site presents its own unique set of circumstances. This information includes, but is not limited to, the following: nature and extent of contamination in all environmental media, factors affecting vapor migration and intrusion, current and future site uses, off-site land uses, presence of alternate sources of volatile chemicals, and completed or proposed remedial actions.

Actions taken to minimize or prevent exposures typically do not preclude the site from being used for a desired purpose or from being developed. If appropriate, mitigation systems can be installed at existing buildings or installed during the construction of new buildings. In many cases, installation of mitigation systems on new buildings may be a prudent, proactive action. The costs associated with installing a system at the time of a building's construction are often considerably less than the costs associated with retrofitting a system to the building after construction is completed. Furthermore, in many parts of New York State, the mitigation system would also address concerns about human exposures to radon. To learn more about radon in New York State, please refer to the Radon: Frequently Asked Questions Fact Sheet in Appendix H or visit the NYSDOH's web site at http://www.health.state.ny.us/nysdoh/radon/radonhom.htm or contact the NYSDOH's Radon Program at 1-800-458-1158.

1.6 Conceptual site model

In accordance with the NYSDEC's *Draft DER-10 Technical Guidance for Site Investigation and Remediation* (NYSDEC 2002), subsurface vapors and soil vapor intrusion should be included in an overall conceptual model for the site. As described in the NYSDEC's technical guidance, a conceptual site model should be used to develop a general understanding of the site to evaluate potential risks to public health and the environment and to assist in identifying and setting priorities for the activities to be conducted at the site. The conceptual site model also identifies potential sources of contamination, types of contaminants and affected media, release mechanisms and potential contaminant pathways, and actual/potential human and environmental receptors.

The components of a conceptual site model specific to soil vapor intrusion are provided throughout Section 1 of the guidance. The general approach for evaluating soil vapor intrusion described in Section 1.5 is analogous to the development of a conceptual site model specific to soil vapor intrusion. For additional information about the use of conceptual site models in the investigation and remediation of sites or a description of the conceptual site model process, the reader is referred to the NYSDEC's technical guidance.

1.7 Applicability of guidance

This guidance should be considered anywhere soil vapor intrusion is evaluated in the State of New York, whether the evaluation is being undertaken voluntarily by a corporation, a municipality, or private citizen, or under one of the state's environmental remediation programs.

1.7.1 Residential and non-residential settings

The guidance should be followed in residential and non-residential settings where people may be exposed involuntarily to chemicals from soil vapor intrusion.

1.7.2 Chlorinated and non-chlorinated volatile chemical sites

The guidance should be used when evaluating soil vapor intrusion at chlorinated and non-chlorinated volatile chemical sites, including petroleum hydrocarbon sites and manufactured gas plant sites. While the likelihood for exposures related to soil vapor intrusion may differ between sites due to site-specific conditions and chemical-specific properties, the extent of volatile chemical contamination and the nature of the contamination, these factors should be considered when developing the conceptual site model and implementing an investigation plan (as discussed in Sections 1.5 and 1.6). For example, if the conceptual site model suggests that soil vapor intrusion is not a concern at a petroleum hydrocarbon site due to biodegradation, the work plan might include the measurement of select bioparameters (e.g., oxygen, carbon dioxide, methane, etc.), along with the petroleum hydrocarbons, at varying depths to demonstrate bioattenuation in the vadose. The work plan might include sub-slab vapor sampling as well to demonstrate that conditions beneath nearby buildings are also resulting in bioattenuation of the petroleum hydrocarbons.

1.7.3 Current, new and past remedial sites

As discussed in the NYSDEC's Program Policy *DER-13: Strategy for Prioritizing Vapor Intrusion Evaluations at Remedial Sites in New York* (NYSDEC 2006), the soil vapor intrusion pathway will be evaluated at all completed, current and future remedial sites New York State. This soil vapor intrusion guidance document complements the NYSDEC's policy by providing recommendations on how to evaluate soil vapor intrusion. The combined goal of the policy and guidance documents is to conduct soil vapor intrusion evaluations as efficiently and effectively as possible at all remedial sites in New York.

1.8 Updates to the guidance

The investigation, evaluation, mitigation and remediation of soil vapor are evolving disciplines and this guidance document will be updated periodically, as appropriate. The history of the document's release is provided on the inside of the cover page. In addition, changes to the document are noted in Appendix A. The current version of the document supercedes previous versions. The current version of the guidance is available on the NYSDOH's web site (*http://www.health.state.ny.us/environmental/indoors/vapor_intrusion/*) or by contacting the NYSDOH's Bureau of Environmental Exposure Investigation [see Contact Information on the inside of the cover page]. Revisions or amendments to the guidance will be posted on the NYSDOH's web site.

Section 2: Investigation of the Soil Vapor Intrusion Pathway

Soil vapor is an environmental medium, like groundwater and soil, that should be characterized during the investigation of a site. This section provides guidance on collecting appropriate and relevant data that can be used to identify current or potential human exposures to contaminated subsurface vapors associated with a site. As discussed in Section 1.5, no two sites are exactly alike. Site-specific and/or building-specific conditions may warrant modifying the recommendations herein. Therefore, guidance provided in this section is presented in terms of general steps and strategies that should be applied when approaching an investigation of soil vapor intrusion.

2.1 Sites at which an investigation is appropriate

Data collected to date do not support the use of pre-determined concentrations of volatile chemicals (i.e., screening criteria) in either groundwater or soil to trigger a soil vapor intrusion investigation. Therefore, although the level of investigation may vary, the pathway should be investigated at any site with the following:

a. an existing subsurface source (e.g., on the basis of preliminary environmental sampling) or likely subsurface source (e.g., on the basis of known previous land uses) of volatile chemicals [Section 1.1]; and

b. existing buildings or the possibility that buildings may be constructed near a subsurface source of volatile chemicals.

2.2 Types of samples

The following are types of samples that are collected to investigate the soil vapor intrusion pathway:

a. subsurface vapor samples:

   1. *soil vapor* samples (i.e., soil vapor samples not beneath the foundation or slab of a building) and

   2. *sub-slab vapor* samples (i.e., soil vapor samples immediately beneath the foundation or slab of a building);

b. crawl space air samples;

c. indoor air samples; and

d. outdoor air samples.

The types of samples that should be collected depend upon the specific objective(s) of the sampling, as described below.

2.2.1 Soil vapor

Soil vapor samples are collected to determine whether this environmental medium is contaminated, characterize the nature and extent of contamination, and identify possible sources of the contamination. Our experience to date indicates soil vapor results alone typically cannot be relied upon to rule out sampling at nearby buildings. For example, concentrations of volatile chemicals in sub-slab vapor samples have been substantially higher (e.g., by a factor of 100 or more) than concentrations found in nearby soil vapor

samples (e.g., collected at 8 feet below grade near the building). This may be due to differences in factors such as soil moisture content and pressure gradients. Therefore, exposures are evaluated primarily based on sub-slab vapor, indoor air and outdoor air sampling results and soil vapor results are primarily used as a tool to guide these investigations.

Soil vapor sampling results are also used when evaluating the effectiveness of direct or indirect measures to remediate contaminated subsurface vapors. (Soil vapor extraction is an example of a direct remedial measure, and groundwater pumping and treating an indirect measure.)

2.2.2 Sub-slab vapor

Sub-slab vapor samples are collected to characterize the nature and extent of soil vapor contamination immediately beneath a building with a basement foundation and/or a slab-on-grade. Sub-slab vapor sampling results are used in conjunction with indoor air and outdoor air sampling results when evaluating the following:

a. *current* human exposures;

b. the potential for *future* human exposures (e.g., if the structural integrity of the building changes or the use of the building changes); and

c. site-specific attenuation factors (i.e., the ratio of indoor air to sub-slab vapor concentrations).

Sub-slab vapor samples are often collected after soil vapor characterization and/or other environmental sampling (e.g., soil and groundwater characterization) indicate they are warranted. Sub-slab samples are typically collected concurrently with indoor and outdoor air samples. However, outside of the heating season, sub-slab vapor samples may be collected independently depending on the sampling objective (e.g., to characterize the extent of subsurface vapor contamination outside of the heating season to develop a more comprehensive, focused investigation plan for the heating season).

2.2.3 Crawl space air

Similar to sub-slab vapor samples, crawl space air samples are collected to characterize the nature and extent of contamination immediately beneath a building with a crawl space foundation. Crawl space air sampling results are used in conjunction with indoor air and outdoor air sampling results when evaluating the following:

a. *current* human exposures; and

b. the potential for *future* human exposures (e.g., if the structural integrity of the building changes or the use of the building changes).

2.2.4 Indoor air

Indoor air samples are collected to characterize exposures to air within a building, including those with earthen floors. Indoor air sampling results are used when evaluating the following:

a. *current* human exposures;

b. the potential for *future* exposures (e.g., if a currently vacant building should become occupied); and

c. site-specific attenuation factors (e.g., the ratio of indoor air to sub-slab vapor concentrations).

Indoor air samples are often collected after subsurface vapor characterization and other environmental sampling (e.g., soil and groundwater characterization) indicate they are warranted. When indoor air samples are collected, concurrent sub-slab vapor, crawl space air (if applicable) and outdoor air samples are collected to evaluate the indoor air results appropriately. However, indoor air and outdoor air samples, without sub-slab vapor samples, may be collected when confirming the effectiveness of a mitigation system [Section 4].

In addition, site-specific situations may warrant collecting indoor air samples prior to characterizing subsurface vapors and/or without concurrent sub-slab vapor sampling to examine immediate inhalation hazards. Examples of such situations may include, but are not limited to, the following:

a. in response to a spill event to qualitatively and/or quantitatively characterize the contamination;

b. if high readings are obtained in a building when screening with field equipment (e.g., a photoionization detector (PID), an organic vapor analyzer, or an explosimeter) and the source is unknown;

c. if significant odors are present and the source needs to be characterized; or

d. if groundwater beneath the building is contaminated, the building is prone to groundwater intrusion or flooding (e.g., sump pit overflows), and subsurface vapor sampling is not feasible. In these situations, the collection of water samples from the sump may also be appropriate.

2.2.5 Outdoor air

Outdoor air samples are collected to characterize site-specific background outdoor air conditions. Outdoor air samples should be collected simultaneously with indoor air samples to evaluate the potential influence, if any, of outdoor air on the indoor air sampled. Outdoor air samples may also be collected concurrently with soil vapor samples to identify potential outdoor air interferences associated with infiltration of outdoor air into the sampling apparatus while the soil vapor was collected.

2.3 Phase of a site investigation in which to sample

There is no single phase (e.g., preliminary site characterization or remedial investigation) of a site investigation during which sampling to evaluate the soil vapor intrusion pathway is appropriate. Initiation of investigation activities for this specific purpose should be determined on a site-by-site basis. However, if exposures due to soil vapor intrusion appear likely at any point during the investigation, evaluation of this exposure pathway should not be delayed.

If the locations of likely source areas are reasonably known, sampling earlier during the investigation of a site rather than later is recommended because of the iterative nature of the sampling process [Section 2.5]. However, if current site conditions are not well-defined, then sampling after contamination in other environmental media (e.g., groundwater and soil) has been characterized may be considered. In the latter scenario, groundwater, soil and other site information may be used to guide an investigation of the soil vapor intrusion pathway, such as selecting locations for subsurface vapor samples based on likely migration pathways and source areas [Sections 2.6.1 and 2.6.2]. At a minimum, depth to groundwater and soil stratigraphy should be identified prior to collecting soil vapor samples.

Sampling may be delayed at parcels that are undeveloped or contain unoccupied buildings provided

a. characterization of the parcel is not needed to
    1. address exposures in the surrounding area;
    2. design remedial measures for subsurface vapor contamination; or
    3. monitor or confirm the effectiveness of remedial measures; and
b. measures are in place that assure that the parcel will not be developed, or buildings occupied, without addressing exposure concerns [Section 3.6].

If exposures due to soil vapor intrusion appear likely, and a delay of sampling is contemplated, the State (i.e., the NYSDEC and NYSDOH) should be informed of the contemplated delay and the rationale for the delay. Furthermore, the party contemplating the delay should consider any comments the State may have on the information provided.

2.4 Time of year in which to sample

2.4.1 Soil vapor

Soil vapor samples are collected at any time during the year. Often, sampling is completed during the summer so the results can be used as a tool when selecting buildings to be sampled during the heating season.

2.4.2 Buildings

Sub-slab vapor samples and, unless immediate sampling is appropriate, indoor air samples are typically collected during the heating season because soil vapor intrusion is more likely to occur when a building's heating system is in operation and doors and windows are closed. In New York State, heating systems are generally expected to be operating routinely from November 15th to March 31st. However, these dates are not absolute; the timeframe for sampling may vary depending on factors such as the location of the site (e.g., upstate versus downstate) and the weather conditions for a particular year.

A soil vapor intrusion investigation at a building may be conducted outside of the heating season if the concern for vapor intrusion is greater during another time of year. This may occur at certain industrial buildings, for instance, where HVAC systems are actively managed to control the ratio of recirculated indoor air to make-up air from outside the building. Information about the site and potentially affected structures, including the factors discussed in Section 1.3, should be considered in determining the timing of an investigation.

Samples may be collected at any time of year if exposures due to soil vapor intrusion appear likely. However, samples collected at times when soil vapor intrusion is not expected to have its greatest effect on indoor air quality (typically, samples collected outside of the heating season) should not be used to rule out exposures. For example, results indicating "no further action" or "monitoring required" should be verified when soil vapor intrusion is believed to be most likely to ensure these actions are protective throughout the year.

2.5 Number of sampling rounds

Investigating the soil vapor intrusion pathway usually involves more than one round of subsurface vapor, indoor air and/or outdoor air sampling, for reasons such as the following:

a. to characterize the nature and extent of subsurface vapor contamination (similar to the delineation of groundwater contamination) and to address corresponding exposure concerns;

b. to evaluate fluctuations in concentrations due to

    1. different weather conditions (e.g., seasonal effects),

    2. changes in building conditions (e.g., various operating conditions of a building's HVAC system),

    3. changes in source strength, or

    4. vapor migration or contaminant biodegradation processes (particularly when degradation products may be more toxic than the parent compounds); or

c. to confirm sampling results or the effectiveness of mitigation or remedial systems.

Overall, as discussed in Section 1.5, successive rounds of sampling should be conducted until the following questions can be answered:

a. Are subsurface vapors contaminated? If so, what are the nature and extent of contamination? What is/are the source(s) of the contamination?

b. What are the current and potential exposures to contaminated subsurface vapors?

c. What actions, if any, are appropriate to prevent or mitigate exposures and to remediate subsurface vapor contamination?

Toward this end, multiple rounds of sampling may be appropriate to characterize the nature and extent of subsurface vapor contamination such that

a. both potential and current exposures are addressed [Section 2.6];

b. measures can be designed to remediate subsurface vapor contamination, either directly (e.g., SVE system) or indirectly (e.g., soil excavation or groundwater remediation), given that monitoring and mitigation are considered temporary measures implemented to address exposures related to vapor intrusion until contaminated environmental media are remediated [Section 3.4]; and

c. the effectiveness of remedial measures can be monitored and confirmed (e.g., endpoint sampling) [Section 4.5].

2.6 Sampling locations

The general approach for selecting sampling locations as part of a soil vapor intrusion investigation is similar to the approach for the investigation of other environmental media (e.g., soil and groundwater). Sampling locations should be selected with consideration of the conceptual site model [Section 1.6]. These locations should be selected to meet the stated objectives of the sampling program. Additionally, similar to the investigation of soil and groundwater, it is typical to start at a known or suspected source and work outward. The specific approach, however, will be dependent upon site-specific and building-specific conditions.

2.6.1 Soil vapor

If available, existing environmental data (e.g., groundwater and soil data) and site background information should be used to select locations for sampling soil vapor as part of a vapor intrusion investigation. Locations will vary depending upon surface features (e.g., presence or absence of buildings, areas of pavement, or vacant lot) and subsurface characteristics (e.g., soil stratigraphy, buried structures, utility corridors, or clay lenses), as well as the specific purpose of the sampling. Therefore, a figure illustrating proposed sampling locations (with respect to both areal position and depth), actual locations sampled in the field, and relevant on-site and off-site features should be included in all sampling work plans and reports.

Examples of how locations may vary given the specific purpose of the sampling follow. They include general guidelines that should be followed when selecting soil vapor sampling locations:

a. to evaluate the **potential for current on-site or off-site exposures**, samples should be collected
    1. in the vicinity of a building's foundation [see special sampling consideration at the end of Section 2.6.1 if sampling around a building with no surrounding surface confining layer], as well as between the building's foundation and the source (if known and not located beneath the building),
    2. along the site's perimeter, and
    3. at a depth comparable to the depth of foundation footings (determined on a building-specific or site-specific basis) or at least 1 foot above the water table in areas where the groundwater table is less than 6 feet below grade;
b. to evaluate the **potential for future exposures if development** on a known or suspected contaminated area on-site or off-site is possible, representative samples should be collected
    1. in areas with either known or suspected subsurface sources of volatile chemicals, in areas where elevated readings were obtained with field equipment during previous environmental investigations, and in areas of varying concentrations of contamination in the upper groundwater,
    2. in a grid pattern across the area (at an appropriate spacing interval for the size of the area) if information is limited for the area, and
    3. at multiple depths from the suspected subsurface source, or former source, to a depth comparable to the expected depth of foundation footings;

c. to evaluate the **potential for off-site soil vapor contamination**, samples should be collected

    1. along the site's perimeter,
    2. in areas of potential subsurface sources of vapor contamination (e.g., a groundwater plume that has migrated off-site), and
    3. at a depth comparable to the depth of foundation footings (determined on a site-specific basis) or at least 1 foot above the water table in areas where the groundwater table is less than 6 feet below grade;

d. to evaluate on-site and off-site **preferential migration pathways** in areas with low permeability soils, samples should be collected

    1. along preferential soil vapor flow paths, such as sewer lines, utility corridors, trenches, pipelines, and other subsurface structures that are likely to be bedded with higher permeability materials, and
    2. at depths corresponding to these subsurface features (will depend on site-specific conditions);

e. to characterize on-site or off-site **contamination in the vadose zone**, samples should be collected

    1. in areas with either known or suspected sources of volatile chemicals, in areas where elevated readings were obtained with field equipment (e.g., PID) during previous soil and groundwater investigations, and in areas of varying concentrations of contamination in the upper groundwater regime, and
    2. at appropriate depths associated with these areas (will depend on site-specific conditions); and

f. to investigate the **influence of contaminated groundwater or soil on soil vapor** and to characterize the **vertical profile** of contamination, samples should be collected from clusters of soil vapor probes at varying depths in the vadose zone [Figure 2.2, Section 2.7.1] and preferably in conjunction with the collection of groundwater or soil samples.

Soil vapor samples collected at depths shallower than 5 feet below grade may be prone to negative bias due to infiltration of outdoor air. Therefore, samples from these depths should be collected only if appropriate (based on site-specific conditions), and sampling procedures and results should be reviewed accordingly. The depth of sampling near buildings with slab-on-grade foundations is dependent upon site-specific conditions (e.g., building surrounded by grassy or surface confining layer).

When collecting soil vapor samples around a building with no surrounding surface confining layer (e.g., pavement or sidewalk), samples should be located in native or undisturbed soils away from fill material surrounding the building (approximately 10 feet away from the building) to avoid sampling in an area that may be influenced by the building's operations. For example, operation of HVAC systems, fireplaces, or mechanical equipment (e.g., clothes dryers or exhaust fans/vents) in a building may exacerbate the infiltration of outdoor air into the vadose zone adjacent to the building. As a result, soil vapor samples collected in uncovered areas adjacent to the building may not be representative.

Investigations of soil vapor contamination should proceed outward from known or suspected subsurface sources, as appropriate, on an areal basis until the nature and extent of

subsurface vapor contamination has been characterized and human exposures have been addressed.

2.6.2 Sub-slab vapor

Existing environmental data (e.g., soil vapor, groundwater and soil data), site background information, and building construction details (e.g., basement, slab-on-grade, or multiple types of foundations, HVAC systems, etc.) should be considered when selecting buildings and locations within buildings for sub-slab vapor sampling.

At a minimum, these general guidelines should be followed when selecting buildings to sample for sub-slab vapors:

a. buildings, including residential dwellings, located above or directly adjacent to known or suspected areas of subsurface volatile chemical contamination should be sampled;

b. buildings in which screening with field equipment (e.g., PID, ppbRAE, Jerome Mercury Vapor Analyzer, etc.) suggests a completed migration pathway, such as when readings are above background and from unidentified sources or when readings show increasing gradients, should be sampled; and

c. buildings within known or suspected areas of subsurface volatile chemical contamination that are used or occupied by sensitive population groups (e.g., daycare facilities, schools, nursing homes, etc.) should be given special consideration for sampling.

Investigations of sub-slab vapor and/or indoor air contamination should proceed outward from known or suspected sources, as appropriate, on an areal basis until the nature and extent of subsurface vapor contamination has been characterized and potential and current human exposures have been addressed. In cases of widespread vapor contamination and depending upon the basis for making decisions (e.g., a "blanket mitigation" approach within a specified area of documented vapor contamination [Section 3.3.1]), a representative number of buildings from an identified study area, rather than each building, may be sampled. Prior to implementation, this type of sampling approach should be approved by State agency personnel.

Within a building, sub-slab vapor samples should be collected

a. in at least one central location away from foundation footings, and

b. from the soil or aggregate immediately below the basement slab or slab-on-grade.

The number of sub-slab vapor samples that should be collected in a building depends upon the number of slabs (e.g., multiple slabs-on-grade in a large warehouse) and foundation types (e.g., combined basement and slab-on-grade in a residence). At least one sub-slab vapor sample should be collected from each representative area.

2.6.3 Indoor air

Existing environmental data (e.g., soil vapor, groundwater and soil data), site background information, and building construction details (e.g., basement, slab-on-grade, or multiple types of foundations; number and operation of HVAC systems; elevator shafts; tunnels or other confined-space entry points; etc.) should be considered when selecting buildings and

locations within buildings for indoor air sampling. Indoor air samples are typically collected concurrently with sub-slab vapor and outdoor air samples [Section 2.2.4].

At a minimum, these general guidelines should be followed when selecting buildings to sample for indoor air:

a. where sub-slab vapor samples were collected without indoor air samples, buildings in which elevated concentrations of contaminants were measured in sub-slab vapor samples should be sampled;

b. buildings, including residential dwellings, located above or directly adjacent to known or suspected subsurface sources of volatile chemicals or known soil vapor contamination should be sampled;

c. buildings in which screening with field equipment (e.g., PID, ppbRAE, Jerome Mercury Vapor Analyzer, etc.) suggests a completed migration pathway, such as when readings are above background and from unidentified sources or when readings show increasing gradients, should be sampled; and

d. buildings within known or suspected areas of subsurface volatile chemical contamination that are used or occupied by sensitive population groups (e.g., daycare facilities, schools, nursing homes, etc.) should be given special consideration for sampling.

To characterize contaminant concentration trends and potential exposures, indoor air samples should be collected

a. from the crawl space area,

b. from the basement (where vapor infiltration is suspected, such as near sump pumps or indoor wells, or in a central location) at a height approximately three feet above the floor to represent a height at which occupants normally are seated and/or sleep,

c. from the lowest level living space (in centrally-located, high activity use areas) at a height approximately three feet above the floor to represent a height at which occupants normally are seated and/or sleep, and

d. if in a commercial setting (e.g., a strip mall), from multiple tenant spaces at a height approximately three feet above the floor to represent a height at which occupants normally are seated.

These locations are illustrated in Figure 2.1.

Investigations of indoor air contamination should proceed outward from known or suspected subsurface sources, as appropriate, on an areal basis until potential and current human exposures associated with soil vapor intrusion have been addressed. In cases of widespread vapor contamination and depending upon the basis for making decisions (e.g., a "blanket mitigation" approach within a specified area of documented vapor contamination), a representative number of buildings from an identified study area, rather than each building, may be sampled. Prior to implementation, this type of sampling approach should be approved by State agency personnel.

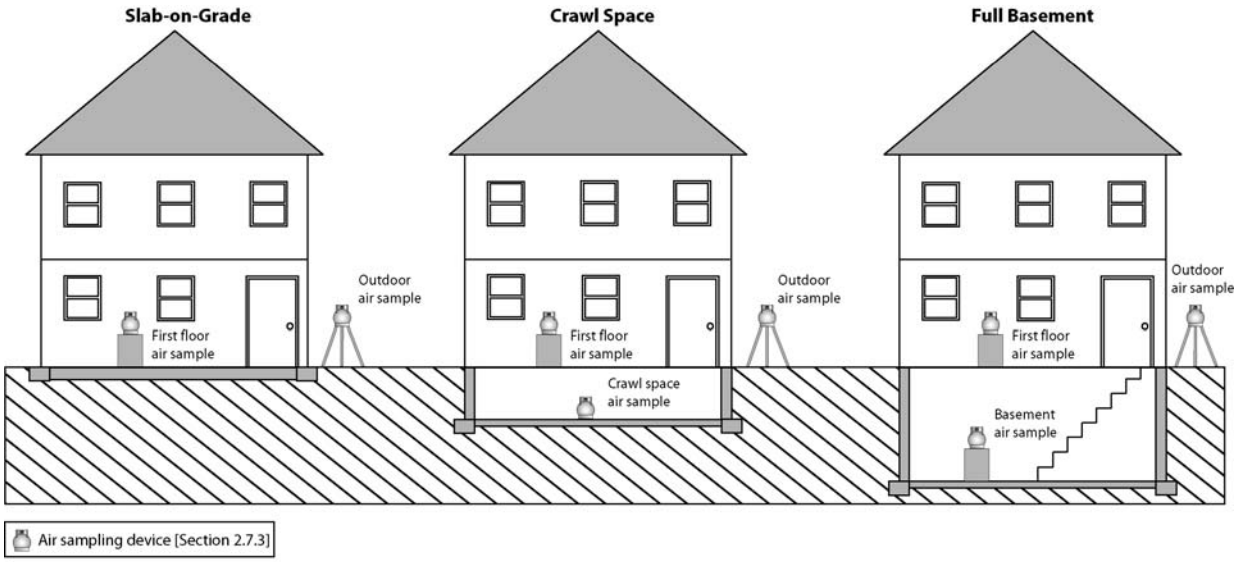


Figure 2.1
Schematic of indoor and outdoor air sampling locations

2.6.4 Outdoor air

Typically, an outdoor air sample is collected outside of each building where an indoor air sample is collected. However, if several buildings are being sampled within a localized area, representative outdoor air samples may be appropriate. For example, one outdoor air sample may be sufficient for three houses being sampled in a cul-de-sac. Outdoor air samples should be collected from a representative upwind location, away from wind obstructions (e.g., trees or bushes), and at a height above the ground to represent breathing zones (3 to 5 feet) [Figure 2.1]. A representative sample is one that is not biased toward obvious sources of volatile chemicals (e.g., automobiles, lawn mowers, oil storage tanks, gasoline stations, industrial facilities, etc.). For buildings with HVAC systems that draw outdoor air into the building, an outdoor air sample collected near the outdoor air intake may be appropriate.

2.7 Sampling protocols

The procedures recommended here may be modified depending on site-specific conditions, the sampling objectives, or emerging technologies and methodologies. Alternative sampling procedures should be described thoroughly and proposed in a work plan submitted for review by the State. The State will review and comment on the proposed procedure and consider the efficacy of the alternative sampling procedure based on the objectives of investigation. In all cases, work plans should thoroughly describe the proposed sampling procedure. Similarly, the procedures that were implemented in the field should be documented and included in the final report of the sampling results.

2.7.1 Soil vapor

Soil vapor probe installations [Figure 2.2] may be permanent, semi-permanent or temporary. In general, permanent or semi-permanent installations are preferred for data consistency reasons and to ensure outdoor air infiltration does not occur. Temporary probes should only be used if measures are taken to ensure that an adequate surface seal is created to prevent outdoor air infiltration and if tracer gas is used at every sampling location. [See Section 2.7.5 for additional information about the use of tracer gas when collecting soil vapor samples.] Soil vapor implants or probes should be constructed in the same manner at all sampling locations to minimize possible discrepancies. The following procedures should be included in any permanent construction protocol:

a. implants should be installed using an appropriate method based on site conditions (e.g., direct push, manually driven, auger — if necessary to attain the desired depth or if sidewall smearing is a concern, etc.);

b. porous, inert backfill material (e.g., glass beads, washed #1 crushed stone, etc.) should be used to create a sampling zone 1 to 2 feet in length;

c. implants should be fitted with inert tubing (e.g., polyethylene, stainless steel, nylon, Teflon®, etc.) of the appropriate size (typically 1/8 inch to 1/4 inch diameter) and of laboratory or food grade quality to the surface;

d. soil vapor probes should be sealed above the sampling zone with a bentonite slurry for a minimum distance of 3 feet to prevent outdoor air infiltration and the remainder of the borehole backfilled with clean material;

e. for multiple probe depths, the borehole should be grouted with bentonite between probes to create discrete sampling zones or separate nested probes should be installed [Figure 2.2]; and

f. steps should be taken to minimize infiltration of water or outdoor air and to prevent accidental damage (e.g., setting a protective casing around the top of the probe tubing and grouting in place to the top of bentonite, sloping the ground surface to direct water away from the borehole like a groundwater monitoring well, etc.).

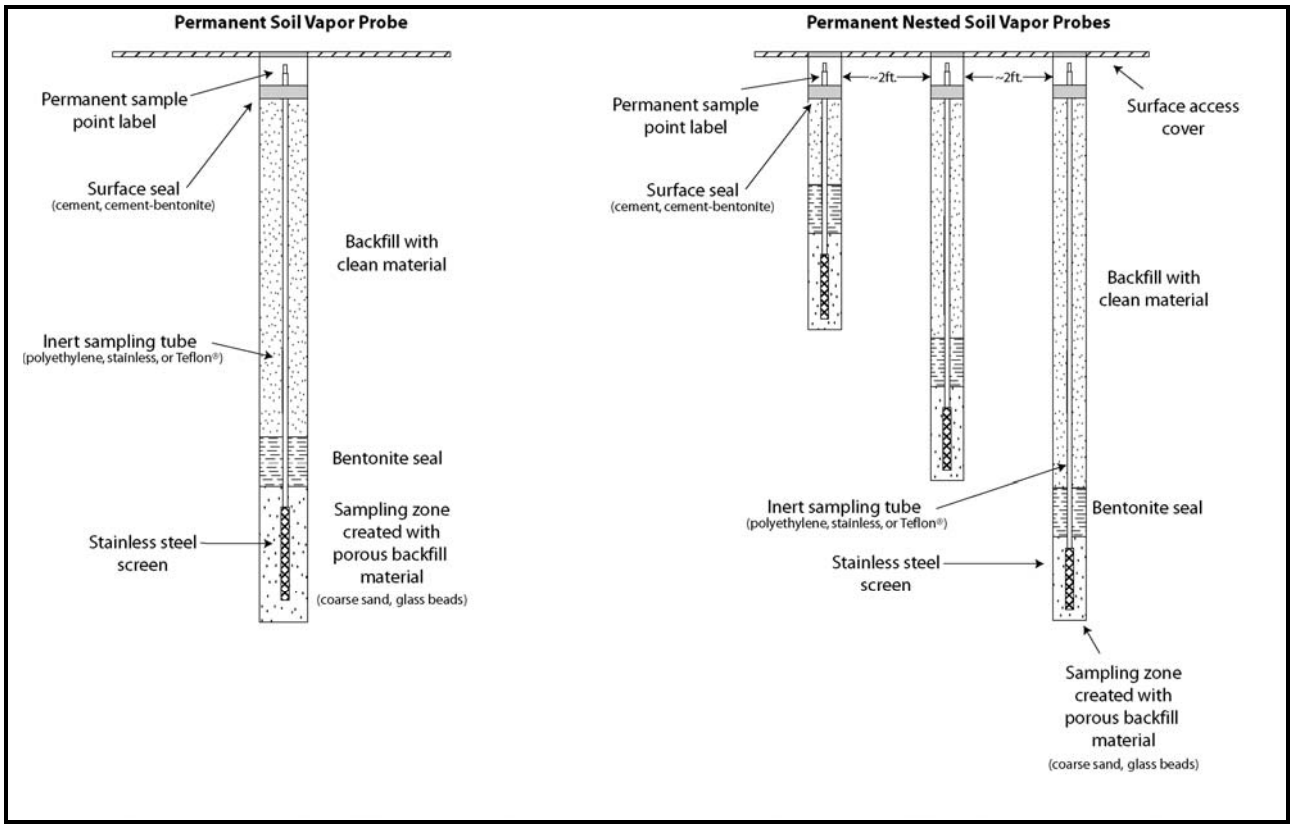


Figure 2.2

Schematics of a generic permanent soil vapor probe
and permanent nested soil vapor probes

[Note: Many variations exist and may be proposed in a work plan. Proposed installations should meet the sampling objectives and requirements of the analytical methods.]

To obtain representative samples and to minimize possible discrepancies, soil vapor samples should be collected in the following manner at all locations:

a. at least 24 hours after the installation of permanent probes and shortly after the installation of temporary probes, one to three implant volumes (i.e., the volume of the sample probe and tube) should be purged prior to collecting the samples;

b. flow rates for both purging and collecting should not exceed 0.2 liters per minute to minimize outdoor air infiltration during sampling;

c. samples should be collected, using conventional sampling methods, in an appropriate container — one which

   i. meets the objectives of the sampling (e.g., investigation of areas where low or high concentrations of volatile chemicals are expected; to minimize losses of volatile chemicals that are susceptible to photodegradation),

   ii. is consistent with the sampling and analytical methods (e.g., low flow rate; Summa® canisters if analyzing by using EPA Method TO-15), and

   iii. is certified clean by the laboratory;

d. sample size depends upon the volume of that will achieve minimum reporting limits [Section 2.9]; and

e. a tracer gas (e.g., helium, butane, sulfur hexafluoride, etc.) should be used when collecting soil vapor samples to verify that adequate sampling techniques are being implemented (i.e., to verify infiltration of outdoor air is not occurring) [Section 2.7.5].

In some cases, weather conditions may present certain limitations on soil vapor sampling. For example, condensation in the sample tubing may be encountered during winter sampling due to low outdoor air temperatures. Devices, such as tube warmers, may be used to address these conditions. Anticipated limitations to the sampling should be discussed prior to the sampling event so appropriate measures can be taken to address these difficulties and produce representative and reliable data.

When soil vapor samples are collected, the following actions should be taken to document local conditions during sampling that may influence interpretation of the results:

a. if sampling near a commercial or industrial building, uses of volatile chemicals during normal operations of the facility should be identified;

b. outdoor plot sketches should be drawn that include the site, area streets, neighboring commercial or industrial facilities (with estimated distance to the site), outdoor air sampling locations (if applicable), and compass orientation (north);

c. weather conditions (e.g., precipitation and outdoor temperature) should be noted for the past 24 to 48 hours; and

d. any pertinent observations should be recorded, such as odors and readings from field instrumentation.

Additional information that could be gathered to assist in the interpretation of the results includes barometric pressure, wind speed and wind direction.

The field sampling team should maintain a sample log sheet summarizing the following:

a. sample identification,

b. date and time of sample collection,

c. sampling depth,

d. identity of samplers,

e. sampling methods and devices,

f. purge volumes,

g. volume of soil vapor extracted,

h. if canisters used, the vacuum before and after samples were collected,

i. apparent moisture content (dry, moist, saturated, etc.) of the sampling zone, and

j. chain of custody protocols and records used to track samples from sampling point to analysis.

2.7.2 Sub-slab vapor

During colder months, heating systems should be operating to maintain normal indoor air temperatures (i.e., 65 – 75 °F) for at least 24 hours prior to and during the scheduled sampling time. Prior to installation of the sub-slab vapor probe, the building floor should be inspected and any penetrations (cracks, floor drains, utility perforations, sumps, etc.) should be noted and recorded. Probes should be installed at locations where the potential for ambient air infiltration via floor penetrations is minimal.

Sub-slab vapor probe installations [Figure 2.3] may be permanent, semi-permanent or temporary. A vacuum should not be used to remove drilling debris from the sampling port. Sub-slab implants or probes should be constructed in the same manner at all sampling locations to minimize possible discrepancies. The following procedures should be included in any construction protocol:

a. permanent recessed probes should be constructed with brass or stainless steel tubing and fittings;

b. temporary probes should be constructed with inert tubing (e.g., polyethylene, stainless steel, nylon, Teflon®, etc.) of the appropriate size (typically 1/8 inch to 1/4 inch diameter), and of laboratory or food grade quality;

c. tubing should not extend further than 2 inches into the sub-slab material;

d. porous, inert backfill material (e.g., glass beads, washed #1 crushed stone, etc.) should be added to cover about 1 inch of the probe tip for permanent installations; and

e. the implant should be sealed to the surface with non-VOC-containing and non-shrinking products for temporary installations (e.g., permagum grout, melted beeswax, putty, etc.) or cement for permanent installations.

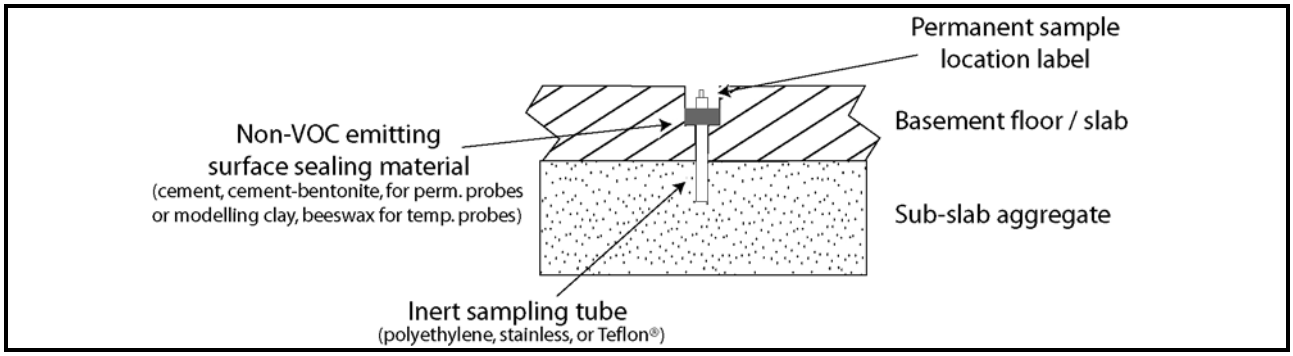


Figure 2.3

Schematic of a generic sub-slab vapor probe

[Note: Many variations exist and may be proposed in a work plan. Proposed installations should meet the sampling objectives and requirements of the analytical methods.]

To obtain representative samples that meet the data quality objectives, sub-slab vapor samples should be collected in the following manner:

a. after installation of the probes, one to three volumes (i.e., the volume of the sample probe and tube) must be purged prior to collecting the samples to ensure samples collected are representative;

b. flow rates for both purging and collecting must not exceed 0.2 liters per minute to minimize ambient air infiltration during sampling; and

c. samples should be collected, using conventional sampling methods, in an appropriate container — one which

    i. meets the objectives of the sampling (e.g., investigation of areas where low or high concentrations of volatile chemicals are expected; to minimize losses of volatile chemicals that are susceptible to photodegradation),

    ii. is consistent with the sampling and analytical methods (e.g., low flow rate; Summa® canisters if analyzing by using EPA Method TO-15), and

    iii. is certified clean by the laboratory;

d. sample size depends upon the volume of that will achieve minimum reporting limits [Section 2.9], the flow rate, and the sampling duration; and

e. ideally, samples should be collected over the same period of time as concurrent indoor and outdoor air samples.

When sub-slab vapor samples are collected, the following actions should be taken to document conditions during sampling and ultimately to aid in the interpretation of the sampling results [Section 3]:

a. historic and current storage and uses of volatile chemicals should be identified, especially if sampling within a commercial or industrial building (e.g., use of volatile chemicals in commercial or industrial processes and/or during building maintenance);

b. the use of heating or air conditioning systems during sampling should be noted;

c. floor plan sketches should be drawn that include the floor layout with sampling locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system air supply and return registers, compass orientation (north), footings that create separate foundation sections, and any other pertinent information should be completed;

d. outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sampling locations (if applicable), compass orientation (north), and paved areas;

e. weather conditions (e.g., precipitation and indoor and outdoor temperature) and ventilation conditions (e.g., heating system active and windows closed) should be reported; and

f. any pertinent observations, such as spills, floor stains, smoke tube results, odors and readings from field instrumentation (e.g., vapors via PID, ppbRAE, Jerome Mercury Vapor Analyzer, etc.), should be recorded.

Additional documentation that could be gathered to assist in the interpretation of the results includes information about air flow patterns and pressure relationships obtained by using smoke tubes or other devices (especially between floor levels and between suspected

contaminant sources and other areas), the barometric pressure and photographs to accompany floor plan sketches.

The field sampling team should maintain a sample log sheet summarizing the following:

a. sample identification,
b. date and time of sample collection,
c. sampling depth,
d. identity of samplers,
e. sampling methods and devices,
f. soil vapor purge volumes,
g. volume of soil vapor extracted,
h. if canisters used, vacuum of canisters before and after samples collected,
i. apparent moisture content (dry, moist, saturated, etc.) of the sampling zone, and
j. chain of custody protocols and records used to track samples from sampling point to analysis.

2.7.3 Indoor air

[Reference: NYSDOH's *Indoor Air Sampling & Analysis Guidance (February 1, 2005)*]

During colder months, heating systems should be operating to maintain normal indoor air temperatures (i.e., 65 – 75 °F) for at least 24 hours prior to and during the scheduled sampling time. If possible, prior to collecting indoor samples, a pre-sampling inspection [Section 2.11.1] should be performed to evaluate the physical layout and conditions of the building being investigated, to identify conditions that may affect or interfere with the proposed sampling, and to prepare the building for sampling. This process is described in Section 2.11.1.

In general, indoor air samples should be collected in the following manner:

a. sampling duration should reflect the exposure scenario being evaluated without compromising the detection limit or sample collection flow rate (e.g., an 8 hour sample from a workplace with a single shift versus a 24 hour sample from a workplace with multiple shifts). To ensure that air is representative of the locations sampled and to avoid undue influence from sampling personnel, samples should be collected for at least 1 hour. If the goal of the sampling is to represent average concentrations over longer periods, then longer duration sampling periods may be appropriate. Typically, 24 hour samples are collected from residential settings;
b. personnel should avoid lingering in the immediate area of the sampling device while samples are being collected;
c. sample flow rates must conform to the specifications in the sample collection method and, if possible, should be consistent with the flow rates for concurrent outdoor air and sub-slab samples; and
d. samples must be collected, using conventional sampling methods, in an appropriate container — one which

i. meets the objectives of the sampling (e.g., investigation of areas where low or high concentrations of volatile chemicals are expected; to minimize losses of volatile chemicals that are susceptible to photodegradation),

ii. is consistent with the sampling and analytical methods (e.g., low flow rate; Summa® canisters if analyzing by using EPA Method TO-15), and

iii. is certified clean by the laboratory.

At sites with tetrachloroethene contamination, passive air monitors that are specifically analyzed for tetrachloroethene (i.e., "perc badges") are commonly used to collect indoor and outdoor air samples. If site characterization activities indicate that degradation products of tetrachloroethene also represent a vapor intrusion concern, perc badges may be used to indicate the likelihood of vapor intrusion (i.e., by using tetrachloroethene as a surrogate) followed, as appropriate, by more comprehensive sampling and laboratory analyses to quantify both tetrachloroethene and its degradation products. Perc badge samples ideally should be collected over a twenty-four hour period, but for no less than eight hours.

The following actions should be taken to document conditions during indoor air sampling and ultimately to aid in the interpretation of the sampling results [Section 3]:

a. historic and current uses and storage of volatile chemicals should be identified, especially if sampling within a commercial or industrial building (e.g., use of volatile chemicals in commercial or industrial processes and/or during building maintenance);

b. a product inventory survey documenting sources of volatile chemicals present in the building during the indoor air sampling that could potentially influence the sample results should be completed [Section 2.11.2];

c. the use of heating or air conditioning systems during sampling should be noted;

d. floor plan sketches should be drawn that include the floor layout with sampling locations, chemical storage areas, garages, doorways, stairways, location of basement sumps or subsurface drains and utility perforations through building foundations, HVAC system supply and return registers, compass orientation (north), footings that create separate foundation sections, and any other pertinent information should be completed;

e. outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sampling locations (if applicable), compass orientation (north), and paved areas;

f. weather conditions (e.g., precipitation and indoor and outdoor temperature) and ventilation conditions (e.g., heating system active and windows closed) should be reported; and

g. any pertinent observations, such as spills, floor stains, smoke tube results, odors and readings from field instrumentation (e.g., vapors via PID, ppbRAE, Jerome Mercury Vapor Analyzer, etc.), should be recorded.

Additional documentation that could be gathered to assist in the interpretation of the results includes information about air flow patterns and pressure relationships obtained by using smoke tubes or other devices (especially between floor levels and between suspected contaminant sources and other areas), the barometric pressure and photographs to accompany floor plan sketches.

The field sampling team should maintain a sample log sheet summarizing the following:

a. sample identification,
b. date and time of sample collection,
c. sampling height,
d. identity of samplers,
e. sampling methods and devices,
f. depending upon the method, volume of air sampled,
g. if canisters are used, vacuum of canisters before and after samples collected, and
h. chain of custody protocols and records used to track samples from sampling point to analysis.

2.7.4 Outdoor air

Outdoor air samples should be collected simultaneously with indoor air samples to evaluate the potential influence, if any, of outdoor air on indoor air quality. They may also be collected simultaneously with soil vapor samples to identify potential outdoor air interferences associated with infiltration of outdoor air into the sampling apparatus while the soil vapor was collected. To obtain representative samples that meet the data quality objectives, outdoor air samples should be collected in a manner consistent with that for indoor air samples (described in Section 2.7.3).

The following actions should be taken to document conditions during outdoor air sampling and ultimately to aid in the interpretation of the sampling results [Section 3]:

a. outdoor plot sketches should be drawn that include the building site, area streets, outdoor air sampling locations, the location of potential interferences (e.g., gasoline stations, factories, lawn movers, etc.), compass orientation (north), and paved areas;
b. weather conditions (e.g., precipitation and outdoor temperature) should be reported; and
c. any pertinent observations, such as odors, readings from field instrumentation, and significant activities in the vicinity (e.g., operation of heavy equipment or dry cleaners) should be recorded.

2.7.5 Tracer gas

When collecting soil vapor samples as part of a vapor intrusion evaluation, a tracer gas serves as a quality assurance/quality control measure to verify the integrity of the soil vapor probe seal. Without the use of a tracer, there is no way to verify that a soil vapor sample has not been diluted by outdoor air.

Depending on the nature of the contaminants of concern, a number of different compounds can be used as a tracer. Typically, sulfur hexafluoride (SF_6) or helium are used as tracers because they are readily available, have low toxicity, and can be monitored with portable measurement devices. Butane and propane (or other gases) could also be used as a tracer in some situations. Compounds other than those mentioned here may be appropriate, provided they meet project-specific data quality objectives. Where applicable, steps should

be taken to ensure that the gas used by the laboratory to clean the air sampling container is different from the gas used as a tracer during sampling (e.g., helium).

The protocol for using a tracer gas is straightforward: simply enrich the atmosphere in the immediate vicinity of the area where the probe intersects the ground surface with the tracer gas, and measure a vapor sample from the probe for the presence of high concentrations (> 10%) of the tracer. A cardboard box, a plastic pail, or even a garbage bag can serve to keep the tracer gas in contact with the probe during the testing. If there are concerns about infiltration of ambient air through other parts of the sampling train (such as around the fittings, not just at the probe/ground interface), then consideration should be given to ensuring that the tracer gas is in contact with the entire sampling apparatus. In these cases, field personnel may prefer to use a liquid tracer — soaking paper towels with a liquid tracer and placing the towels around the probe/ground interface, around fittings, and/or in the corner of a shroud.

There are two basic approaches to testing for the tracer gas:

1. include the tracer gas in the list of target analytes reported by the laboratory; or
2. use a portable monitoring device to analyze a sample of soil vapor for the tracer prior to and after sampling for the compounds of concern. (Note that the tracer gas samples can be collected via syringe, Tedlar® bag etc. They need not be collected in Summa® canisters or minicans.)

The advantage of the second approach is that the real time tracer sampling results can be used to confirm the integrity of the probe seals prior to formal sample collection.

Figure 2.4 depicts common methods for using tracer gas. In examples a, b and c, the tracer gas is released in the enclosure prior to initially purging the sample point. Care should be taken to avoid excessive purging prior to sample collection. Care should also be taken to prevent pressure build-up in the enclosure during introduction of the tracer gas. Inspection of the installed sample probe, specifically noting the integrity of the surface seal and the porosity of the soil in which the probe is installed, will help to determine the tracer gas setup. Figure 2.4a may be most effective at preventing tracer gas infiltration, however, it may not be appropriate in some situations depending on site-specific conditions. Figures 2.4b and 2.4c may be sufficient for probes installed in tight soils with well-constructed surface seals. Figure 2d provides an example of using a liquid tracer. In all cases, the same tracer gas application should be used for all probes at any given site.

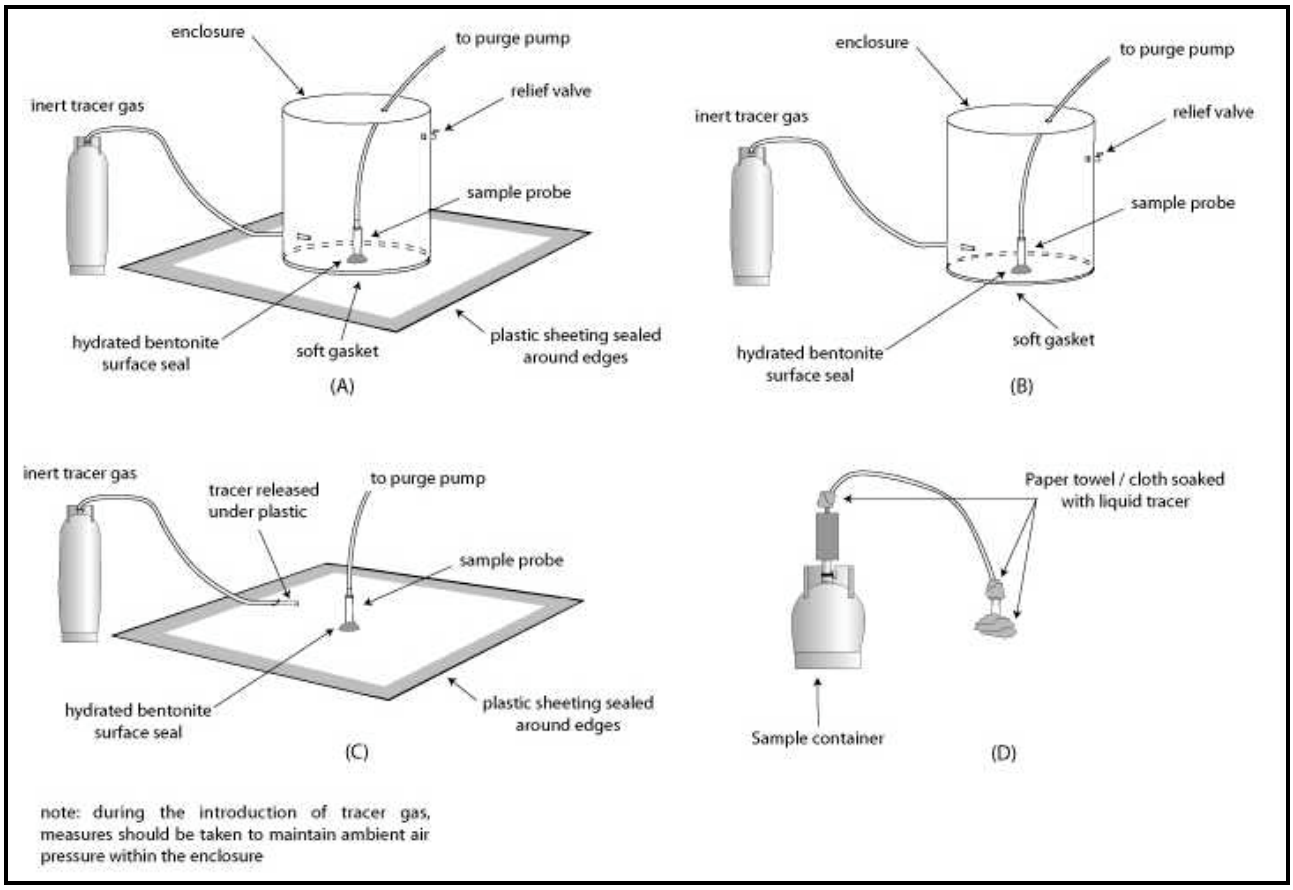


Figure 2.4
Schematics of generic tracer gas applications when collecting soil vapor samples

Because minor leakage around the probe seal should not materially affect the usability of the soil vapor sampling results, the mere presence of the tracer gas in the sample should not be a cause for alarm. Consequently, portable field monitoring devices with detection limits in the low ppm range are more than adequate for screening samples for the tracer. If high concentrations (> 10%) of tracer gas are observed in a sample, the probe seal should be enhanced to reduce the infiltration of outdoor air.

Where permanent or semi-permanent sampling probes are used, tracer gas samples should be collected at each of the sampling probes during the initial stages of a soil vapor sampling program. If the results of the initial samples indicate that the probe seals are adequate, reducing the number of locations at which tracer gas samples are employed may be considered. At a minimum, tracer gas samples should be collected with at least 10% of the soil vapor samples collected in subsequent sampling rounds. When using permanent soil vapor probes as part of a long-term monitoring program, annual testing of the probe integrity is recommended. Where temporary probes are used, tracer gas should be used at every sampling location, every time.

2.8 Quality assurance / quality control (QA / QC)

[Reference: NYSDOH's *Indoor Air Sampling & Analysis Guidance (February 1, 2005)*]

In general, appropriate QA/QC procedures should be followed during all aspects of sample collection and analysis to ensure that sampling error is minimized and high quality data are obtained. Sampling team members should avoid actions (e.g., fueling vehicles, using permanent marking pens, wearing freshly dry-cleaned clothing or personal fragrances, etc.) which can cause sample interference in the field. Portable air monitoring equipment or field instrumentation should be properly maintained, calibrated and tested to ensure validity of measurements. Air sampling equipment should be stored, transported and between samples decontaminated in a manner consistent with the best environmental consulting practices to minimize problems such as field contamination and cross-contamination. Samples should be collected using certified clean sample devices. Where applicable, steps should be taken to ensure that the gas used by the laboratory to clean the sample device is different from the gas used as a tracer during sampling (e.g., helium). Samples should meet sample holding times and temperatures, and should be delivered to the analytical laboratory as soon as possible after collection. In addition, laboratory accession procedures should be followed, including field documentation (sample collection information and locations), chain of custody, field blanks, field sample duplicates and laboratory duplicates, as appropriate.

Some methods call for collecting samples in duplicate (e.g., indoor air sampling using passive sampling devices for tetrachloroethene) to assess errors. Duplicate and/or split samples should be collected in accordance with the sampling and analytical methods being implemented.

For certain regulatory programs, a Data Usability Summary Report (DUSR) or equivalent report may be required to determine whether or not the data, as presented, meets the site or project specific criteria for data quality and data use. This requirement may dictate the level of QC and the category of data deliverable to request from the laboratory. Guidance on preparing these reports is available by contacting the NYSDEC's Division of Environmental Remediation.

New York State Public Health Law requires laboratories analyzing environmental samples collected from within New York State to have current Environmental Laboratory Approval Program (ELAP) certification for the appropriate analyte and environmental matrix combinations. If ELAP certification is not currently required for an analyte (e.g., trichloroethene), the analysis should be performed by a laboratory that has ELAP certification for similar compounds in air and uses analytical methods with minimum reporting limits similar to background (e.g., tetrachloroethene via EPA Method TO-15). Questions about a laboratory's current certification status should be directed to an ELAP representative at 518-485-5570 or by email at elap@health.state.ny.us.

The work plan should state that all samples that will be used to make decisions on appropriate actions to address exposures and environmental contamination will be analyzed by an ELAP-certified laboratory. The name of the laboratory should also be provided. Similarly, the name of the laboratory that was used should be included in the report of the sampling results. For samples collected and tested in the field for screening purposes by using field testing technology, the qualifications of the field technician should be documented in the work plan.

2.9 Analytical methods

[Reference: NYSDOH's *Indoor Air Sampling & Analysis Guidance (February 1, 2005)*]

Proposed analytical procedures should be identified in work plans. Similarly, the analytical procedures that were used and corresponding reporting limits should be identified when reporting the sampling results. When selecting an appropriate analytical method, the data quality objectives should be considered. As described in Section 3, comparing sampling results for volatile chemicals with background concentrations and with indoor air/sub-slab vapor matrices are critical components of the data evaluation process. Therefore, samples should be analyzed by methods that can achieve minimum reporting limits to allow for comparison of the results with background levels and with the levels presented in the matrices [Section 3.4.2]. If there are additional data quality objectives, they should be considered also. Typically, a minimum reporting limit of 1 microgram per cubic meter (1 mcg/m^3) or less is sufficient for most analytes. Examples of commonly used analytical methods include the following:

a. EPA Method TO-15 for a wide range of VOCs (e.g., samples from evacuated canisters),

b. NYSDOH Method 311-9 for tetrachloroethene (i.e., samples from perc badges),

c. EPA Method TO-17 for VOCs (e.g., samples collected with sorbent tubes), and

d. EPA Method TO-15 for VOCs with selective ion monitoring (SIM) (e.g., to achieve minimum reporting limits lower than those achieved with Method TO-15 alone).

The laboratory should verify that they are capable of detecting the appropriate analytes and can report them at the appropriate reporting limit.

2.9.1 Subsurface vapor

Soil vapor and sub-slab vapor samples should be analyzed for a wide range of volatile chemicals during the first round of sampling (at a minimum) — unless it can be demonstrated that an abbreviated or site-specific analyte list is appropriate. This is analogous to analyzing groundwater samples for a suite of compounds (e.g., EPA's target analyte list/target compound list (TAL/TCL) chemicals) during the initial rounds of site characterization. Based on the initial sampling results, development and application of a site-specific analyte list may be considered for analysis of subsequent soil vapor and sub-slab vapor samples.

If a site-specific analyte list is developed, it should include the following:

a. volatile chemicals which have been previously detected in environmental media (e.g., soil, groundwater and air) at the site;

b. volatile chemicals which are known or demonstrated constituents of the contamination in question (e.g., petroleum products or tars from former manufactured gas plants); and

c. expected degradation products of the chemicals mentioned in a or b.

A site-specific analyte list might also include indicator compounds to assist in identifying and differentiating subsurface sources of volatile chemical contamination. The following are examples of indicator compounds that have been included in site-specific analyte lists given the nature of the contamination or type of site:

a. gasoline: benzene, toluene, ethylbenzene, xylenes, trimethylbenzene isomers, individual C-4 to C-8 aliphatics (e.g., hexane, cyclohexane, dimethylpentane, 2,2,4-trimethylpentane, etc.), and appropriate oxygenate additives (e.g., methyl-*tert*-butyl ether, ethanol, etc.);

b. middle distillate fuels (#2 fuel oil, diesel and kerosene): n-nonane, n-decane, n-undecane, n-dodecane, ethylbenzene, xylenes, trimethylbenzene isomers, tetramethylbenzene isomers, naphthalene, 1-methylnaphthalene, and 2-methylnaphthalene;

c. manufactured gas plant sites: trimethylbenzene isomers, tetramethylbenzene isomers, thiopenes, indene, indane, and naphthalene;

d. natural gas: propane, propene, butane, iso-butane, methylbutane, and n-pentane with lower levels of higher molecular weight aliphatic, olefinic, and some aromatic compounds; and

e. solvent-using industries: the solvent and its expected degradation products (e.g., tetrachloroethene, trichloroethene, dichloroethene(s), and vinyl chloride).

2.9.2 Indoor air

Indoor and outdoor air samples should be analyzed for a wide range of volatile chemicals if there are no existing data for subsurface vapors — unless it can be demonstrated that an abbreviated or site-specific analyte list is appropriate. If indoor air sampling is appropriate based on the levels of volatile chemicals in subsurface vapors, analysis of indoor air samples specifically for those volatile chemicals may be considered.

2.9.3 Outdoor air

Outdoor air samples should be analyzed in a manner consistent with corresponding indoor air samples.

2.10 Field laboratories and mobile gas chromatographs (GCs)

Use of field laboratories and mobile GCs as screening tools when collecting soil vapor samples may be considered on a site-specific basis. However, without ELAP certification, screening tools such as these are not acceptable when collecting sub-slab vapor, indoor air and outdoor air samples for the purpose of evaluating exposures related to soil vapor intrusion. ELAP certification for a particular laboratory does not indicate mobile laboratory or GC certification. Mobile laboratories and GCs have specific certification requirements through ELAP. Questions regarding a mobile laboratory's certification should be directed to the laboratory itself.

2.11 Surveys and pre-sampling building preparation

[Reference: NYSDOH's *Indoor Air Sampling & Analysis Guidance (February 1, 2005)*]

2.11.1 Pre-sampling building inspection and preparation

A pre-sampling inspection should be performed prior to each sampling event to identify and minimize conditions that may interfere with the proposed testing. The inspection should evaluate the type of structure, floor layout, air flows and physical conditions of the building(s) being studied. This information, along with information on sources of potential

indoor air contamination [Section 2.11.2], should be identified on a building inventory form. An example of a building inventory form is given in Appendix B. Items to be included in the building inventory include the following:

a. construction characteristics, including foundation cracks and utility penetrations or other openings that may serve as preferential pathways for vapor intrusion;

b. presence of an attached garage;

c. recent renovations or maintenance to the building (e.g., fresh paint, new carpet or furniture);

d. mechanical equipment that can affect pressure gradients (e.g., heating systems, clothes dryers or exhaust fans);

e. use or storage of petroleum products (e.g., fuel containers, gasoline operated equipment and unvented kerosene heaters); and

f. recent use of petroleum-based finishes or products containing volatile chemicals.

Each room on the floor of the building being tested and on lower floors, if possible, should be inspected. This is important because even products stored in another area of a building can affect the air of the room being tested.

The presence and description of odors (e.g., solvent, moldy) and portable vapor monitoring equipment readings (e.g., PIDs, ppbRAE, Jerome Mercury Vapor Analyzer, etc.) should be noted and used to help evaluate potential sources. This includes taking readings near products stored or used in the building. Where applicable, readings should be provided in units that denote the calibration gas (e.g., isobutylene-equivalent ppm, benzene-equivalent ppm, etc.).

Potential interference from products or activities releasing volatile chemicals should be controlled to the extent practicable. Removing the source from the indoor environment prior to testing is the most effective means of reducing interference. Ensuring that containers are tightly sealed may be sufficient. When testing for volatile organic compounds, containers should be tested with portable vapor monitoring equipment to determine whether compounds are leaking. The inability to eliminate potential interference may be justification for not testing, especially when testing for similar compounds at low levels. The investigator should consider the possibility that chemicals may adsorb onto porous materials and may take time to dissipate.

In some cases, the goal of the testing is to evaluate the impact from products used or stored in the building (e.g., pesticide misapplications, school renovation projects). If the goal of the testing is to determine whether products are an indoor volatile chemical contaminant source, the removing these sources does not apply.

Once interfering conditions are corrected (if applicable), ventilation may be appropriate prior to sampling to minimize residual contamination in the indoor air. If ventilation is appropriate, it should be completed 24 hours or more prior to the scheduled sampling time. Where applicable, ventilation can be accomplished by operating the building's HVAC system to maximize outside air intake. Opening windows and doors, and operating exhaust fans may also help or may be appropriate if the building has no HVAC system.

Air samples are sometimes designed to represent typical exposure in a mechanically ventilated building and the operation of HVAC systems during sampling should be noted on

the building inventory form [Appendix B]. When samples are collected, the building's HVAC system should be operating in a manner consistent with normal operating conditions when the building is occupied (e.g., schools, businesses, etc.). Unnecessary building ventilation should be avoided within 24 hours prior to and during sampling. During colder months, heating systems should be operating to maintain normal indoor air temperatures (i.e., 65 – 75 °F) for at least 24 hours prior to and during the scheduled sampling time.

Depending upon the goal of the indoor air sampling, some situations may warrant deviation from the above protocol regarding building ventilation. In such cases, building conditions and sampling efforts should be understood and noted within the framework and scope of the investigation.

To avoid potential interferences and dilution effects, occupants should make a reasonable effort to avoid the following for 24 hours prior to sampling:

a. opening any windows, fireplace dampers, openings or vents;
b. operating ventilation fans unless special arrangements are made;
c. smoking in the building;
d. painting;
e. using a wood stove, fireplace or other auxiliary heating equipment (e.g., kerosene heater);
f. operating or storing automobile in an attached garage;
g. allowing containers of gasoline or oil to remain within the house or garage area, except for fuel oil tanks;
h. cleaning, waxing or polishing furniture, floors or other woodwork with petroleum- or oil-based products;
i. using air fresheners, scented candles or odor eliminators;
j. engaging in any hobbies that use materials containing volatile chemicals;
k. using cosmetics including hairspray, nail polish, nail polish removers, perfume/cologne, etc.;
l. lawn mowing, paving with asphalt, or snow blowing;
m. applying pesticides;
n. using building repair or maintenance products, such as caulk or roofing tar; and
o. bringing freshly dry-cleaned clothing or furnishings into the building.

2.11.2 Product inventory

The primary objective of the product inventory is to identify potential air sampling interference by characterizing the occurrence and use of chemicals and products throughout the building, keeping in mind the goal of the investigation and site-specific contaminants of concern. For example, it is not appropriate to provide detailed information for each individual container of like items. However, it is appropriate to indicate that "20 bottles of perfume" or "12 cans of latex paint" were present with containers in good condition. This information is used to help formulate an indoor environment profile.

An inventory should be provided for each room on the floor of the building being tested and on lower floors, if possible. This is important because even products stored in another area of a building can affect the air of the room being tested.

The presence and description of odors (e.g., solvent, moldy) and portable vapor monitoring equipment readings (e.g., PIDs, ppbRAE, Jerome Mercury Vapor Analyzer, etc.) should be noted and used to help evaluate potential sources. This includes taking readings near products stored or used in the building. Where applicable, readings should be provided in units that denote the calibration gas (e.g., isobutylene-equivalent ppm, benzene-equivalent ppm, etc.).

Products in buildings should be inventoried every time air is tested to provide an accurate assessment of the potential contribution of volatile chemicals. If available, chemical ingredients of interest (e.g., analyte list) should be recorded for each product. If the ingredients are not listed on the label, record the product's exact and full name, and the manufacturer's name, address and telephone number, if available. In some cases, material Safety Data Sheets may be useful for identifying confounding sources of volatile chemicals in air. Adequately documented photographs of the products and their labeled ingredients can supplement the inventory and facilitate recording the information.

2.12 Role of modeling

At sites where there is a potential for human exposures to subsurface contamination due to soil vapor intrusion (as described in Section 2.1), use of modeling as the sole means of evaluating potential exposures should be avoided. The limitations of modeling (e.g., exclusion of preferential migration pathways) introduce uncertainty as to whether human exposure is occurring, in absence of actual field data. Conclusions drawn from modeling should be verified with actual field data. For example, if modeling results indicate indoor air concentrations are predicted to be below applicable guidelines or levels of concern, indoor air and/or sub-slab vapor sampling would be appropriate to verify a conclusion that mitigation or other actions are not needed.

Modeling may, however, be used as a tool in the evaluation process. Examples of situations in which modeling may be used as a tool include, but are not limited to, the following:

a. to help identify potential migration pathways on the basis of site-specific conditions;

b. to estimate potential exposures when field samples cannot be collected (e.g., access to collect the samples is denied or buildings have not yet been constructed over the subsurface contamination); and

c. to identify a preferred order for sampling buildings by predicting expected indoor air concentrations within each of the buildings if there are numerous buildings overlying the subsurface contamination.

Use of any model at a site should be discussed with the agencies prior to the model's development and application. If a model is used, it should incorporate site-specific parameters (e.g., attenuation factors, soil conditions, concentrations of volatile chemicals, depth to subsurface source, characteristics of subsurface source, and foundation slab thickness) as much as possible. Furthermore, both the limitations of the model (e.g., exclusion of preferential migration pathways) and the sensitivity of the variables in the model should be understood and identified with the modeling results.

Section 3: Data Evaluation and Recommendations for Action

Section 3 describes the process by which data obtained during the investigation are evaluated. The goals of the evaluation are as follows:

a. to determine what volatile chemicals, if any, are present in the investigated media;

b. to identify the likely cause(s) of their presence; and

c. to identify completed and potential human exposures whether actions to address exposures should be taken.

Also discussed are actions typically recommended based on the evaluation. Actions to remediate the source(s) of soil vapor contamination, such as soil excavation or air-sparge/soil vapor extraction systems, are beyond the scope of this guidance and are not included.

3.1 Data quality

Before the data are evaluated, their representativeness and reliability should be verified. To assess analytical errors and the usability of the data, a qualified person should review the analytical data package and all associated QA/QC information to make sure that

a. the data package is complete;

b. holding times have been met;

c. the QC data fall within the protocol limits and specifications;

d. the data have been generated using established and agreed upon analytical protocols;

e. the raw data confirm the results provided in the data summary sheets and QC verification forms; and

f. correct data qualifiers have been used.

As discussed in Section 2.8, for sites in an environmental remediation program (e.g., State Superfund), a DUSR or equivalent report should be generated in accordance with NYSDEC guidance and should be submitted for regulatory review and approval.

If the investigation was not completed in accordance with the guidelines set forth in Section 2, additional investigation may be appropriate to either replace or complement the existing data. For example, product inventories [Section 2.11.2] filled out incompletely or incorrectly may need to be redone (and in some cases with additional air sampling) so that likely sources of volatile chemicals in the indoor air can be identified and appropriate actions to mitigate exposures can be recommended.

3.2 Overview

The results of individual soil vapor, sub-slab vapor, indoor air and outdoor air samples are not reviewed in isolation. Rather, they are evaluated with the consideration of several additional factors, which include the following:

a. the nature and extent of contamination in *all* environmental media;

b. factors that affect vapor migration and intrusion;

c. completed or proposed remedial actions;

d. sources of volatile chemicals;

e. background levels of volatile chemicals in air;

f. relevant standards, criteria and guidance values; and

g. past, current and future land uses.

These factors are described in detail in this subsection.

3.2.1 Nature and extent of contamination in all environmental media

The type of volatile chemicals present and the extent of contamination in all environmental media — including soil, groundwater, subsurface vapors, indoor air and outdoor air — is considered when evaluating the data. Trends in environmental data (e.g., groundwater monitoring results show concentrations of volatile chemicals are decreasing) are also considered. This information is used to identify possible sources of contamination and migration pathways, as well as to recommend appropriate actions to address exposures.

3.2.2 Factors that affect vapor migration and intrusion

As discussed in Section 1.3, there are numerous site-specific environmental factors [Table 1.1] and building factors [Table 1.2] that can affect soil vapor migration and intrusion. This information is used to identify possible sources of contamination and migration pathways, as well as to recommend appropriate actions to address exposures.

3.2.3 Sources of volatile chemicals

An understanding of the likely sources of the chemicals is crucial for determining appropriate actions to address exposure, as well as identifying the parties responsible for implementing the actions. Volatile chemicals that are not site-related may be present in the investigated media for reasons such as the following:

a. *subsurface vapors* — misuse, misapplication, or improper disposal of the chemicals to the subsurface, unidentified subsurface sources of vapor contamination, presence of septic systems (where products, such as cleaning agents or degreasers, may be disposed), biodegradation of natural organic matter in soil, infiltration into the subsurface from a building under positive pressure in which the chemicals are heavily used (i.e., reverse process from soil vapor intrusion), etc.;

b. *indoor air* — use and storage (current or historic) of volatile chemical-containing products, off-gassing from building materials or new furnishings, use of contaminated groundwater during private well usage, infiltration of outdoor air containing volatile chemicals, etc. [Table 1.3]; and

c. *outdoor air* — emissions from automobiles, lawn mowers, oil storage tanks, gasoline stations, dry cleaners or other commercial/industrial facilities, etc. [Table 1.3].

Site-related chemicals may also be present for these same reasons. Information about household products and their ingredients are available on web sites, such as the National Institute of Health's site at http://householdproducts.nlm.nih.gov.

3.2.4 Background levels of volatile chemicals in air

Chemicals are part of our everyday life [Section 1.4]. As such, they are found in the indoor air of buildings not affected by intrusion of contaminated soil vapor. They are also found in the outdoor air that enters a home or place of business. Commonly found concentrations of these chemicals in indoor and outdoor air are referred to as "background levels." Background levels of volatile chemicals are one of the factors considered when evaluating sampling results at a site [Section 3.3.2 – 3.3.4]. Estimates of background levels come from studies where air samples were collected in homes, offices and outdoor areas.

Several studies have been conducted, both nationally and in the State of New York, to provide information on indoor and outdoor air background levels in a variety of settings (e.g., residential or commercial buildings). Each of these studies offers useful information and has its own limitations. Each database provides statistical measures of background levels and the criteria used to select sampling locations. The criteria in some of the studies required that sampling locations not be located near known sources of volatile chemicals (for example, not near a chemical spill, hazardous waste site, dry-cleaner, or factory). The criteria may also have included checking containers of volatile chemicals in or near the building to make sure they are tightly closed or removing those products before samples are taken. Depending on the criteria for site selection and sampling conditions, statistical measures of background levels in a given study may differ from what would be expected if indoor air were sampled in randomly selected homes.

The background databases that are used for evaluating indoor and outdoor air data are introduced below. A more detailed description of each database along with statistical measures of background levels are provided in Appendix C.

a. *NYSDOH 2003: Study of Volatile Organic Chemicals in Air of Fuel Oil Heated Homes*

   Results of indoor and outdoor air samples collected from 104 single-family fuel oil heated homes throughout New York State. Samples collected in evacuated canisters and analyzed for 69 aromatic, aliphatic, and halogenated hydrocarbons, and ketones by modified EPA Method TO-15. Limitations: only fuel oil heated homes were included, homes were not randomly selected, and five boroughs of New York City were excluded.

b. *EPA 2001: Building Assessment and Survey Evaluation (BASE) Database*

   Study of measured concentrations of volatile organic compounds from 100 randomly selected public and commercial office buildings. Samples collected by evacuated canisters and/or tube methodologies. Limitations: only represents office settings, two methodologies used for sampling and analysis that are not completely overlapping and do not show agreement in results in some cases.

c. *NYSDOH 1997: Control Home Database*

   Indoor and outdoor air samples compiled from 53 residences in New York State that were considered "control Homes" with neighborhood, construction, and occupancy similar to potentially impacted homes that were being investigated at the time. Limitations: multiple methodologies for sampling and analysis, small sample size, and varying detection limits often higher than current background levels.

d. *EPA 1988: National Ambient Volatile Organic Compounds (VOCs) Data Base Update*

   Published and unpublished air data compiled by the EPA in 1988. The document includes data from studies between 1970 to 1987. The database covers more than

300 chemicals in indoor and outdoor settings. Limitations: data are compiled from numerous studies with limitations on selection or screening criteria, data are 20-35 years old, indoor air data include both residential and office spaces, sample size for some analytes is very small (less than 10). Outdoor air data include rural, suburban, urban, source dominated and remote locations.

e. *Health Effects Institute (HEI) 2005: Relationship of Indoor, Outdoor, and Personal Air (RIOPA)*

Indoor, outdoor and personal air concentrations of 18 VOCs, 10 carbonyl compounds and particulate matter (PM2.5) were measured in 100 homes in each of 3 cities between the summer of 1999 and the spring of 2001. Limitations: limited numbers of VOCs, passive organic vapor badge method is subject to sampling bias in stationary versus mobile locations, the passive organic vapor badge method is only approved for tetrachloroethene in New York State.

Among the databases, the Upper Fence (see *NOTE below) values from the NYSDOH Fuel Oil Study data may be used as initial benchmarks when evaluating residential indoor air (see Appendix C.1) and the 90th percentile values from the EPA BASE data for indoor air in office and commercial buildings (see Appendix C.2). These initial benchmark values should be considered along with the overall distribution of results in the background database to characterize sampling results from a single building or from multiple buildings in a community. The Health Effects Institute 2005 database and the older NYSDOH and EPA databases can also provide useful information on the range of concentrations found in air. The database or combination of databases that best represents site-specific conditions should be used as the basis for comparison. State agency personnel should review and have the opportunity to comment on the proposed use of other databases or subsets of data within a database for evaluating test results.

*NOTE: The Upper Fence is calculated as 1.5 times the interquartile range (difference between the 25th and 75th percentile values) above the 75th percentile value. It is a boundary estimate used to account for outliers in the data.

3.2.5 Relevant standards, criteria and guidance values

a. *Subsurface vapors*

The State of New York does not have any standards, criteria or guidance values for concentrations of volatile chemicals in subsurface vapors (either soil vapor or sub-slab vapor).

b. *Indoor and outdoor air*

The NYSDOH has developed several guidelines for chemicals in air. The development process is initiated for specific situations. For example, in New York State, particularly in New York City, dry cleaners are often located in apartment buildings. Because air in buildings mixes to some extent and the dry cleaning chemical tetrachloroethene (PCE) is volatile, it may migrate to residential apartments. When the NYSDOH became aware of this problem and how widespread it is, the NYSDOH developed an air guideline for PCE of 100 micrograms per cubic meter (mcg/m^3). In addition to PCE, the NYSDOH has developed guidelines for methylene chloride (also referred to as dichloromethane) and trichloroethene (TCE) in air, as well as dioxin and polychlorinated biphenyls (PCBs) in indoor air. Each guideline went through a peer review process, in which expert scientists outside of the NYSDOH reviewed the technical documentation that describes

the scientific basis for the guidance value. The peer reviewers provided technical comments on the data and methods used to derive the guidelines, each of which were addressed by the NYSDOH. Upon completion of the reviews and responses to comments, the guidelines were finalized.

Air guideline values derived by the NYSDOH are summarized in Table 3.1. Additional information about these guidelines is provided in the following:

- Appendix D — overview of how the NYSDOH develops air guidelines; and
- Appendix H — copies of fact sheets that discuss the air guidelines for PCE and TCE.

The purpose of a guideline is to help guide decisions about the nature of efforts to reduce exposure to the chemical. Reasonable and practical actions should be taken to reduce exposures when indoor air levels are above background, even when they are below the guideline. The urgency to complete these actions increases with indoor air levels, particularly when air levels are above the guideline, and additional actions taken if the initial actions do not sufficiently reduce levels. In all cases, the specific corrective actions to be taken depend on a case-by-case evaluation of the situation. The goal of the recommended actions is to reduce chemical levels in indoor air to as close to background as practical.

Table 3.1 Air guideline values derived by the NYSDOH

Chemical		Air Guideline Value (mcg/m^3)	Reference
methylene chloride (also referred to as dichloromethane)	MeCl	60	1
polychlorinated biphenyls	PCBs	1*	2,3
tetrachlorodibenzo-*p*-dioxin equivalents	TCDD	0.00001*	3,4
tetrachloroethene	PCE	100	5
trichloroethene	TCE	5	6,7

*The guideline is specific to indoor air.

References:

[1] NYSDOH. 1988. Letter from N. Kim to T. Allen, Division of Air, New York State Department of Environmental Conservation. November 28, 1988.
[2] NYSDOH. 1985. Binghamton State Office Building (BSOB) Re-Entry Guidelines: PCBs. Document 1330P. Albany, NY: Bureau of Toxic Substance Assessment.
[3] NYSDOH. 1988. Letter from D. Axelrod to J. Egan, New York State Office of General Services. March 8, 1988.
[4] NYSDOH. 1984. Re-Entry Guidelines. Binghamton State Office Building. Document 0549P. Albany, NY: Bureau of Toxic Substance Assessment.
[5] NYSDOH. 1997. Tetrachloroethene Ambient Air Criteria Document. Albany, NY: Bureau of Toxic Substance Assessment.
[6] NYSDOH. 2003. Letter from N. Kim to D. Desnoyers, Division of Environmental Remediation, New York State Department of Environmental Conservation. October 31, 2003. [Provided in Appendix D.]
[7] NYSDOH. 2006. Final Report: Trichloroethene (TCE) Air Criteria Document. Center for Environmental Health, Bureau of Toxic Substance Assessment. Troy, NY.

3.2.6 Completed or proposed remedial actions

The status and effectiveness of actions taken to remediate environmental contamination (e.g., soil removal, groundwater treatment, soil vapor extraction, etc.) are considered when making decisions pertaining to additional sampling and the selection of mitigation actions. For example,

a. if a comparison of pre-remediation and post-remediation subsurface vapor sampling results indicates negligible improvement in the quality of subsurface vapors,

    1. additional sampling may be appropriate to document a decreasing trend in subsurface vapor concentrations;

    2. termination of mitigation system operations may not be appropriate without additional sampling; or

    3. additional remedial actions may be appropriate to address contaminated subsurface vapors;

b. when monitoring a building is appropriate, it may be more cost-effective to install a mitigation system if subsurface contamination is wide-spread and is expected to take many years to remediate; and

c. if exposures in an on-site building will be addressed concurrently by a method selected to remediate subsurface contamination (e.g., a soil vapor extraction system), installation of a mitigation system may be redundant. However, if the remedial system is not expected to be operational in the immediate future, or if it is not expected to mitigate indoor air levels in a reasonable time frame, a mitigation system may still be appropriate. [Refer to Section 4.1 for a description of the appropriate use of concurrent techniques.]

3.2.7 Past, current and future land uses

Past, current and future land uses are considered when evaluating the investigation data and determining appropriate actions for further investigation or measures to address exposures. For example,

a. if the parcel or buildings were historically used for commercial or industrial purposes (e.g., gasoline station, automotive repair facility, electroplating facility, etc.), but are currently used for residential purposes or commercial or industrial purposes where volatile chemicals are not used in current operations, off-gassing of volatile chemicals from building materials [Table 1.3] or additional subsurface sources should be considered;

b. subsurface vapor sampling of a parcel that is undeveloped or contains unoccupied buildings may be appropriate based on the data evaluation. However, sampling may be delayed as discussed in Section 2.3;

c. air sampling of a building may be appropriate based on the data evaluation. However, provisions may be put in place to defer sampling until occupancy of the building is expected; or

d. if actions should be taken to mitigate exposures related to soil vapor intrusion should the site be developed, the appropriate mitigation method will depend upon the proposed land use — a parking lot, recreational field, single-family home, commercial building, high-rise building with underground parking, occupied or unoccupied building, etc. — since each presents a different exposure scenario.

3.3 Sampling results and recommended actions

This subsection describes the process for evaluating sampling results. It also describes actions that may be recommended based on the evaluation. The evaluation procedures and actions described may not be directly applicable to samples collected as part of an emergency response. For guidance on how to proceed in such situations, refer to Section 3.5.

3.3.1 Soil vapor

If soil vapor samples are collected from locations where there are no known sources of volatile chemicals, we do not expect the chemicals to reach detectable levels in the samples. However, concentrations of volatile chemicals in soil vapor are commonly detected. This is likely due to several factors, including infiltration of outdoor air into the subsurface (to a limited extent) and background interferences (similar to indoor and outdoor air [Section 3.2.4]).

New York State currently does not have any standards, criteria or guidance values for concentrations of compounds in soil vapor. Additionally, there are currently no databases available of background levels of volatile chemicals in soil vapor. In the absence of this information, soil vapor sampling results are reviewed "as a whole," in conjunction with the results of other environmental sampling and the site conceptual model, to identify trends and spatial variations in the data [Section 3.2.1]. To put some perspective on the data, soil vapor results might be compared to background outdoor air levels [Section 3.2.4], site-related outdoor air sampling results, or the NYSDOH's guidelines for volatile chemicals in air [Table 3.1].

These comparisons are used to

a. identify areas of relatively elevated concentrations of volatile chemicals in soil vapor;
b. select buildings for sub-slab vapor, indoor air and outdoor air sampling;
c. identify possible sources of subsurface vapor contamination;
d. monitor the progress, or verify the completion, of efforts to remediate subsurface vapor contamination (either directly or indirectly); and
e. characterize the nature and extent of subsurface vapor contamination.

When determining appropriate actions, the following should also be considered:

a. Soil vapor results may not indicate a traditional plume-like pattern of contamination (as is often described for groundwater). Rather, the nature and extent of contamination may follow a "hit and miss" pattern.

b. Our experience to date indicates soil vapor results alone typically cannot be relied upon to rule out sampling at nearby buildings. For example, concentrations of volatile chemicals in sub-slab vapor samples have been substantially higher (e.g., by a factor of 100 or more) than concentrations found in nearby soil vapor samples (e.g., collected at 8 feet below grade near the building). This may be due to differences in factors such as soil moisture content and pressure gradients. Therefore, exposures are evaluated primarily based on sub-slab vapor, indoor air and outdoor air sampling results and soil vapor results are primarily used as a tool to guide these investigations.

There are no concentrations of volatile chemicals in soil vapor that automatically trigger action or no further action. Based on the comparisons and considerations described, the following actions may be recommended:

a. *No further soil vapor sampling*

The nature and extent of subsurface vapor contamination has been adequately characterized with respect to addressing exposures and designing measures to remediate subsurface vapor contamination (either directly or indirectly).

Sub-slab vapor samples, rather than soil vapor samples, will be used to identify potential exposures and to characterize the nature and extent of subsurface vapor contamination since soil vapor results are not following a consistent pattern (i.e., hit and miss).

b. *Additional soil vapor sampling*

To characterize the nature and extent of subsurface vapor contamination if soil vapor results are following a consistent pattern (e.g., traditional plume-like pattern).

To identify possible sources of subsurface vapor contamination.

To verify sampling results that appear inconsistent with previous sampling and/or the current understanding of the site [Sections 3.2.1 and 3.2.2].

To resample locations where results may have been invalidated by short-circuiting (outdoor air infiltration), cross contamination, or other problems.

To monitor the progress, or verify the completion, of efforts to remediate subsurface vapor contamination (either directly or indirectly).

c. *Sub-slab vapor, indoor air and outdoor air sampling*

Generally, if soil vapor results are fairly consistent throughout the study area, buildings closest to the site are sampled first. The investigation then proceeds outward, as appropriate, on an areal basis until potential and current human exposures have been adequately addressed. If there is an area of relatively elevated concentrations of volatile chemicals in soil vapor (when looking at the soil vapor results as a whole), then the buildings in this area are also sampled.

d. *Address exposures related to soil vapor intrusion*

Provisions on parcels may be appropriate so that the parcel will not be developed or buildings occupied without addressing exposure concerns [Sections 2.3 and 3.6].

As discussed previously, soil vapor sampling results alone typically do not drive actions to mitigate exposures in existing buildings. Rather, they guide sampling efforts in buildings. However, a "blanket mitigation" approach may be taken provided the nature and extent of soil vapor contamination has been sufficiently characterized. A "blanket mitigation" approach is where an area is defined within which each building may be offered a mitigation system. The offer is made regardless of what actions may be appropriate based on an evaluation of air results (e.g., no further action or monitoring).

Notes:

a. The recommended actions may be modified or supported upon consideration of the factors given in Section 3.2.

b. Additional sampling may become appropriate based on the migration of subsurface contamination (e.g., contaminated groundwater or vapors) or if environmental monitoring indicates a change in chemical constituents (e.g., the production of degradation products that may be more toxic than the parent compounds).

3.3.2 Sub-slab vapor

The goals of collecting sub-slab vapor samples are to identify potential and current (when collected concurrently with indoor and outdoor air samples) exposures associated with soil vapor intrusion and to characterize the nature and extent of subsurface vapor contamination. As discussed in Sections 3.2.5 and 3.3.1, New York State currently does not have any standards, criteria or guidance values for concentrations of compounds in sub-slab vapor. Additionally, there are no databases available of background levels of volatile chemicals in subsurface vapors.

The detection of volatile chemicals in sub-slab vapor samples does not necessarily indicate soil vapor intrusion is occurring or actions should be taken to address exposures. When making these decisions, the State considers the following:

a. the sampling results — sub-slab vapor, indoor air, outdoor air, soil vapor;

b. background concentrations of volatile chemicals in indoor air;

c. the NYSDOH's guidelines for volatile chemicals in air [Table 3.1];

d. human health risks (i.e., cancer and non-cancer health effects) associated with exposure to the volatile chemical in air;

e. attenuation factors (i.e., the ratio of indoor air to sub-slab vapor concentrations),

f. the NYSDOH's decision matrices [described in Section 3.4], and

g. the factors described in Section 3.2.

Based on this evaluation, the following actions may be recommended:

a. *No further action*

When the volatile chemical is not detected in the indoor air and sub-slab sample results are not expected to substantially affect indoor air quality.

b. *Take reasonable and practical actions to identify source(s) and reduce exposures*

The concentration detected in the indoor air sample is likely due to indoor and/or outdoor sources rather than soil vapor intrusion given the concentration detected in the sub-slab vapor sample. Therefore, steps should be taken to identify potential source(s) and to reduce exposures accordingly (e.g., by keeping containers tightly capped or by storing volatile organic compound-containing products in places where people do not spend much time, such as a garage or outdoor shed). Resampling may be recommended to demonstrate the effectiveness of actions taken to reduce exposures.

c. *Resampling*

Resampling may also be recommended when the results are not consistent with the conceptual site model. For example, when the sub-slab vapor results of a building do not indicate a need to take action, but the sub-slab vapor results of adjacent buildings indicate a need to take actions to address exposures related to soil vapor intrusion.

Resampling may be appropriate if samples were collected outside of the heating season. As discussed in Section 2.4.2, results obtained outside of the heating season should not be used to rule out exposures related to soil vapor intrusion.

d. *Monitoring*

Monitoring, including sub-slab vapor, basement air, lowest occupied living space air, and outdoor air sampling, may be recommended to determine whether concentrations in indoor air or sub-slab vapor have changed. It is also recommended to determine what affect, if any, active soil and groundwater remediation techniques (e.g., chemical oxidation, air sparging, etc.) may be having on subsurface vapor and indoor air quality. The type and frequency of monitoring is determined on a site-specific and building-specific basis, taking into account applicable environmental data and building operating conditions.

e. *Mitigate*

Mitigation may be appropriate to minimize current or potential exposures associated with soil vapor intrusion. Mitigation methods are described in Section 4.

Notes:

a. The recommended actions may be modified or supported upon consideration of the factors given in Section 3.2.

b. Additional sampling may be appropriate based on the migration of subsurface contamination (e.g., contaminated groundwater or vapors) or if environmental monitoring indicates a change in chemical constituents (e.g., the production of degradation products that may be more toxic than the parent compounds).

c. Monitoring and mitigation measures to address exposures related to soil vapor intrusion are considered interim measures implemented until contaminated environmental media (e.g., soil, groundwater and/or soil vapor) are remediated.

d. Actions more protective of human health may be proposed. For example, such a decision may be based on a comparison of the costs associated with resampling or monitoring to the costs associated with installation and monitoring of a mitigation system.

e. Additional sampling associated with post-mitigation testing, operation, maintenance and monitoring activities, and termination of mitigation system operations is described in Section 4.

3.3.3 Indoor air

Indoor air samples are used to assess current exposures to volatile chemicals in air. The detection of volatile chemicals in indoor air samples does not necessarily indicate soil vapor intrusion is occurring or actions should be taken to address exposures. When making these decisions, the State considers the following:

a. the sampling results — sub-slab vapor, indoor air, outdoor air, soil vapor;

b. background concentrations of volatile chemicals in indoor air;

c. the NYSDOH's guidelines for volatile chemicals in air [Table 3.1];

d. human health risks (i.e., cancer and non-cancer health effects) associated with exposure to the volatile chemical in air;

e. attenuation factors (i.e., the ratio of indoor air to sub-slab vapor concentrations), and

f. the NYSDOH's decision matrices [described in Section 3.4], and

g. the factors described in Section 3.2.

When evaluating indoor air data, the results are compared to background levels of volatile chemicals in indoor air [Section 3.2.4], the NYSDOH's guidelines for volatile chemicals in air [Table 3.1], the NYSDOH's decision matrices [Section 3.4], and human health risks (i.e., cancer and non-cancer health effects) associated with exposure to the volatile chemical in air. This helps to put the results into perspective and to determine the need for action and the urgency with which actions should be taken. As discussed in Section 3.2.5, the urgency to complete reasonable and practical actions to reduce exposures increases with indoor air levels, particularly when air levels are above a guideline.

Generally, if the results are comparable to background levels, then no further action is needed to address *current* human exposures. However, additional sampling may be appropriate if

a. samples were collected at times when vapor intrusion is not expected to have its greatest effect on indoor air quality (typically, samples collected outside of the heating season). As discussed in Section 2.4, these results may not be used to rule out exposures related to soil vapor intrusion;

b. the potential for exposures related to soil vapor intrusion should be monitored based on the sub-slab vapor results [Section 3.3.2]; and/or

c. subsurface conditions change over time (e.g., due to the migration of contaminated groundwater or vapors).

If the concentrations of volatile chemicals are not consistent with background levels, then the likely cause of the exposure should be determined. Understanding the source is crucial for selecting the best method to address exposures. For example, although a volatile chemical may be detected in the sub-slab vapor sample, the results may indicate that indoor air effects are more likely to be coming from products stored in the building or from outdoor air rather than from contaminated soil vapors. Therefore, a sub-slab depressurization system to minimize exposures associated with soil vapor intrusion may not be appropriate.

As discussed in Sections 1.4 and 3.2.3, volatile chemicals may be present in the indoor air due to any one, or a combination, of the following:

a. the indoor environment itself and/or building characteristics;

b. off-gassing of volatile chemicals from contaminated water that may enter the building at the tap or shower head, or during flooding events, or contaminated water that rests in a sump or a subsurface drain;

c. outdoor sources; and/or

d. migration from the subsurface (i.e., soil vapor intrusion).

To determine the likely cause, the following assessment is completed:

a. qualitative and quantitative comparisons are made between the types and concentrations of the contaminants found in the indoor air sample(s) and those found in the outdoor air and sub-slab vapor sample;

b. qualitative and quantitative comparisons are made between indoor air results obtained in different locations of the building (e.g., different floors or rooms);

c. indoor air results are compared to the product inventory to evaluate the extent to which indoor sources are affecting indoor air quality; and

d. the indoor air quality questionnaire and building inventory form is reviewed to identify potential preferential pathways for soil vapor intrusion into the building, potential outdoor sources of volatile chemicals to the outdoor air (e.g., gasoline station or dry cleaner), and routes of air distribution within the building (e.g., HVAC system operations, airflow observations, etc.).

If a likely source or multiple sources can be identified from the available information, one or more of the following actions may be recommended given the source:

a. *Indoor source or building characteristics*

Products containing volatile chemicals should be tightly capped. Alternatively, the products can be stored in places where people do not spend much time, such as a garage or outdoor shed. If the products are no longer needed, consideration should be given to disposing of them properly (e.g., hazardous waste cleanup days). The list of products and corresponding readings from field instrumentation provided in the product inventory [Appendix B] can help identify products that may be contributing to the levels that were detected in the indoor air.

If exposures are assumed to be associated with off-gassing of new building materials, paint, etc., resampling may be appropriate to confirm this assumption or to confirm that actions taken to address these exposures have been effective.

b. *Off-gassing from contaminated groundwater within the building*

Measures should be taken to prevent contaminated groundwater from entering the house (e.g., filter on private well supply, sealed sump, etc.).

c. *Outdoor source*

No further action to address exposures related to soil vapor intrusion, unless the evaluation for soil vapor intrusion cannot be completed until outdoor interferences are addressed.

d. *Soil vapor intrusion*

Depending upon the relationship between indoor air concentrations and sub-slab vapor concentrations and the results of environmental sampling in the area, resampling, monitoring or mitigation may be recommended by the State.

1. Resampling, including sub-slab vapor, basement air, lowest occupied living space air, and outdoor air sampling, may be recommended when the results are not consistent with the conceptual site model. For example, when indoor air results are comparable or higher than the corresponding sub-slab vapor results and the results do not appear to be due to building characteristics or alternate sources (either indoor or outdoor).

2. Monitoring, including sub-slab vapor, basement air, lowest occupied living space air, and outdoor air sampling, may be recommended to determine whether concentrations in indoor air or sub-slab vapor have changed. It is also recommended to determine what affect, if any, active soil and groundwater remediation techniques (e.g., chemical oxidation, air sparging, etc.) may be having on subsurface vapor and indoor air quality. The type and frequency of monitoring is determined on a site-specific and building-specific basis, taking into account applicable environmental data and building operating conditions.

3. Methods to mitigate exposures related to soil vapor intrusion are described in Section 4.

The party responsible for implementing the recommended actions will differ depending upon several factors, including the identified source of the volatile chemicals, the environmental remediation program, and site-specific and building-specific conditions. For example, to the extent that all site data and site conditions demonstrate that soil vapor intrusion is not occurring and that the potential for soil vapor intrusion to occur is not likely, the vapor intrusion investigation would be considered complete. In general, if indoor exposures represent a concern due to indoor sources, then the State will provide guidance to the property owner and/or tenant on ways to reduce their exposure. If indoor exposures represent a concern due to outdoor sources, then the NYSDEC will decide who is responsible for further investigation and any necessary remediation. Depending upon the outdoor source, this responsibility may or may not fall upon the party conducting the soil vapor intrusion investigation.

Likely sources may not be evident given the information available. Therefore, the above recommendations cannot be made. This situation most often arises for the following reasons:

a. Interfering indoor sources are identified. However, the possibility of vapor intrusion cannot be ruled out due to the concentrations of the same volatile chemicals detected in the sub-slab vapor sample. Differentiating the contribution of each source is not possible.

b. Indoor air samples were collected without concurrent outdoor air and sub-slab vapor samples. Depending upon other information that may be available (e.g., building inventory and well-characterized subsurface vapor contamination), identifying likely sources and recommending appropriate actions may not be possible.

c. All appropriate air samples are collected. However, the indoor air quality questionnaire and building inventory forms are filled out incompletely or incorrectly. The contribution of indoor sources cannot be evaluated.

When the source(s) of volatile chemicals to indoor air cannot be identified with confidence, resampling is typically recommended with corrections made as appropriate. For example, using the three scenarios presented above:

a. resampling occurs after interferences are removed;

b. concurrent indoor air, outdoor air and sub-slab vapor samples are collected; and

c. an indoor air quality questionnaire and building inventory form is filled out completely and correctly when samples are collected.

Notes: See notes presented in Section 3.3.2.

3.3.4 Outdoor air

Outdoor air sampling results are primarily used to evaluate the extent to which outdoor air may be contributing to the levels of volatile chemicals detected in indoor air. However, people are also exposed to the outdoor air and the outdoor air results are indicative of outdoor air conditions. As such, outdoor air results are also reviewed to determine whether outdoor air conditions present a potential concern that requires further investigation.

As discussed in Sections 1.4 and 3.2.3, volatile chemicals may be present in outdoor air due to emissions from automobiles, lawn mowers, oil storage tanks, gasoline stations, and dry cleaners or other commercial and industrial facilities. To determine what extent, if any, outdoor air is affecting indoor air quality, indoor air results are compared to outdoor air results. To determine whether outdoor air conditions present a potential concern that requires further investigation, the State looks at the data set as a whole and considers the following:

a. background concentrations of volatile chemicals in outdoor air;

b. the NYSDOH's guidelines for volatile chemicals in air [Table 3.1];

c. human health risks (i.e., cancer and non-cancer health effects) associated with exposure to the volatile chemical in air; and

d. the factors described in Section 3.2.

3.4 Decision matrices

3.4.1 Overview

Decision matrices are risk management tools, developed by the NYSDOH in conjunction with other agencies, to provide guidance on a case-by-case basis about actions that should be taken to address current and potential exposures related to soil vapor intrusion. The matrices are intended to be used when evaluating the results from buildings with full slab foundations. The matrices encapsulate the data evaluation processes and actions recommended to address exposures discussed in Sections 3.3.2 and 3.3.3. The general format of a decision matrix is shown in Table 3.2.

Table 3.2 General format of a decision matrix

	Indoor Air Concentration of Volatile Chemical (mcg/m^3)		
Sub-slab Vapor Concentration of Volatile Chemical (mcg/m^3)	Concentration Range 1	Concentration Range 2	Concentration Range 3
Concentration Range 1	ACTION	ACTION	ACTION
Concentration Range 2	ACTION	ACTION	ACTION
Concentration Range 3	ACTION	ACTION	ACTION

Indoor air and sub-slab vapor concentration ranges in a matrix are selected based on a number of considerations in addition to health risks. For example, factors that are considered when selecting the ranges include, but are not limited to, the following:

a. human health risks (i.e., cancer and non-cancer health effects) associated with exposure to the volatile chemical in air;

b. the NYSDOH's guidelines for volatile chemicals in air [Table 3.1];

c. background concentrations of volatile chemicals in air [Section 3.2.4];

d. analytical capabilities currently available; and

e. attenuation factors (i.e., the ratio of indoor air to sub-slab vapor concentrations).

3.4.2 Matrices

The NYSDOH has developed two matrices, which are included at the end of Section 3.4, to use as tools in making decisions when soil vapor may be entering buildings. The first decision matrix was originally developed for TCE and the second for PCE. As summarized in Table 3.3, four chemicals have been assigned to the two matrices to date.

Table 3.3 Volatile chemicals and their decision matrices

Chemical	**Soil Vapor/Indoor Air Matrix***
Carbon tetrachloride	Matrix 1
Tetrachloroethene (PCE)	Matrix 2
1,1,1-Trichloroethane (1,1,1-TCA)	Matrix 2
Trichloroethene (TCE)	Matrix 1

*The decision matrices are available at the end of Section 3.4.

Because the matrices are risk management tools and consider a number of factors, the NYSDOH intends to assign chemicals to one of these two matrices, if possible. For example, if a chemical other than those already assigned to a matrix is identified as a chemical of concern during a soil vapor intrusion investigation, assignment of that chemical into one of the existing decision matrices will be considered by the NYSDOH. Factors that will be considered in assigning a chemical to a matrix include, but are not limited to, the following:

a. human health risks, including such factors as a chemical's ability to cause cancer, reproductive, developmental, liver, kidney, nervous system, immune system or other effects, in animals and humans and the doses that may cause those effects;

b. the data gaps in its toxicologic database;

c. background concentrations of volatile chemicals in indoor air [Section 3.2.4]; and

d. analytical capabilities currently available.

If the NYSDOH determines that the assignment of the chemical into an existing matrix is inappropriate, then the NYSDOH will either modify an existing matrix or develop a new matrix.

To use the matrices appropriately as a tool in the decision-making process, the following should be considered:

a. The matrices are generic. As such, it may be appropriate to modify a recommended action to accommodate building-specific conditions (e.g., dirt floor in basement, crawl spaces, etc.) and/or factors provided in Section 3.2 of the guidance (e.g., current land use, environmental conditions, etc.). For example, resampling may be recommended when the matrix indicates "no further action" for a particular building, but the results of adjacent buildings (especially sub-slab vapor results) indicate a need to take actions to address exposures related to soil vapor intrusion. Additionally, actions more protective of public health than those specified within the matrix may be proposed at any time. For example, the party implementing the actions may decide to install sub-slab depressurization systems on buildings where the matrix indicates "no further action" or "monitoring." Such an action is usually undertaken for reasons other than public health (e.g., seeking community acceptance, reducing excessive costs, etc.).

b. Indoor air concentrations detected in samples collected from the building's basement or, if the building has a slab-on-grade foundation, from the building's lowest occupied living space should be used.

c. Actions provided in the matrix are specific to addressing human exposures. Implementation of these actions does not preclude investigating possible sources of vapor contamination, nor does it preclude remediating contaminated soil vapors or the source of soil vapor contamination.

d. When current exposures are attributed to sources other than vapor intrusion, the agencies should be provided documentation(e.g., applicable environmental data, completed indoor air sampling questionnaire, digital photographs, etc.) to support a proposed action other than that provided in the matrix and to support assessment and follow-up by the agencies.

3.4.3 Description of recommended actions

Actions recommended in the matrix are based on the relationship between sub-slab vapor concentrations and corresponding indoor air concentrations. They are intended to address both potential and current human exposures and include the following:

a. *No further action*

When the volatile chemical is not detected in the indoor air sample and the concentration detected in the corresponding sub-slab vapor sample is not expected to substantially affect indoor air quality.

b. *Take reasonable and practical actions to identify source(s) and reduce exposures*

The concentration detected in the indoor air sample is likely due to indoor and/or outdoor sources rather than soil vapor intrusion given the concentration detected in the sub-slab vapor sample. Therefore, steps should be taken to identify potential source(s) and to reduce exposures accordingly (e.g., by keeping containers tightly capped or by storing volatile chemical-containing products in places where people do not spend much time, such as a garage or shed). Resampling may also be recommended to demonstrate the effectiveness of actions taken to reduce exposures.

d. *Monitor*

Monitoring, including sub-slab vapor, basement air, lowest occupied living space air, and outdoor air sampling, is appropriate to determine whether concentrations in the indoor air or sub-slab vapor have changed. Monitoring may also be appropriate to determine whether existing building conditions (e.g., positive pressure HVAC systems) are maintaining the desired mitigation endpoint and to determine whether changes are appropriate.

The type and frequency of monitoring is determined on a site-specific and building-specific basis, taking into account applicable environmental data and building operating conditions.

e. *Mitigate*

Mitigation is appropriate to minimize current or potential exposures associated with soil vapor intrusion. Methods to mitigate exposures related to soil vapor intrusion are described in Section 4.

f. *Monitor / Mitigate*

Monitoring or mitigation may be recommended after considering the magnitude of sub-slab vapor and indoor air concentrations along with building- and site-specific conditions.

Soil Vapor / Indoor Air Matrix 1

October 2006

	INDOOR AIR CONCENTRATION of COMPOUND (mcg/m^3)			
SUB-SLAB VAPOR CONCENTRATION of COMPOUND (mcg/m^3)	< 0.25	0.25 to < 1	1 to < 5.0	5.0 and above
< 5	1. No further action	2. Take reasonable and practical actions to identify source(s) and reduce exposures	3. Take reasonable and practical actions to identify source(s) and reduce exposures	4. Take reasonable and practical actions to identify source(s) and reduce exposures
5 to < 50	5. No further action	6. MONITOR	7. MONITOR	8. MITIGATE
50 to < 250	9. MONITOR	10. MONITOR / MITIGATE	11. MITIGATE	12. MITIGATE
250 and above	13. MITIGATE	14. MITIGATE	15. MITIGATE	16. MITIGATE

No further action:
Given that the compound was not detected in the indoor air sample and that the concentration detected in the sub-slab vapor sample is not expected to significantly affect indoor air quality, no additional actions are needed to address human exposures.

Take reasonable and practical actions to identify source(s) and reduce exposures:
The concentration detected in the indoor air sample is likely due to indoor and/or outdoor sources rather than soil vapor intrusion given the concentration detected in the sub-slab vapor sample. Therefore, steps should be taken to identify potential source(s) and to reduce exposures accordingly (e.g., by keeping containers tightly capped or by storing volatile organic compound-containing products in places where people do not spend much time, such as a garage or outdoor shed). Resampling may be recommended to demonstrate the effectiveness of actions taken to reduce exposures.

MONITOR:
Monitoring, including sub-slab vapor, basement air, lowest occupied living space air, and outdoor air sampling, is needed to determine whether concentrations in the indoor air or sub-slab vapor have changed. Monitoring may also be needed to determine whether existing building conditions (e.g., positive pressure heating, ventilation and air-conditioning systems) are maintaining the desired mitigation endpoint and to determine whether changes are needed. The type and frequency of monitoring is determined on a site-specific and building-specific basis, taking into account applicable environmental data and building operating conditions. Monitoring is an interim measure required to evaluate exposures related to soil vapor intrusion until contaminated environmental media are remediated.

MITIGATE:
Mitigation is needed to minimize current or potential exposures associated with soil vapor intrusion. The most common mitigation methods are sealing preferential pathways in conjunction with installing a sub-slab depressurization system, and changing the pressurization of the building in conjunction with monitoring. The type, or combination of types, of mitigation is determined on a building-specific basis, taking into account building construction and operating conditions. Mitigation is considered a temporary measure implemented to address exposures related to soil vapor intrusion until contaminated environmental media are remediated.

MONITOR / MITIGATE:
Monitoring or mitigation may be recommended after considering the magnitude of sub-slab vapor and indoor air concentrations along with building- and site-specific conditions.

See additional notes on page 2.

ADDITIONAL NOTES FOR MATRIX 1

This matrix summarizes the minimum actions recommended to address current and potential exposures related to soil vapor intrusion. To use the matrix appropriately as a tool in the decision-making process, the following should be noted:

[1] The matrix is generic. As such, it may be appropriate to modify a recommended action to accommodate building-specific conditions (e.g., dirt floor in basement, crawl spaces, etc.) and/or factors provided in Section 3.2 of the guidance (e.g., current land use, environmental conditions, etc.). For example, resampling may be recommended when the matrix indicates "no further action" for a particular building, but the results of adjacent buildings (especially sub-slab vapor results) indicate a need to take actions to address exposures related to soil vapor intrusion. Additionally, actions more protective of public health than those specified within the matrix may be proposed at any time. For example, the party implementing the actions may decide to install sub-slab depressurization systems on buildings where the matrix indicates "no further action" or "monitoring." Such an action is usually undertaken for reasons other than public health (e.g., seeking community acceptance, reducing excessive costs, etc.).

[2] Actions provided in the matrix are specific to addressing human exposures. Implementation of these actions does not preclude investigating possible sources of vapor contamination, nor does it preclude remediating contaminated soil vapors or the source of soil vapor contamination.

[3] Appropriate care should be taken during all aspects of sample collection to ensure that high quality data are obtained. Since the data are being used in the decision-making process, the laboratory analyzing the environmental samples must have current Environmental Laboratory Approval Program (ELAP) certification for the appropriate analyte and environmental matrix combinations. Furthermore, samples should be analyzed by methods that can achieve a minimum reporting limit of 0.25 microgram per cubic meter for indoor and outdoor air samples. For sub-slab vapor samples, a minimum reporting limit of 5 micrograms per cubic meter is recommended for buildings with full slab foundations, and 1 microgram per cubic meter for buildings with less than a full slab foundation.

[4] Sub-slab vapor and indoor air samples are typically collected when the likelihood of soil vapor intrusion to occur is considered to be the greatest (i.e., worst-case conditions). If samples are collected at other times (typically, samples collected outside of the heating season), then resampling during worst-case conditions may be appropriate to verify that actions taken to address exposures related to soil vapor intrusion are protective of human health.

[5] When current exposures are attributed to sources other than soil vapor intrusion, the agencies should be given documentation (e.g., applicable environmental data, completed indoor air sampling questionnaire, digital photographs, etc.) to support a proposed action other than that provided in the matrix box and to support agency assessment and follow-up.

[6] The party responsible for implementing the recommended actions will differ depending upon several factors, including the identified source of the volatile chemicals, the environmental remediation program, and site-specific and building-specific conditions. For example, to the extent that all site data and site conditions demonstrate that soil vapor intrusion is not occurring and that the potential for soil vapor intrusion to occur is not likely, the soil vapor intrusion investigation would be considered complete. In general, if indoor exposures represent a concern due to indoor sources, then the State will provide guidance to the property owner and/or tenant on ways to reduce their exposure. If indoor exposures represent a concern due to outdoor sources, then the NYSDEC will decide who is responsible for further investigation and any necessary remediation. Depending upon the outdoor source, this responsibility may or may not fall upon the party conducting the soil vapor intrusion investigation.

Soil Vapor / Indoor Air Matrix 2

October 2006

	INDOOR AIR CONCENTRATION of COMPOUND (mcg/m^3)			
SUB-SLAB VAPOR CONCENTRATION of COMPOUND (mcg/m^3)	< 3	3 to < 30	30 to < 100	100 and above
< 100	1. No further action	2. Take reasonable and practical actions to identify source(s) and reduce exposures	3. Take reasonable and practical actions to identify source(s) and reduce exposures	4. Take reasonable and practical actions to identify source(s) and reduce exposures
100 to < 1,000	5. MONITOR	6. MONITOR / MITIGATE	7. MITIGATE	8. MITIGATE
1,000 and above	9. MITIGATE	10. MITIGATE	11. MITIGATE	12. MITIGATE

No further action:
Given that the compound was not detected in the indoor air sample and that the concentration detected in the sub-slab vapor sample is not expected to significantly affect indoor air quality, no additional actions are needed to address human exposures.

Take reasonable and practical actions to identify source(s) and reduce exposures:
The concentration detected in the indoor air sample is likely due to indoor and/or outdoor sources rather than soil vapor intrusion given the concentration detected in the sub-slab vapor sample. Therefore, steps should be taken to identify potential source(s) and to reduce exposures accordingly (e.g., by keeping containers tightly capped or by storing volatile organic compound-containing products in places where people do not spend much time, such as a garage or outdoor shed). Resampling may be recommended to demonstrate the effectiveness of actions taken to reduce exposures.

MONITOR:
Monitoring, including sub-slab vapor, basement air, lowest occupied living space air, and outdoor air sampling, is needed to determine whether concentrations in the indoor air or sub-slab vapor have changed. Monitoring may also be needed to determine whether existing building conditions (e.g., positive pressure heating, ventilation and air-conditioning systems) are maintaining the desired mitigation endpoint and to determine whether changes are needed. The type and frequency of monitoring is determined on a site-specific and building-specific basis, taking into account applicable environmental data and building operating conditions. Monitoring is an interim measure required to evaluate exposures related to soil vapor intrusion until contaminated environmental media are remediated.

MITIGATE:
Mitigation is needed to minimize current or potential exposures associated with soil vapor intrusion. The most common mitigation methods are sealing preferential pathways in conjunction with installing a sub-slab depressurization system, and changing the pressurization of the building in conjunction with monitoring. The type, or combination of types, of mitigation is determined on a building-specific basis, taking into account building construction and operating conditions. Mitigation is considered a temporary measure implemented to address exposures related to soil vapor intrusion until contaminated environmental media are remediated.

MONITOR / MITIGATE:
Monitoring or mitigation may be recommended after considering the magnitude of sub-slab vapor and indoor air concentrations along with building- and site-specific conditions.

See additional notes on page 2.

ADDITIONAL NOTES FOR MATRIX 2

This matrix summarizes the minimum actions recommended to address current and potential exposures related to soil vapor intrusion. To use the matrix appropriately as a tool in the decision-making process, the following should be noted:

[1] The matrix is generic. As such, it may be appropriate to modify a recommended action to accommodate building-specific conditions (e.g., dirt floor in basement, crawl spaces, etc.) and/or factors provided in Section 3.2 of the guidance (e.g., current land use, environmental conditions, etc.). For example, resampling may be recommended when the matrix indicates "no further action" for a particular building, but the results of adjacent buildings (especially sub-slab vapor results) indicate a need to take actions to address exposures related to soil vapor intrusion. Additionally, actions more protective of public health than those specified within the matrix may be proposed at any time. For example, the party implementing the actions may decide to install sub-slab depressurization systems on buildings where the matrix indicates "no further action" or "monitoring." Such an action is usually undertaken for reasons other than public health (e.g., seeking community acceptance, reducing excessive costs, etc.).

[2] Actions provided in the matrix are specific to addressing human exposures. Implementation of these actions does not preclude investigating possible sources of vapor contamination, nor does it preclude remediating contaminated soil vapors or the source of soil vapor contamination.

[3] Appropriate care should be taken during all aspects of sample collection to ensure that high quality data are obtained. Since the data are being used in the decision-making process, the laboratory analyzing the environmental samples must have current Environmental Laboratory Approval Program (ELAP) certification for the appropriate analyte and environmental matrix combinations. Furthermore, samples should be analyzed by methods that can achieve a minimum reporting limit of 3 micrograms per cubic meter for indoor and outdoor air samples. For sub-slab vapor samples, a minimum reporting limit of 5 micrograms per cubic meter is recommended.

[4] Sub-slab vapor and indoor air samples are typically collected when the likelihood of soil vapor intrusion to occur is considered to be the greatest (i.e., worst-case conditions). If samples are collected at other times (typically, samples collected outside of the heating season), then resampling during worst-case conditions may be appropriate to verify that actions taken to address exposures related to soil vapor intrusion are protective of human health.

[5] When current exposures are attributed to sources other than soil vapor intrusion, the agencies should be given documentation (e.g., applicable environmental data, completed indoor air sampling questionnaire, digital photographs, etc.) to support a proposed action other than that provided in the matrix box and to support agency assessment and follow-up.

[6] The party responsible for implementing the recommended actions will differ depending upon several factors, including the identified source of the volatile chemicals, the environmental remediation program, and site-specific and building-specific conditions. For example, to the extent that all site data and site conditions demonstrate that soil vapor intrusion is not occurring and that the potential for soil vapor intrusion to occur is not likely, the soil vapor intrusion investigation would be considered complete. In general, if indoor exposures represent a concern due to indoor sources, then the State will provide guidance to the property owner and/or tenant on ways to reduce their exposure. If indoor exposures represent a concern due to outdoor sources, then the NYSDEC will decide who is responsible for further investigation and any necessary remediation. Depending upon the outdoor source, this responsibility may or may not fall upon the party conducting the soil vapor intrusion investigation.

3.5 Emergency response

The NYSDOH's staff are responsible for recommending that residents relocate in cases where there may be health risks resulting from exposure to petroleum spills. These roles and responsibilities are outlined in Environmental Health Manual Technical Reference and Procedural Items BTSA-01. Air sampling is appropriate in some cases for demonstrating that spill cleanup and engineering controls have been effective in reducing indoor air impacts and associated health risks to residents. At a minimum, air samples are collected from the basement, first floor and from outdoors. Whether sub-slab or soil gas samples will be taken is evaluated on a case-by-case basis. Air testing data are sometimes used as the basis for ending emergency relocation financial support. For additional information, please contact the NYSDOH's Bureau of Toxic Substance Assessment by calling 1-800-458-1158.

Emergency actions not related to petroleum spills are handled on a case-by-case basis.

3.6 Parcels that are undeveloped or contain unoccupied buildings

If investigation of a parcel that is undeveloped or contains unoccupied buildings is being delayed until the site is being developed or occupied, measures should be in place that assure the State that no development or occupation will occur without addressing the exposures. Institutional controls may be used for this purpose. An institutional control is any non-physical means of enforcing a restriction on the use of real property that

a. limits human or environmental exposure,

b. provides notice to potential owners, operators or members of the public, or

c. prevents actions that would interfere with the effectiveness of remedial actions or with the effectiveness and/or integrity of operation, maintenance or monitoring activities at a site.

An institutional control that is often used is an environmental easement. An environmental easement is an enforced mechanism used for property where the remedial actions leave residual contamination that makes the property suitable for some, but not all uses, or includes engineering controls that must be maintained for the easement to be effective. The purpose of the easement is to ensure that such use restrictions or engineering controls remain in place. An environmental easement

a. can only be created by the property owner (the *grantor*) through a written instrument recorded in the appropriate county recording office. It can only be granted to the State (the *grantee*) and can only be extinguished or amended by a written instrument executed by the Commissioner of the Department of Environmental Conservation and duly recorded;

b. is binding upon all subsequent owners and occupants of the property. The deed or deeds for the property (as well as any other written instruments conveying any interest in the property) must contain a prominent notice that it is subject to an environmental easement; and

c. may be enforced in perpetuity against the *grantor*, subsequent owners of the property, lessees, and any person using the property by its grantor, by the State, or by the municipality in which the property is located.

If these actions cannot be implemented, alternative measures should be in place that assure the State that the parcel will not be developed or buildings occupied without addressing the exposure concerns. For example, arrangements should be made for the town, village or city

to notify the appropriate party when new construction or tenants are proposed for the parcel (e.g., permit applications and grants) or ownership of the parcel changes.

Section 4: Soil Vapor Intrusion Mitigation

As discussed in Section 1.1, soil vapor can enter a building through cracks or perforations in slabs or basement floors and walls, and through openings around sump pumps or where pipes and electrical wires go through the foundation primarily because of a difference between interior and exterior pressures. This intrusion is similar to how radon gas enters buildings from the subsurface. Fortunately, given this similarity, well-established techniques for mitigating exposures to radon may also be used to mitigate exposures related to soil vapor intrusion.

Once it is determined that steps should to be taken to address exposures associated with soil vapor intrusion, they should be implemented with all due expediency. This section provides an overview of:

a. methods of mitigation,
b. installation and design of mitigation systems,
c. post-mitigation testing,
d. operation, maintenance and monitoring of mitigation systems,
e. termination of mitigation system operations, and
f. annual certification.

Mitigation is considered to be an interim measure to address exposures until contaminated environmental media are remediated, or until mitigation is no longer needed to address exposures related to soil vapor intrusion.

4.1 Methods of mitigation

The most effective mitigation methods involve sealing infiltration points and actively manipulating the pressure differential between the building's interior and exterior (on a continuous basis). As discussed in the following subsections, the appropriate method to use will largely depend upon the building's foundation design. Furthermore, buildings having more than one foundation design feature (e.g., a basement under one portion of the house and a crawl space beneath the remainder) may require a combination of mitigation methods. This section describes methods of mitigation that are expected to be the most reliable options under a wide range of circumstances. Occasionally, there are site-specific or building-specific conditions under which alternative methods (such as HVAC modification, sealing, room pressurization, passive ventilation systems, or vapor barriers) may be more appropriate. Such mitigation proposals may be considered on a case-by-case basis.

4.1.1 Buildings with a basement slab or slab-on-grade foundation

In conjunction with *sealing* potential subsurface vapor entry points, an active *sub-slab depressurization system* (SSD system) is the preferred mitigation method for buildings with a basement slab or slab-on-grade foundation. A SSD system uses a fan-powered vent and piping to draw vapors from the soil beneath the building's slab (i.e., essentially creating a vacuum beneath the slab) and discharge them to the atmosphere. This results in lower sub-slab air pressure relative to indoor air pressure, which prevents the infiltration of sub-slab vapors into the building.

The most common approach to achieving depressurization beneath the slab is to insert the piping through the floor slab into the crushed rock or soil underneath. However, the EPA, in their "Consumer's Guide to Radon Reduction" (EPA 402-K-03-002; revised February 2003), lists the following approaches as ways to reduce radon levels in a building, either in place of the more common sub-slab suction point method or in conjunction with that method:

a. *Drain tile suction* — Some houses have drain tiles or perforated pipe to direct water away from the foundation of the house. Suction on these tiles or pipes is often effective;

b. *Sump hole suction* — If the building has a sump pump to remove unwanted water, the sump can be capped so that it can continue to drain water and serve as the location for piping. If the sump is not used as the suction or extraction point, the associated wiring and piping should be sealed and an air-tight cover should be installed to enhance the performance of the SSD system; and

c. *Block wall suction* — If the building has hollow block foundation walls, the void network within the wall may be depressurized by drawing air from inside the wall and venting it to the outside. This method is often used in combination with sub-slab depressurization.

The depressurization approach, or combination of approaches, selected for a building should be determined on a building-specific basis due to building-specific features that may be conducive to a specific depressurization approach. For example, if the contaminants are entering the building through a block wall, block wall suction in conjunction with traditional sub-slab depressurization may be more effective at minimizing exposures related to soil vapor intrusion rather than sub-slab depressurization alone.

Although sealing is not a reliable mitigation technique on its own, it can significantly improve the effectiveness of a SSD system since it limits the flow of subsurface vapors into the building. All joints, cracks and other penetrations of slabs, floor assemblies and foundation walls below or in contact with the ground surface should be sealed with materials that prevent air leakage.

If the State concurs that a SSD system is not a practicable alternative or that exposures will be mitigated concurrently by a method selected to remediate subsurface contamination, alternative mitigation methods may be considered, such as the following:

a. *HVAC modification* — a technique where the building's HVAC system is modified to avoid depressurization of the building relative to underlying and surrounding soil (i.e., to maintain a positive pressure within the building); and

b. S*oil vapor extraction (SVE) system* — a technique used to remediate contaminated subsurface soil vapor. SVE systems use high flow rates, induced vacuum or both to collect and remove contamination, while SSD systems use a minimal flow rate to effect the minimum pressure gradient (see the EPA's technical guidance documents for recommended gradients; Section 4.2.3) needed to reverse air flow across a building's foundation. Depending upon the SVE system's design, the system may also serve to mitigate exposures. For example, the SVE system's radius of influence includes the subsurface beneath affected buildings or horizontal legs of the system will be installed beneath affected buildings. However, complications can arise if the SVE system is no longer effective at remediating contaminated vapors, exposures should still be mitigated due to residual vapor contamination.

4.1.2 Buildings with a crawl space foundation

A *soil vapor retarder* with *sub-membrane depressurization (SMD) system* is the preferred mitigation method for buildings with a crawl space foundation. A soil vapor retarder is a synthetic membrane or other comparable material that is placed on the ground in the crawl space to retard the flow of soil vapors into the building. A SMD system is similar to a SSD system. It uses a fan-powered vent and piping to draw vapors from beneath the soil vapor retarder and discharge them to the atmosphere. This results in lower air pressure beneath the membrane relative to air pressure in the crawl space, which prevents the infiltration of subsurface vapors into the building.

If the State concurs that a soil vapor retarder with a SMD system is not a practicable alternative or that exposures will be mitigated concurrently by a method selected to remediate subsurface contamination, alternative mitigation methods may be considered, such as the following:

a. *HVAC modification* — a technique where the building's HVAC system is modified to avoid depressurization of the building relative to the crawl space;

b. C*rawl space ventilation with sealing* — a technique that uses a fan to draw air out of the crawl space; and

c. *SVE system* [Section 4.1.1].

4.1.3 Buildings with dirt floor basements

Either a SSD system with a newly poured slab or a SMD system with a soil vapor retarder may be used. However, the former method is preferred.

4.1.4 Buildings with multiple foundation types

Mitigation in a building with a combination of foundations should be achieved by applying the specific methods described previously [Sections 4.1.1 through 4.1.3] to the corresponding foundation segments of the building. Special consideration should be given to the points at which different foundation types join, since additional soil vapor entry routes exist in such locations. Often, the various systems can be installed and connected to a common depressurization system and fan.

4.1.5 Undeveloped parcels

If sampling results indicate a mitigation system is recommended to address exposures in buildings that may be constructed, then a SSD system with sealing, or a SMD system with a soil vapor retarder, or a combination of these methods is recommended, as appropriate to the design of the proposed buildings.

4.1.6 Additional references

The following documents provide additional information on selecting an appropriate mitigation method:

a. *A Consumer's Guide to Radon Reduction*
EPA [EPA 402-K-03-002, revised February 2003]

This document provides assistance in selecting a qualified radon mitigation contractor to reduce the radon levels in a home, determining an appropriate radon reduction method, and maintaining a radon reduction system. It is available at the EPA's web site: http://www.epa.gov/iaq/radon/pubs/index.html; and

b. *Reducing Radon in Schools: A Team Approach*
EPA [EPA 402-R-94-008, April 1994]

This document will provide assistance in determining the best way to reduce elevated radon levels found in a school. It provides guidance on the process of confirming a radon problem, selecting the best mitigation strategy, and directing the efforts of a multidisciplinary team assembled to address elevated radon levels in a way that will contribute to the improvement of the overall indoor air quality of the school. Copies can be ordered from the EPA's Indoor Air Quality Information Clearinghouse at 1-800-438-4318.

4.2 Design and installation of mitigation systems

Once a mitigation method is selected, it should be designed and installed. The components of the design and installation of mitigation systems, the procedures for specific mitigation techniques, and references for technical guidance are provided in the following subsections.

4.2.1 General recommendations

Systems should be designed and installed by a professional engineer or environmental professional. In most areas of the state, there are contractors who have met certain requirements and are trained to identify and fix radon problems in buildings. To obtain the names of local contractors, contact the NYSDOH's Radon Program at 1-800-458-1158, extension 27556, or visit the National Radon Safety Board's web site (www.nrsb.org) or National Environmental Health Association's web site (www.neha.org).

Typically, the party responsible for remediating the site is responsible for arranging design and installation activities. If no responsible party is available, the State will arrange for the design and installation of the system. All design and installation activities should be documented and reported to the agencies. Furthermore, once a mitigation system is installed, an information package should be given to the building's owner and tenants, if applicable, to facilitate their understanding of the system's operation, maintenance and monitoring [Section 5.6].

With the exception of SVE systems, the mitigation methods introduced in Section 4.1 are not intended to remediate the source of subsurface vapors (e.g., contaminated groundwater, soil, etc.). Rather, they are designed to minimize the infiltration of subsurface vapors into a building. For consistency in implementing the techniques in residential buildings, mitigation systems should be designed and installed in accordance with the following:

a. *Standard Practice for Installing Radon Mitigation Systems in Existing Low-rise Residential Buildings* (ASTM E-2121)

American Society for Testing and Materials (ASTM) International [ASTM E-2121-03, February 10, 2003]

This document applies to existing buildings. The purpose of this document is to provide radon mitigation contractors with uniform standards that will ensure quality and effectiveness in the design, installation, and evaluation of radon mitigation systems in detached and attached residential buildings three stories or less in height. Information on how to obtain a copy of this standard is available in Appendix E; and

b. *Model Standards and Techniques for Control of Radon in New Residential Buildings* EPA [EPA 402-R-94-009, March 1994]

This document applies to new construction and contains information on how to incorporate radon reduction techniques and materials in residential construction. A copy of this document is provided in Appendix F.

4.2.2 System-specific recommendations

Basic design and installation recommendations for mitigation systems follow. These are based upon recommendations and requirements given by the EPA for mitigating exposures related to radon intrusion (for additional information see EPA's web site on radon at http://www.epa.gov/iaq/radon/pubs/index.html).

a. *Sealing* — To improve the effectiveness of depressurization and ventilation systems and to limit the flow of subsurface vapors into the building, materials that prevent air leakage should be used, such as elastomeric joint sealant (as defined in ASTM C920-87), compatible caulks, non-shrink mortar, grouts, expanding foam, "Dranjer" drain seals, or airtight gaskets. Some effective sealants may contain volatile organic compounds; in some situations, this may be a consideration in choosing an appropriate sealing material.

b. *Soil vapor retarder (membrane)* —

1. To retard the infiltration of subsurface vapors into the building and enhance the performance of a SMD system, a minimum 6 mil (or 3 mil cross-laminated) polyethylene or equivalent flexible sheeting material should be used.

2. The sheet should cover the entire floor area and be sealed at seams (with at least a 12 inch overlap) and penetrations, around the perimeter of interior piers and to the foundation walls.

3. Enough of the sheeting should be used so it will not be pulled away from the walls when the depressurization system is turned on and the sheet is drawn down.

4. If a membrane is installed in areas that may have future foot traffic (e.g., a dirt floor in a basement), consideration should be given to also installing a wearing surface such as sand or stone to protect the integrity of the membrane. Additionally, a layer of fine sand may be prudent beneath the membrane to protect it from penetrations by sharp objects in the dirt floor.

c. *Depressurization systems —*

1. The systems should be designed to avoid the creation of other health, safety, or environmental hazards to building occupants (e.g., backdrafting of natural draft combustion appliances).

2. The systems should be designed to minimize soil vapor intrusion effectively while minimizing excess energy usage, to avoid compromising moisture and temperature controls and other comfort features, and to minimize noise.

3. To evaluate the potential effectiveness of a SSD before it is installed, a diagnostic test (commonly referred to as a "communication" test) should be performed to measure the ability of a suction field and air flow to extend through the material beneath the slab. This test is commonly conducted by applying suction on a centrally located hole drilled through the concrete slab and simultaneously observing the movement of smoke downward into small holes drilled in the slab at locations separated from the central suction hole. A similar quantitative evaluation may also be performed by using a digital micromanometer or comparable instrument. Depending on test results, multiple suction points may be needed to achieve the desired effectiveness of the system.

4. Passive systems (i.e., a SSD system without a vent fan) are not as effective as active systems and their performance varies depending upon ambient temperatures and wind conditions. Therefore, active systems should be used to ensure exposures are being addressed.

5. The vent fan and discharge piping should not be located in or below a livable or occupied area of the building to avoid entry of extracted subsurface vapors into the building in the event of a fan or pipe leak.

6. To avoid entry of extracted subsurface vapors into the building, the vent pipe's exhaust should be

    i. above the eave of the roof (preferably, above the highest eave of the building at least 12 inches above the surface of the roof),

    ii. at least 10 feet above ground level,

    iii. at least 10 feet away from any opening that is less than 2 feet below the exhaust point, and

    iv. 10 feet from any adjoining or adjacent buildings, or HVAC intakes or supply registers.

7. Rain caps, if used, should be installed so as not to increase the potential for extracted subsurface vapors to enter the building.

8. To avoid accidental changes to the system that could disrupt its function, the depressurization system should be labeled clearly. An example of such labeling is shown in Figure 5.1.

9. A warning device or indicator should be installed to alert building occupants if the active system stops working properly. Examples of system failure warning devices and indicators include the following: a liquid gauge (e.g., a

manometer), a sound alarm, a light indicator, and a dial (needle display) gauge. The warning device or indicator should be placed where it can be easily heard or seen. The party installing the system should verify the warning device or indicator is working properly. Building occupants should be made aware of the warning device or indicator (what it is, where it is located, how it works, how to read/understand it, and what to do if it indicates the system is not working properly).

d. *HVAC systems* — HVAC systems should be carefully designed, installed and operated to avoid depressurization of basements and other areas in contact with the soil.

e. *Crawl space ventilation* —

1. Ventilation systems should be designed to avoid the creation of other health, safety, or environmental hazards to building occupants (e.g., backdrafting of natural draft combustion appliances).

2. Openings and cracks in floors above the crawl space that would permit conditioned air to pass into or out of the occupied spaces of the building, should be identified, closed and sealed.

f. *SVE systems designed to also mitigate exposures* —

1. The systems should be designed to avoid the creation of other health, safety, or environmental hazards to building occupants (e.g., backdrafting of natural draft combustion appliances).

2. To avoid reentry of soil vapor into the building(s), the exhaust point should be located away from the openings of buildings and HVAC air intakes. Depending upon the concentrations of volatile chemicals in subsurface vapors and the expected mass removal rate, treatment (e.g., via carbon filters) of the SVE system effluent may be appropriate to minimize outdoor air effects.

3. The SVE system's radius of influence should adequately address buildings requiring mitigation, as well as subsurface sources requiring remediation. If it does not, additional actions may be appropriate. For example, if the radius of influence does not completely extend beneath a building, a complementary air monitoring program may be appropriate to confirm that exposures are being addressed adequately while the SVE system is operating.

4.2.3 Technical guidance

To address exposures effectively in larger buildings, some of the same techniques used in residential buildings can be scaled up in size, number, or performance (e.g., adjustments in the size and air movement capacity of the vent pipe fan, or installation of multiple suction points through the slab instead of a single point). The design of the techniques may also be modified (e.g., installation of horizontal pipes beneath the building instead of a single suction point).

Detailed technical guidance on designing and installing mitigation systems in residential and non-residential buildings is provided in various documents, such as the following, released by the EPA and others:

a. References provided in ASTM's E-2121 (see Appendix E for information on how to obtain a copy) and the EPA's *Model Standards and Techniques for Control of Radon in New Residential Buildings* (Appendix F);

b. *Radon Reduction Techniques for Existing Detached Houses: Technical Guidance (Third Edition) for Active Soil Depressurization Systems*
EPA [EPA 625/R-93-011, October 1993]

This technical guidance document has been prepared to serve as a comprehensive aid in the detailed selection, design, installation, and operation of indoor radon reduction measures for existing houses based on active soil depressurization techniques. It is intended for use by radon mitigation contractors, building contractors, concerned homeowners, state and local officials and other interested persons. Copies can be ordered from the EPA's Indoor Air Quality Information Clearinghouse at 1-800-438-4318;

c. *Protecting Your Home From Radon: A Step-by-Step Manual for Radon Reduction*
Kladder *et al.*, 1993

This manual is designed to provide sufficient information to a homeowner to make many of the basic repairs that can significantly reduce radon levels in the home;

d. *Building Radon Out: A Step-by-Step Guide on How to Build Radon-Resistant Homes*
EPA [EPA 402-K-01-002, April 2001]

This fully illustrated guide contains all the information needed in one place to educate home builders about radon-resistant new construction (RRNC), including the following: basic questions and detailed answers about radon and RRNC, specific planning steps before installing a system, detailed installation instructions with helpful illustrations, tips and tricks when installing a system, marketing know-how when dealing with homebuyers, and architectural drawings. This document is available at the EPA's web site: http://www.epa.gov/iaq/radon/pubs/index.html; and

e. *Radon Prevention in the Design and Construction of Schools and Other Large Buildings*
EPA [EPA 625-R-92-016, June 1994]

It is typically easier and much less expensive to design and construct a new building with radon-resistant and/or easy-to-mitigate features, than to add these features after the building is completed and occupied. Specific guidelines on how to incorporate radon prevention features in the design and construction of schools and other large buildings are detailed in this manual. Copies can be ordered from the EPA's Indoor Air Quality Information Clearinghouse at 1-800-438-4318. This document is also available on the EPA Office of Research and Development's web site: http://www.epa.gov/ORD/NRMRL/pubs/625r92016/625r92016.htm.

4.3 Post-mitigation or confirmation testing

Once a mitigation system is installed, its effectiveness and proper installation should be confirmed. The party that installed the system should conduct post-mitigation testing and for developing a post-mitigation testing plan. Minimum objectives for post-mitigation testing associated with specific mitigation methods are provided in the following

subsections. All post-mitigation testing activities should be documented and reported to the agencies.

4.3.1 SSD systems with sealing

a. Reasonable and practical actions should be taken to identify and fix leaks. With the depressurization system operating, smoke tubes are used to check for leaks through concrete cracks, floor joints, and at the suction point. Any leaks identified should be resealed until smoke is no longer observed flowing through the opening.

b. Once a depressurization system is installed, its operation may compete with the proper venting of fireplaces, wood stoves and other combustion or vented appliances (e.g., furnaces, clothes dryers, and water heaters), resulting in the accumulation of exhaust gases in the building and the potential for carbon monoxide poisoning. Therefore, in buildings with natural draft combustion appliances, the building should be tested for backdrafting of the appliances. Backdrafting conditions should be corrected before the depressurization system is placed in operation.

c. The distance that a pressure change is induced in the sub-slab area (i.e., a pressure field extension test) should be conducted. Analogous to a communication test, this test is commonly conducted by operating the depressurization system and simultaneously observing the movement of smoke downward into small holes (e.g., 3/8 inch) drilled through the slab at sufficient locations to demonstrate that a vacuum is being created beneath the entire slab. A similar quantitative evaluation may also be performed by using a digital micromanometer or comparable instrument. If adequate depressurization is not occurring, the reason (e.g., improper fan operation) should be identified and corrected.

d. Adequate operation of the warning device or indicator should be confirmed.

e. Except as indicated below, post-mitigation indoor and outdoor air sampling should be conducted in all buildings where pre-mitigation samples were collected and in all buildings where physical data suggest possible impediments to comprehensive sub-slab communication of the depressurization system (i.e., locations with wet or dense sub-slab soils, multiple foundations and footings, minimal pressure differentials between the interior and sub-slab). Generally, indoor and outdoor air sampling locations, protocols and analytical methods should be consistent between pre-mitigation and post-mitigation sampling, where applicable. In buildings with basements, post-mitigation indoor air sampling from the basement alone (i.e., without a concurrent indoor air sample from the first floor) is recommended in most circumstances.

   Typically, post-mitigation sampling should be conducted no sooner than 30 days after installing a depressurization system. If the system is installed outside of the heating season or at the end of a season, post-mitigation air sampling may be postponed until the heating season.

   In cases of widespread mitigation due to vapor contamination and depending upon the basis of making decisions (e.g., a "blanket mitigation" approach within a specified area of documented vapor contamination [Section 3.3.1]), a representative number of buildings from an identified study area, rather than each building, may be

sampled. Prior to implementation, this type of post-mitigation sampling approach should be approved by State agency personnel.

In newly constructed buildings, a site-specific and building-specific indoor air sampling plan is recommended due to potential interferences caused by the off-gassing of volatile chemicals in new building materials (e.g., paints, carpets, furniture, etc. [Section 1.4]). In these situations, if indoor air sampling is appropriate samples should be

i. collected while the system is operational but before potentially interfering factors are brought into the building,

ii. analyzed for a targeted list of volatile chemicals based on previous environmental sampling (e.g., groundwater, soil, soil vapor, etc.), and/or

iii. collected while the system is operational but after potentially interfering factors have had an opportunity to off-gas.

If post-mitigation sampling results do not indicate a significant decrease in the concentrations of volatile chemicals previously believed to be present in the indoor air due to soil vapor intrusion, the reason (e.g., indoor or outdoor sources, improper operation of the mitigation system, etc.) should be identified and corrected as appropriate.

4.3.2 SMD systems with soil vapor retarder

a. Reasonable and practical actions should be taken to identify and fix leaks. With the depressurization system operating, smoke tubes are used to check for leaks in the membrane at seams, edge seals and at locations where the sheet was sealed around obstructions. Any leaks identified should be resealed until smoke is no longer observed flowing through the opening.

b. Backdrafting conditions should be evaluated and corrected [Section 4.3.1].

c. Adequate operation of the warning device or indicator should be confirmed.

d. Post-mitigation indoor and outdoor air testing should be conducted in buildings where pre-mitigation samples were collected [as discussed in Section 4.3.1].

4.3.3 HVAC modifications

a. Check the building for positive pressure conditions (e.g., verify a pressure controller is maintaining the desired pressure differential and/or measure the pressure differential between the sub-slab and indoor air by using field instruments).

b. Backdrafting conditions should be evaluated and corrected [Section 4.3.1].

c. Adequate operation of the warning device or indicator, if applicable, should be confirmed.

d. Post-mitigation indoor and outdoor air testing should be conducted in buildings where pre-mitigation samples were collected [Section 4.3.1].

4.3.4 Crawl space ventilation and sealing

a. Reasonable and practical actions should be taken to identify and fix leaks. With the ventilation system operating, smoke tubes are used to check for leaks in openings and cracks in floors above the crawl space that were sealed during installation of the system. Any leaks identified should be resealed until smoke is no longer observed flowing through the opening.

b. Backdrafting conditions should be evaluated and corrected [Section 4.3.1].

c. Adequate operation of the warning device or indicator, if applicable, should be confirmed.

d. Post-mitigation indoor and outdoor air testing should be conducted in buildings where pre-mitigation samples were collected [as discussed in Section 4.3.1].

4.3.5 SVE systems designed to also mitigate exposures

a. Backdrafting conditions should be evaluated and corrected [Section 4.3.1].

b. The distance that a pressure change is induced in the sub-slab area should be conducted. This may be done by operating the SVE system and simultaneously observing the movement of smoke downward into small holes (e.g., 3/8 inch) drilled through the building's slab at sufficient locations to demonstrate that a vacuum is being created beneath the entire slab.

c. Adequate operation of the warning device or indicator, if applicable, should be confirmed.

d. Post-mitigation indoor and outdoor air testing should be conducted in buildings where pre-mitigation samples were collected [Section 4.3.1].

4.4 Operation, maintenance and monitoring of mitigation systems

When mitigation systems are implemented at a site, the operation, maintenance and monitoring (OM&M) protocols for the systems should be included in a site-specific site management plan (formerly referred to as operation, maintenance and monitoring plan). The party that installed the system should conduct OM&M activities and should develop the site management plan. Recommendations for minimum OM&M activities associated with specific mitigation methods are provided in the following subsections. Also included is a discussion of non-routine maintenance. All routine and non-routine OM&M activities should be documented and reported to the agencies.

4.4.1 SSD and SMD systems

Routine maintenance should commence within 18 months after the system becomes operational, and should occur every 12 to 18 months thereafter. Based upon a demonstration of the system's reliability, the State recommends that, if a different frequency is desired, a petition describing the alternative frequency and the reasons that frequency is preferred be submitted to the State. Any comments the State may have on the petition should be considered before the frequency is altered.

During routine maintenance, the following activities (at a minimum) should be conducted:

a. a visual inspection of the complete system (e.g., vent fan, piping, warning device or indicator, labeling on systems, soil vapor retarder integrity, etc.),
b. identification and repair of leaks [Sections 4.3.1 and 4.3.2], and
c. inspection of the exhaust or discharge point to verify no air intakes have been located nearby.

As appropriate preventative maintenance (e.g., replacing vent fans), repairs and/or adjustments should be made to the system to ensure its continued effectiveness at mitigating exposures related to soil vapor intrusion. The need for preventative maintenance will depend upon the life expectancy and warranty for the specific part, as well as visual observations over time. The need for repairs and/or adjustments will depend upon the results of a specific activity compared to that obtained when system operations were initiated.

If significant changes are made to the system or when the system's performance is unacceptable, the system may need to be redesigned and restarted. Many, if not all, of the post-mitigation testing activities, as described in Sections 4.3.1 and/or 4.3, may be appropriate. The extent of such activities will primarily depend upon the reason for the changes and the documentation of sub-slab depressurization.

Generally, air monitoring is not recommended if the system has been installed properly and is maintaining a vacuum beneath the entire slab.

In addition to the routine OM&M activities described here, the building's owner and tenants are given information packages that explains the system's operation, maintenance and monitoring [Section 5.6]. Therefore, at any time during the system's operation, the building's owner or tenants may check that the system is operating properly.

4.4.2 Other mitigation systems

For other mitigation systems (e.g., HVAC modifications, crawl space ventilation, etc.), routine maintenance activities are generally comparable to post-mitigation testing activities [Section 4.3]. Activities typically include a visual inspection of the complete system, and identification and repair of leaks. System performance checks, such as air stream velocity measurements of ventilation systems, also should be performed.

As appropriate, preventative maintenance (e.g., replacing filters, cleaning lines, etc.), repairs and/or adjustments should be made to the system to ensure its continued effectiveness at mitigating exposures related to soil vapor intrusion. If significant changes are made to the system or when the system's performance is unacceptable, redesigning and restarting the system may be appropriate[Section 4.4.1].

Air monitoring, such as periodic sub-slab vapor, indoor air and outdoor air sampling, may be appropriate to determine whether existing building conditions are maintaining the desired mitigation endpoint and to determine whether changes are appropriate. The type and frequency of monitoring is determined based upon site-specific and building-specific conditions, taking into account applicable environmental data, building operating conditions, and the mitigation method employed.

4.4.3 Non-routine maintenance

Non-routine maintenance may also be appropriate during the operation of a mitigation system. Examples of such situations include the following:

a. the building's owners or occupants report that the warning device or indicator indicates the mitigation system is not operating properly;

b. the mitigation system becomes damaged; or

c. the building has undergone renovations that may reduce the effectiveness of the mitigation system.

Activities conducted during non-routine maintenance visits will vary depending upon the reason for the visit. In general, building-related activities may include examining the building for structural or HVAC system changes, or other changes that may affect the performance of the depressurization system (e.g., new combustion appliances, deterioration of the concrete slab, or significant changes to any of the building factors listed in Table 1.2). Depressurization system-related activities may include examining the operation of the warning device or indicator and the vent fan, or the extent of sub-slab depressurization. Repairs or adjustments should be made to the system as appropriate. If appropriate, the system should be redesigned and restarted [Section 4.4.1].

4.5 Termination of mitigation system operations

Mitigation systems should not be turned off, until the State receives, and has had the opportunity to comment on, a proposal to turn off mitigation systems. The party seeking to turn off the mitigation systems should consider any comments the State may have on the proposal, except in emergency situations. Systems should remain in place and operational until they are no longer needed to address current or potential exposures related to soil vapor intrusion. This determination should be based upon several factors, including the following:

a. subsurface sources (e.g., groundwater, soil, etc.) of volatile chemical contamination in subsurface vapors have been remediated based upon an evaluation of appropriate post-remedial sampling results;

b. residual contamination, if any, in subsurface vapors is not expected to affect indoor air quality significantly based upon soil vapor and/or sub-slab vapor sampling results;

c. residual contamination, if any, in subsurface vapors is not affecting indoor air quality when active mitigation systems are turned off based upon indoor air, outdoor air and sub-slab vapor sampling results at a representative number of buildings; and

d. there is no "rebound" effect for which additional mitigation efforts would be appropriate observed when the mitigation system is turned off for prolonged periods of time. This determination should be based upon indoor air, outdoor air and/or sub-slab vapor sampling from the building over a time period, determined by site-specific conditions.

Given the prevalence of radon throughout the State of New York, consideration should be given to leaving the system in place and operating to address exposures related to radon intrusion after concurrence is reached that the system is no longer needed to mitigate exposures related to soil vapor intrusion. This action should be done only with permission of the property owner and after the property owner is aware of their responsibilities in

operating, monitoring and maintaining the system for this specific purpose. If the property owner declines the offer, the system should be shut down and, if requested, removed in a timely manner.

4.6 Annual certification and notification recommendations

Mitigation systems are considered engineering controls, defined as any physical barrier or method employed to

1. actively or passively contain, stabilize, or monitor hazardous waste or petroleum,
2. restrict the movement of hazardous waste or petroleum to ensure the long-term effectiveness of remedial actions, or
3. eliminate potential exposure pathways to hazardous waste or petroleum.

Therefore, depending upon the remedial program, submission of an annual certification to the State may be required. This certification must be prepared and submitted by a professional engineer or environmental professional and affirm that the engineering controls are in place, are performing properly and remain effective. This requirement of certification remains in effect until the State provides notification, in writing, that this certification is no longer needed.

If a property owner declines a mitigation system, the party responsible for arranging the design and installation of the system should renew the offer on an annual basis, unless they demonstrate environmental conditions have changed such that a system is no longer needed.

Section 5: Community Outreach

While community outreach is an essential component of the investigation and remediation of any site, it is particularly critical when evaluating soil vapor intrusion at a site due to the following:

a. a heightened awareness by environmental professionals and the general public (both nationally and state-wide) for the importance of soil vapor intrusion;

b. the relatively complicated nature of the exposure pathway (e.g., chemicals in groundwater or soil ending up in the indoor air of buildings versus contaminated groundwater entering the house through the use of a private well);

c. the unknowns associated with the evolving science of investigating, evaluating, and mitigating exposures related to soil vapor intrusion; and

d. the relatively complicated nature of mitigating the exposure pathway (e.g., the design, installation and operation of a sub-slab depressurization system in a home versus an immediate switch from using private well water to using bottled water).

When people have been or may be exposed to contamination, providing them with accurate and timely information about those exposures is extremely important. This information should include details about the types of chemicals, the levels of exposure, and possible health effects from those exposures. In addition, information should include details about the planning and progress of the investigation and remediation efforts. Techniques commonly used to inform the community about soil vapor intrusion issues are described in this section. The type, or types, of techniques selected for a site will vary depending upon the community's needs, site-specific conditions and remedial program-specific requirements.

5.1 Site contact list

A contact list contains names, addresses and telephone numbers of individuals and organizations with interest or involvement in a site. They may be affected by or interested in the site, or have information that staff needs to make effective remedial decisions. Contact lists typically include residents near the site, elected officials, appropriate federal, state, and local government contacts, local media, organized environmental groups and the responsible party, as well as local businesses, civic and recreational groups, religious facilities, school district officials, and all staff (NYSDEC, NYSDOH, county health department, EPA, etc.) involved in the site. The checklist provided in Appendix G.1 will help to identify who should be included in a particular site's contact list.

With respect to soil vapor intrusion, the site contact list is often used to

a. send a fact sheet announcing a proposed investigation in the area, a major project decision or proposal, the project's status or progress, a public meeting or availability session, or the availability of documents in the repositories;

b. contact building owners and tenants to arrange sampling dates and times and to transmit sampling results (in written form and/or verbally); and

c. provide community members with verbal updates on the project's status or progress.

The member of the project team (defined as the NYSDEC, NYSDOH, responsible party, etc.) that develops and maintains the site contact list is determined on a site-specific and/or

program-specific basis. Development and revision of the contact list are ongoing activities throughout the site's investigation and remediation. Guidance on how to create a site contact list is provided in Appendix G.1.

5.2 Project staff contact sheet

As implied by the name, this is a summary of the contact information for staff working on the site that can be handed out to the community. Often included on the sheet are the name, title, affiliation, role or area of expertise, address, telephone number, email address, facsimile number for each staff member. The contact sheet provides the community with a quick reference on whom to call with questions, comments or concerns about the site. Project staff may also use the site contact sheet to direct inquiries to the most appropriate person. This is particularly useful when there are many agencies working on the site and many issues, such as site investigation, health studies, medical outreach, etc., being addressed.

The site contact sheet should be handed out at public meetings or availability sessions, when door-to-door visits and sampling are conducted, and in conjunction with other appropriate outreach activities. The sheet should be developed early on in the process and kept up-to-date. The member of the project team that develops and maintains the staff contact sheet is determined on a site-specific and/or program-specific basis.

5.3 Fact sheets

A fact sheet is a written summary of important information about a site. It presents information in clear and concise terms for the community. Fact sheets aid consistent distribution of information and citizens' understanding of significant issues associated with site-related activities. With respect to soil vapor intrusion, fact sheets are often used to

a. announce a proposed soil vapor intrusion investigation in the area, either as a stand-alone activity or in conjunction with the site's overall investigation;

b. summarize the results of an investigation and the anticipated next steps in the process;

c. invite the public to a meeting or availability session to discuss the proposed investigation, the results of a recently completed investigation, the anticipated next steps, etc.; and

d. provide additional information on topics associated with soil vapor intrusion, such as specific air guidelines for volatile chemicals.

The member of the project team that plans, develops and distributes the fact sheet is determined on a site-specific and/or program-specific basis. Factors to consider when designating the lead include the site's remedial program, the expected content of the fact sheet, and the relationship of various team members with the community. For example, if the community strongly distrusts the responsible party and wants to know how the state is determining that their actions are appropriate, the state should be the lead. A combination of team members may also be suitable.

All team members should be included in reviewing and finalizing the fact sheet. Once the state approves the fact sheet, it may be released to the public. Timely distribution of the fact sheet is important. Sufficient time should be allowed in the development and review

schedule to ensure that the fact sheet is distributed — *and that it is received* — before the critical activity takes place. Specific timeframes for release include the following:

a. 2 weeks prior to a public meeting or availability session, or commencement of field activities;

b. within 24 hours of receiving a specific request for an available fact sheet from the community (e.g., members of the community that did not receive a copy of the fact sheet in the mail);

c. if applicable, before a comment period begins (otherwise a 30-day comment period becomes, in reality, a 25-day comment period); and

d. if appropriate, concurrently with letters to the community explaining sampling results.

Copies of fact sheets commonly used to supplement discussions related to soil vapor intrusion are provided in Appendix H. They are also available from the NYSDOH's soil vapor intrusion web page: http://www.health.state.ny.us/environmental/indoors/vapor_intrusion/. Additional guidance on how to plan, develop and distribute fact sheets is provided in Appendix G.2.

5.4 Public gatherings

The following are several types of public gatherings where project staff can meet with the community:

a. Traditional Public Meetings: Project staff generally present information and answer questions. Citizens are encouraged to ask questions and provide comments;

b. Public Availability Sessions: The session is held in a casual setting, without a formal agenda and presentation. Staff generally conduct an availability session about a specific aspect of a site, which it publicizes ahead of time. The format promotes detailed individual or small group discussion between staff and the public. An availability session may be targeted to a specific subgroup of the overall community. For example, a session may be held where project staff meet with building owners and tenants to discuss their individual sampling results;

c. Public Forum: The forum is held in a casual setting, without a formal presentation. Typically, the format is one of "question and answer" — a panel of project staff (or, if applicable, outside experts) answer questions asked by community members in an open discussion; and

d. Other: Project staff may be invited to give presentations or to make themselves available for questions at community group meetings, such as community or neighborhood board meetings, school board meetings, etc.

If appropriate, a combination of the above may be used. The type, or combination of types, of gathering (if any) selected should be decided based on site-specific, program requirements and community-specific conditions, such as the following:

a. Is the investigation limited to on-site buildings, to a localized area of off-site buildings, or to the off-site neighborhood surrounding the site?;

b. Is the soil vapor investigation being performed as part of ongoing site investigation activities (and consequently ongoing outreach activities), or is this issue being revisited at a site where remediation was considered "complete?";

c. What type of outreach has the community favored in the past?;

d. What are the objectives of the meeting? Can one meeting type accomplish each of the objectives or are different meeting types needed on successive days (e.g., public meeting followed by an availability session)?; and

e. Who is the desired audience? Should the meeting be held in the afternoon to accommodate an elderly population and repeated in the evening for people who work during normal business hours?

The member of the project team that coordinates and implements the gathering is determined on a site-specific and/or program-specific basis. Factors to consider when designating the lead include the site's remedial program, the expected subject of the meeting, and the relationship of various team members with the community. A combination of team members may also be appropriate.

Additional guidance on how to plan and conduct a public meeting and an availability session is provided in Appendices G.3 and G.4.

5.5 Letters transmitting results

When indoor air and/or sub-slab vapor samples are collected from within or beneath a building, a letter providing the sampling results and the conclusions drawn from the data evaluation should be transmitted to the building's owner. If the building is a rental property, the transmittal letter should be sent to the tenants residing in the areas where the samples were collected and a copy to the property owner/landlord. In some cases where responsible parties are carrying out indoor air sampling, access agreements are commonly executed between such a party and the property owner. Consequently, the transmittal letter may be sent to the property owner, and where feasible by prior arrangement with the property owner and/or tenant, with a copy to the tenant.

A transmittal letter should include the following (as applicable):

a. the address of the building sampled;

b. the date samples were collected;

c. the type of samples collected (e.g., sub-slab vapor, indoor air and outdoor air);

d. indoor air sampling locations (e.g., basement, crawl space, first floor living room, etc.)

e. who collected the samples (e.g., the state, or [Consultant Name] on behalf of [Responsible Party name], etc.);

f. why samples were collected (e.g., to evaluate the potential for exposures associated with soil vapor intrusion);

g. the site name and number (usually included in the subject line);

h. the compound(s) or group of compounds of concern (e.g., trichloroethene or volatile organic compounds);

i. an overview of the sampling results (e.g., a table summarizing compounds detected in each sample and/or a figure illustrating sampling locations and corresponding results);

j. copies of the laboratory sheets for each sample collected and the completed building questionnaire/inventory;

k. a statement of the conclusions drawn and the next steps (e.g., soil vapor intrusion appears to be the likely source of volatile chemicals in your indoor air and we would like to install a sub-slab depressurization system to minimize exposures);

l. if applicable, what information should be shared with employees and/or patrons of the facility (e.g., the transmittal letter and enclosed fact sheets, a situation-specific fact sheet and cover memorandum, etc.);

m. contact information for project staff; and

n. fact sheets that supplement information provided in the letter.

The member of the project team that transmits the letter is typically the member that conducted the investigation. A representative of each member should be copied on each transmittal. For example, for investigations conducted by the state, letters are transmitted by the NYSDOH; state and local agencies, as well as a representative for the responsible party (or other non-agency project staff), should be copied. For investigations conducted by the responsible party, the responsible party should transmit letters that have been reviewed and approved by the state, and copy state and local agency representatives.

The level of detail provided in the letter will depend upon who transmits the letter. For example, letters written by the NYSDOH may recommend actions to reduce exposures to indoor sources (i.e., not site-related sources) of volatile chemicals, or address expected risks associated with an identified exposure. Letters transmitted by a responsible party generally focus on site-related contamination and their identified next steps. These letters generally refer the recipients to the state for questions regarding non-site-related compounds and health concerns. For additional guidance on the content of the transmittal letters, contact the NYSDOH's Bureau of Environmental Exposure Investigation at 1-800-458-1158, extension 27850.

Timely distribution of the transmittal letter is important. Generally, final (i.e., verified) sampling results from the laboratory are available 6 to 8 weeks after the samples are submitted. As soon as they are available, final results should be forwarded to the team member that is transmitting them. Sufficient time should be allowed in the development and review schedule to ensure that the letter is transmitted within 2 weeks after final results are available.

If there is significant community interest in the sampling results, reasonable attempts should be made to inform the building owners and tenants of their results verbally in addition to sending a transmittal letter. Other interested community members, such as residents, press and elected officials, may be given an overview of the investigation results and the conclusions drawn *after* each building owner and tenant has been notified.

5.6 Soil vapor intrusion mitigation information

Once a mitigation system (e.g., sub-slab depressurization system) is installed in a building, an information package should be given to the building's owner and tenants, if applicable, to facilitate their understanding of the system's operation, maintenance and monitoring. This package should include the following:

a. a description of the mitigation system installed and its basic operating principles;

b. how the owner or tenant can check that the system is operating properly;

c. how the system will be maintained and monitored and by whom;

d. a list of appropriate actions for the owner or tenant to take if the system's warning device or indicator (e.g., pressure gauge, alarm, etc.) indicates system degradation or failure; and

e. contact information (e.g., names, telephone numbers, etc.) if the owner or tenant has questions, comments or concerns.

The building's owner should also receive the following information:

a. any building permits required by local codes;

b. copies of contracts and warranties; and

c. a description of the proper operating procedures of any mechanical or electrical system installed, including manufacturer's operation and maintenance instructions and warranties.

Wherever possible, illustrations should be provided. For example, pictures of a manometer under normal operating conditions [Figure 5.1], as well as drawings or schematics showing the system at work [Figure 5.2].

The member of the project team who provides this information is the member who installed the mitigation system.



Figure 5.1
Manometer indicating the SSD system is operating properly.

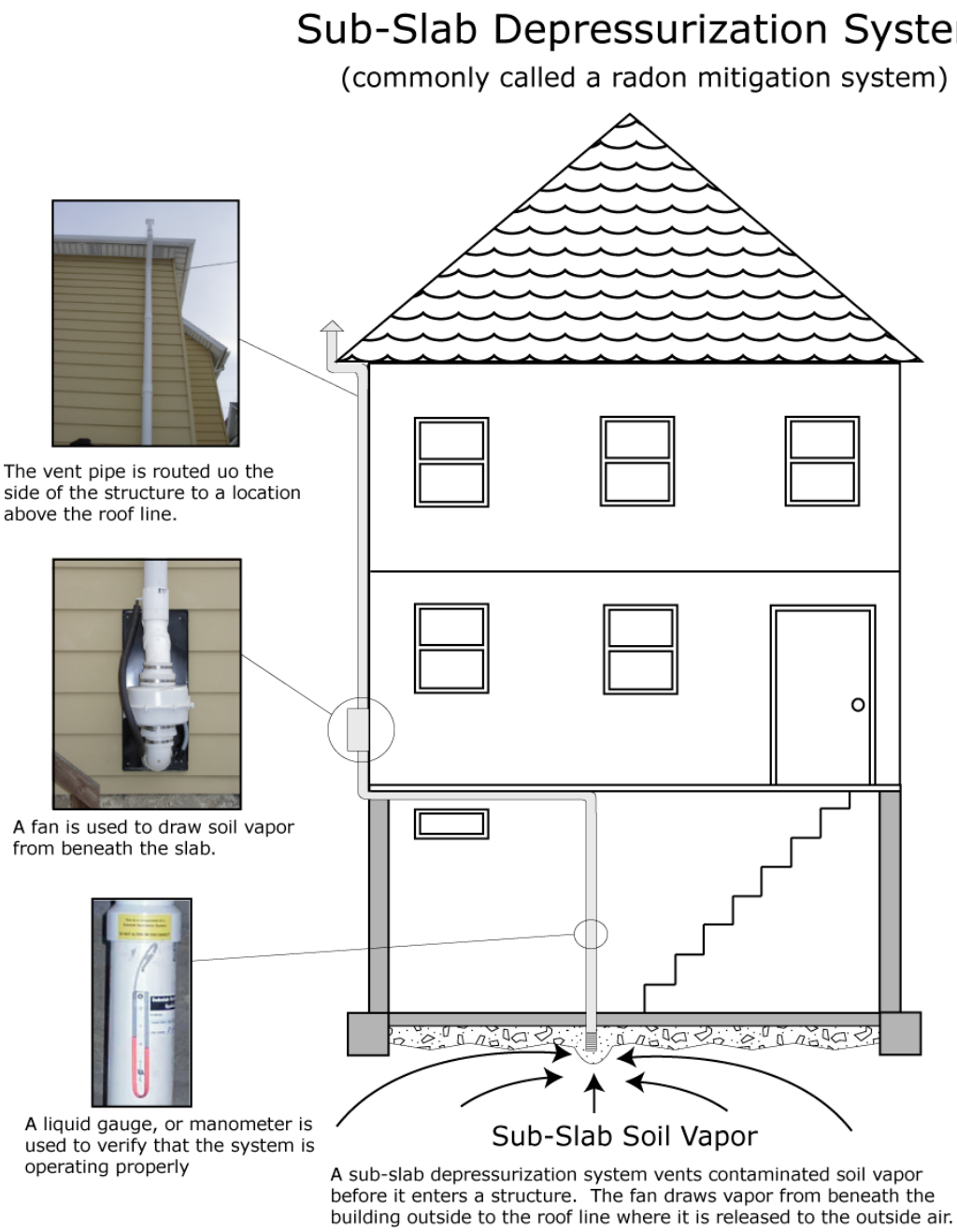


Figure 5.2
Example of an illustration showing how a SSD system works.

5.7 Toll-free "800" numbers

Toll-free information numbers provide quick, easy access for people who have questions, comments or concerns about a site. At a minimum, the NYSDOH site project manager's name and the following "800" number should be shared with the community in fact sheets and transmittal letters, at public gatherings, when samples are collected, and with other outreach techniques for their use if they have health-related questions, concerns or comments related to soil vapor intrusion at the site.

NYSDOH
Center for Environmental Health
Bureau of Environmental Exposure Investigation
Toll-free Information Line

1-800-458-1158, ext. 27850

Note: The "800" number is an *information* line — not a "*hotline*" — because callers may not receive immediate response, such as on nights or weekends.

Similarly, applicable toll-free numbers setup and maintained by other project team members should also be shared with the community whenever appropriate. Additional information on the use of toll-free "800" numbers as an outreach tool is provided in Appendix G.5.

5.8 Door-to-door visits

Door-to-door visits involve gathering or distributing site information by meeting individuals at their residences or businesses. Typically, this outreach technique is used to supplement other communication, such as telephone calls and letters. With respect to soil vapor intrusion, project staff may visit residents near a site to provide information, answer questions, or obtain permission for activities on private properties. All team members should be aware of the specifics of the door-to-door visits (e.g., who will be conducting the visits, the reason, the dates, etc.).

Additional information on conducting door-to-door visits is provided in Appendix G.6.

5.9 Document repositories

A document repository is a collection of documents and other information developed during the investigation and remediation of a site. It is located in a convenient, public facility, such as a library, so that affected and interested members of the public can easily access and review important information about the site. A repository is maintained through the site's operation and maintenance phase, or until its release from the applicable remedial program.

A site document repository helps the public review

a. documents about which the state is seeking public comment;

b. studies, reports and other information; and

c. complete versions of documents summarized in fact sheets, meeting presentations or media releases (summaries should note the locations of local repositories where the complete documents are available).

The member of the project team that establishes and maintains the document repository is determined on a site-specific and/or program-specific basis. Additional guidance on how to establish and maintain a document repository is provided in Appendix G.7.

5.10 Medical community outreach

Outreach to the medical community is an activity or combination of activities undertaken to assist local health care providers in caring for people who have concerns about site-specific environmental exposures. The goal of this type of outreach is to assist the individual provider by giving him/her much of the site-specific information related to the contaminants and to provide information about the site itself. This type of outreach is undertaken whenever the NYSDOH and/or other health agencies determine that the site-specific contaminants may be unfamiliar to the local medical community. Conversely, this outreach can be undertaken when community members express the concern that their health care providers may be unfamiliar with potential adverse health effects related to contaminants at the site.

The targeted audience for this type of outreach consists of specific groups of health care providers most likely to treat people with concerns about potential environmental exposures. Some examples of targeted groups of specialists could include any combination of the following: Family Practice, Internal Medicine, Preventive Medicine, Oncology, Neurology, Allergy, Pediatrics, Obstetrics, Dermatology and Emergency Medicine. Likewise, materials can be sent to medical and nursing schools, residency programs, and medical libraries if they are located nearby. Developing the targeted list of health-care providers is a cooperative effort between local and state departments of health, with input from the community as well.

The NYSDOH, in partnership with the Agency for Toxic Substance and Disease Registry (ATSDR) and the local health department, can conduct these activities, which could include any one or a combination of the following:

a. announcements made at public meetings that the NYSDOH Center for Environmental Health will mail out information packets to individual physicians at the request of any concerned citizen;

b. an article placed in a local newspaper, or, if applicable, in a newsletter periodically sent to residents, stating that the NYSDOH Center for Environmental Health will mail out packets to individual physicians at the request of any concerned citizen. The NYSDOH "800" number and two NYSDOH contact names would be given;

c. an article submitted to the newsletter of the local county medical society, stating that the NYSDOH and the ATSDR have information to help providers with questions about site-related contamination in the area of the site. The NYSDOH "800" number and two NYSDOH contact names would be given; and

d. materials sent to medical and nursing schools, residency programs, and medical libraries if they are located nearby.

Local and state departments of health, and ATSDR, have developed appropriate outreach materials. The information packets should contain a letter to the physician, site-specific fact sheets, brochures, and booklets about potential exposures and about the contaminants in the area of the site. As an example, here is a list of fact sheets and pamphlets that an information packet for a site with PCE and TCE as contaminants of concern might contain:

a. a letter of explanation to the provider, including the NYSDOH "800" number to call for access to more information, as well as two NYSDOH contacts with whom to speak initially;

b. a site-specific fact sheet written for the community, explaining various site-related issues;

c. a compact disc of ATSDR case studies in environmental medicine (CSEMs), with opportunities for earning many free continuing medical education (CME) credits through the Centers for Disease Control and Prevention;

d. a hard copy of both the "Trichloroethylene (TCE) Toxicity" and "Taking an Environmental Exposure History" case studies;

e. two small "quick reference guides" produced by ATSDR about evaluating environmental exposures and doing an exposure history;

f. a NYSDOH fact sheet on Trichloroethene (TCE) in indoor and outdoor air;

g. an ATSDR fact sheet on Trichloroethylene (TCE);

h. a NYSDOH fact sheet on Tetrachloroethene (PERC) in indoor and outdoor air; and

i. an ATSDR fact sheet on Tetrachloroethylene (PERC).

For additional information on this outreach tool, please contact the NYSDOH Center for Environmental Health's Outreach and Education Unit at 1-800-458-1158, extension 27530.

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Attachment C

Superfund Program Representative Sampling Guidances

OSWER Directive 9360.4-10
EPA 540/R-95/141
PB96-963207
December 1995

SUPERFUND PROGRAM

REPRESENTATIVE SAMPLING GUIDANCE

VOLUME 1: SOIL

Interim Final

Environmental Response Team

Office of Emergency and Remedial Response
Office of Solid Waste and Emergency Response
U.S. Environmental Protection Agency
Washington, DC 20460

Notice

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

The policies and procedures established in this document are intended solely for the guidance of government personnel, for use in the Superfund Program. They are not intended, and cannot be relied upon, to create any rights, substantive or procedural, enforceable by any party in litigation with the United States. The Agency reserves the right to act at variance with these policies and procedures and to change them at any time without public notice.

For more information on Soil Sampling and Surface Geophysics procedures, refer to the *Compendium of ERT Soil Sampling and Surface Geophysics Procedures*, OSWER directive 9360.4-02, EPA/540/P-91/006. Topics covered in this compendium include Sampling Equipment Decontamination, Soil Sampling, Soil Gas Sampling, and General Surface Geophysics. The compendium describes procedures for collecting representative soil samples and provides a quick means of waste site evaluation. It also addresses the general procedures used to acquire surface geophysical data.

Questions, comments, and recommendations are welcomed regarding the *Superfund Program Representative Sampling Guidance, Volume 1 -- Soil*. Send remarks to:

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1.0 INTRODUCTION

1.1 OBJECTIVE AND SCOPE

This is the first volume in a series of guidance documents that assist Superfund Program Site Managers, On-Scene Coordinators (OSCs), Remedial Project Managers (RPMs), and other field staff in obtaining representative samples at Superfund sites. The objective of representative sampling is to ensure that a sample or a group of samples accurately characterizes site conditions. This document specifically addresses representative sampling for soil. The information presented here is valid throughout the Superfund program, but focuses on the objectives of early action activities and emergency responses. Topics covered in the document include: assessing available information; selecting an appropriate sampling approach; selecting and utilizing geophysical, analytical screening, and sampling equipment; utilizing proper sample preparation techniques; incorporating suitable types and numbers of Quality Assurance/Quality Control (QA/QC) samples; and interpreting and presenting the analytical and geophysical data.

In the Superfund program, representative sample data collected during emergency responses or early actions may form the basis of remedial response. Longer, more complex responses require a variety of sampling objectives, including identifying threat, delineating sources and extent of contamination, and confirming the achievement of clean-up standards. Many important and potentially costly decisions are based on the sampling data, making it very important that OSCs and field personnel understand how accurately the sampling data characterize the actual site conditions. In keeping with this strategy, this document emphasizes analytical screening and geophysical techniques as cost effective approaches to characterize the site and to select sampling locations.

1.2 Conceptual Site Model

A conceptual site model is a useful tool for selecting sampling locations. It helps ensure that sources, pathways, and receptors throughout the site have been considered before sampling locations are chosen. The conceptual model assists the Site Manager in evaluating the interaction of different site features. Risk assessors use conceptual models to help plan for risk assessment activities. Frequently, a conceptual model is created as a site map (see Figure 1) or it may be developed as a flow diagram which describes potential migration of contaminants to site receptors (see Appendix A).

A conceptual model follows contaminants from their sources, to pathways (e.g., air, surface water), and eventually to the assessment endpoints. Consider the following when creating a conceptual model:

- The state(s) of each contaminant and its potential mobility
- Site topographical features
- Meteorological conditions (e.g., wind direction/speed, average precipitation, temperature, humidity)
- Human/wildlife activities on or near the site

The conceptual site model on the next page is an example created for this document. The model assists in identifying the following site characteristics:

Potential Sources:

Site (waste pile); drum dump; agricultural activities

Potential Exposure Pathway (Soil):

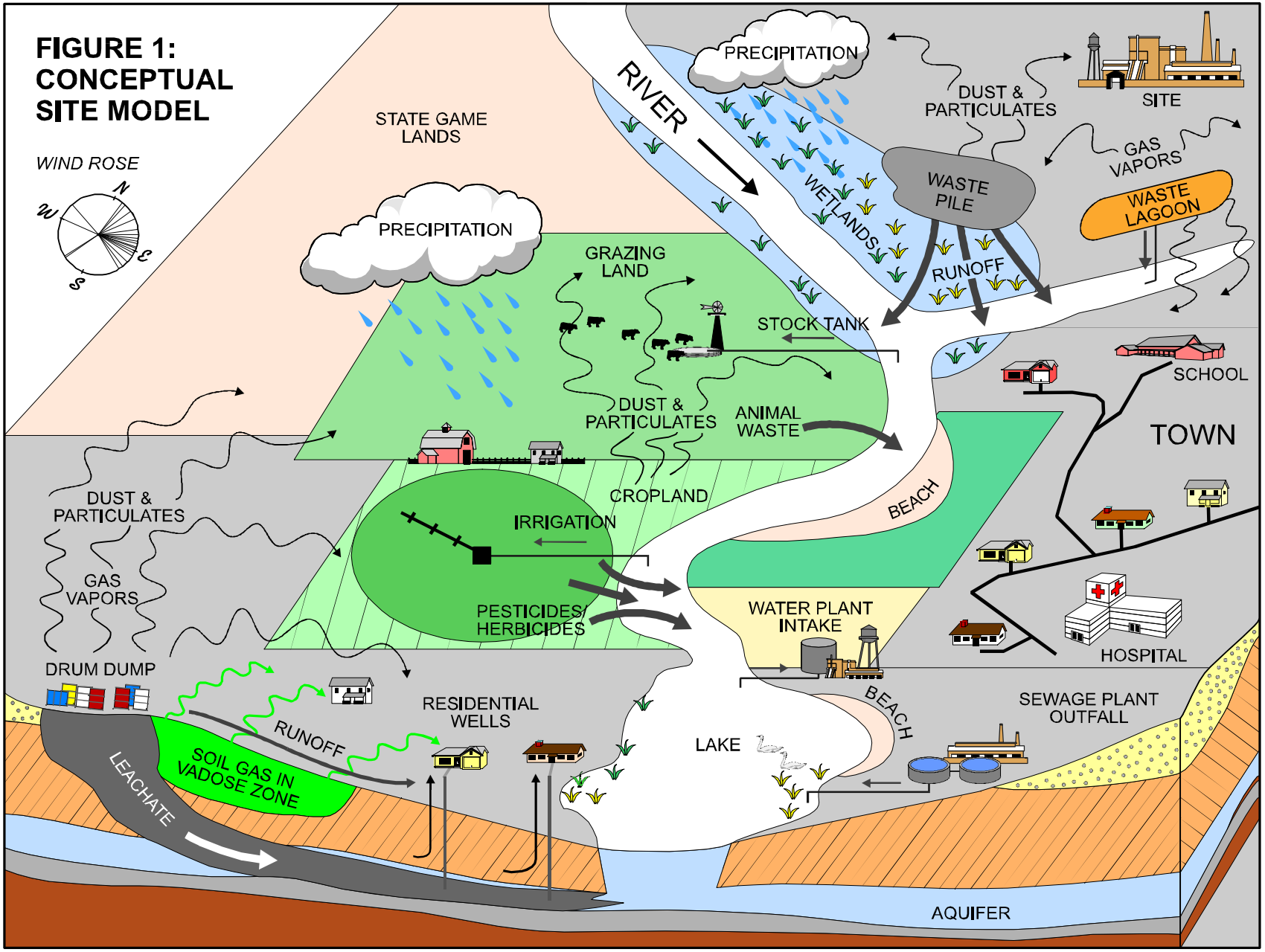
Leachate from the waste pile or drum dump; contaminated soil from direct contact with the waste pile or drum dump; agricultural activities such as pesticide application onto cropland

NOTE: Soil is described as an *exposure* pathway rather than a *migration* pathway because, unlike other media (e.g., air), contact between contaminated soil and a receptor is initiated by the receptor.

Potential Exposure Routes:

Ingestion -- Soil particles from the waste pile, drum dump or area of agricultural activity

Absorption/direct contact -- Soil near the waste pile, drum dump or area of agricultural activity


FIGURE 1:
CONCEPTUAL
SITE MODEL
WIND ROSE
N
E
S
W
STATE GAME
LANDS
RIVER
PRECIPITATION
DUST &
PARTICULATES
SITE
GAS
VAPORS
WASTE
PILE
WASTE
LAGOON
WETLANDS
RUNOFF
PRECIPITATION
GRAZING
LAND
STOCK TANK
SCHOOL
TOWN
DUST &
PARTICULATES
ANIMAL
WASTE
CROPLAND
BEACH
DUST &
PARTICULATES
IRRIGATION
GAS
VAPORS
PESTICIDES/
HERBICIDES
WATER PLANT
INTAKE
HOSPITAL
DRUM DUMP
RESIDENTIAL
WELLS
BEACH
SEWAGE PLANT
OUTFALL
RUNOFF
SOIL GAS IN
VADOSE ZONE
LEACHATE
LAKE
AQUIFER

Potential Receptors of Concern (and associated potential exposure routes):

Human Population

Residents/Trespassers:

Leachate into soil from the drum dump; direct contact with soil contaminated by pesticides or other agricultural activities in the cropland

Workers/Trespassers:

Leachate into soil from the waste pile; contaminated soil associated with the waste pile or agricultural activities in the cropland

Biota

Endangered/threatened species or human food chain organisms, if suspected to be in contact with an area of potentially contaminated soil

Preliminary site information may provide the identification of the contaminant(s) of concern and the level(s) of the contamination. A sampling plan should be developed based upon the selected receptors of concern and the suspected sources and pathways. The model may assist in the selection of on-site and off-site sampling locations.

1.3 REPRESENTATIVE SAMPLING OBJECTIVES

Representative sampling applies to all phases of a Superfund response action. Representative sampling objectives for soil include:

1. Establishing threat to public health or welfare or to the environment;

2. Locating and identifying potential sources of contamination;

3. Defining the extent of contamination;

4. Determining treatment and disposal options; and

5. Documenting the attainment of clean-up goals.

These objectives are discussed in detail in Section 2.5.

1.4 REPRESENTATIVE SAMPLING

Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration throughout a site.

This document concentrates on the variables that are introduced in the field -- namely, those that relate to the site-specific conditions, the sampling design approach, and the techniques for collection and preparation of samples. The following variables affect the representativeness of samples and subsequent measurements:

- Geological variability -- Regional and local variability in the mineralogy of rocks and soils, the buffering capacity of soils, lithologic permeability, and in the sorptive capacity of the vadose zone.

- Contaminant concentration variability -- Variations in the contaminant concentrations throughout the site.

- Collection and preparation variability -- Deviations in analytical results attributable to bias introduced during sample collection, preparation, and transportation (for analysis).

- Analytical variability -- Deviations in analytical results attributable to the manner in which the sample was stored, prepared, and analyzed by the on-site or off-site laboratory. Although analytical variability cannot be corrected through representative sampling, it can falsely lead to the conclusion that error is due to sample collection and handling procedures.

1.5 EXAMPLE SITE



An example site, presented at the end of each chapter, illustrates the development of a representative soil sampling plan that meets Superfund Program objectives for early actions or emergency responses.

2.0 SAMPLING DESIGN

2.1 INTRODUCTION

The following procedures are recommended for developing a sound sampling design. Many steps can be performed simultaneously, and the sequence is not rigid.

- Review existing historical site information;
- Perform a site reconnaissance;
- Evaluate potential migration pathways and receptors;
- Determine the sampling objectives;
- Establish the data quality objectives;
- Utilize screening techniques;
- Select parameters for which to be analyzed;
- Select an appropriate sampling approach; and
- Determine the locations to be sampled.

Real-time analytical screening techniques can be used throughout the removal action. The results can be used to modify the site sampling plan as the extent of contamination becomes known.

2.2 HISTORICAL DATA REVIEW

Unless the site is considered a classic emergency, every effort should be made to first thoroughly review relevant site information. An historical data review examines past and present site operations and disposal practices, providing an overview of known and potential site contamination and other site hazards. Sources of information include federal, state and local officials and files (e.g., site inspection reports and legal actions), deed or title records, current and former facility employees, potentially responsible parties, local residents, and facility records or files. For any previous sampling efforts, obtain information regarding sample locations (on maps, if possible), matrices, methods of collection and analysis, and relevant contaminant concentrations. Assess the reliability and usefulness of existing analytical data. Even data which are not substantiated by documentation or QA/QC controls may still be useful.

Collect information that describes any specific chemical processes used on site, as well as descriptions of raw materials used, products and wastes, and waste storage and disposal practices. Whenever possible, obtain site maps, facility blueprints, and historical aerial photographs, detailing past and present storage, process, and waste disposal locations. The local Agricultural Extension Agent, a Soil Conservation Service (SCS) representative, has information on soil types and drainage patterns. County property and tax records, and United States Geological Survey (USGS) topographic maps are also useful sources of site and regional information.

2.3 SITE RECONNAISSANCE

A site reconnaissance, conducted either prior to or in conjunction with sampling, is invaluable to assess site conditions, to evaluate areas of potential contamination, to evaluate potential hazards associated with sampling, and to develop a sampling plan. During the reconnaissance, fill data gaps left from the historical review by:

- Interviewing local residents, and present or past employees about site-related activities;
- Researching facility files or records (where records are made accessible by owner/operator);
- Performing a site entry, utilizing appropriate personal protective equipment and instrumentation. Observe and photo-document the site; note site access routes; map process and waste disposal areas such as landfills, lagoons, and effluent pipes; inventory site wastes; and map potential transport routes such as ponds, streams, and irrigation ditches. Note topographic and structural features, dead animals and dead or stressed vegetation, potential safety hazards, and visible label information from drums, tanks, or other containers found on the site.

2.4 MIGRATION PATHWAYS AND RECEPTORS

The historical review and site visit are the initial steps in defining the source areas of contamination which could pose a threat to human health and the environment. This section addresses how to delineate the spread of contamination away from the source areas. Included are pollutant migration pathways and

the routes by which persons or the environment may be exposed to the on-site chemical wastes.

2.4.1 Migration Pathways and Transport Mechanisms

Migration pathways are routes by which contaminants have moved or may be moved away from a contamination source. Pollutant migration pathways may include man-made pathways, surface drainage, vadose zone transport, and wind dispersion. Human activity (such as foot or vehicular traffic) also transports contaminants away from a source area. These five transport mechanisms are described below.

- Man-made pathways -- A site located in an urban setting has the following man-made pathways which can aid contaminant migration: storm and sanitary sewers, drainage culverts, sumps and sedimentation basins, French drain systems, and underground utility lines.

- Surface drainage -- Contaminants can be adsorbed onto sediments, suspended independently in the water column, or dissolved in surface water runoff and be rapidly carried into drainage ditches, streams, rivers, ponds, lakes, and wetlands. Consider prior surface drainage routes; historical aerial photographs can be invaluable for delineation of past surface drainage patterns. An historical aerial photograph search can be requested through the EPA Regional Remote Sensing Coordinator.

- Vadose zone transport -- Vadose zone transport is the vertical or horizontal movement of water and of soluble and insoluble contaminants within the unsaturated zone of the soil profile. Contaminants from a surface source or a leaking underground storage tank can percolate through the vadose zone and be adsorbed onto subsurface soil or reach groundwater.

- Wind dispersion -- Contaminants deposited over or adsorbed onto soil may migrate from a waste site as airborne particulates. Depending on the particle-size distribution and associated settling rates, these particulates may be deposited downwind or remain suspended, resulting in contamination of surface soils and/or exposure of nearby populations.

- Human and animal activity -- Foot and vehicular traffic of facility workers, response personnel, and trespassers can move contaminants away from a source. Animal burrowing, grazing, and migration can also contribute to contaminant migration.

2.4.2 Receptors

Once the migration pathways have been determined, identify all receptors (i.e., potentially affected human and environmental populations) along these pathways. Human receptors include on-site and nearby residents and workers. Note the attractiveness and accessibility of site wastes (including contaminated soil) to children and other nearby residents. Environmental receptors include Federal- or state-designated endangered or threatened species, habitats for these species, wetlands, and other Federal- and state-designated wilderness, critical, and natural areas.

2.5 SOIL REPRESENTATIVE SAMPLING OBJECTIVES

Collect samples if any of the following sampling objectives in the scope of the project are not fulfilled by existing data.

1. Establishing Threat to Public Health or Welfare or to the Environment -- The Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA) and the National Contingency Plan (NCP) establish the funding mechanism and authority which allow the OSC to activate a Federal removal action. The OSC must establish (often with sampling) that the site poses a threat to public health or welfare or to the environment.

2. Locating and Identifying Potential Sources of Contamination -- Sample to identify the locations and sources of contamination. Use the results to formulate removal priorities, containment and clean-up strategies, and cost projections.

3. Defining the Extent of Contamination -- Where appropriate, sample to assess horizontal and vertical extent of contaminant concentrations. Use the results to determine the site boundaries (i.e., extent of contamination), define clean areas, estimate volume of contaminated soil, establish a clearly defined removal approach, and assess removal costs and timeframe.

4. Determining Treatment and Disposal Options -- Sample to characterize soil for in situ or other on-site treatment, or excavation and off-site treatment or disposal.

5. Documenting the Attainment of Clean-up Goals -- During or following a site cleanup, sample to determine whether the goals were achieved, and to delineate areas requiring further treatment or excavation when appropriate.

2.6 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) state the level of uncertainty that is acceptable from data collection activities. DQOs also define the data quality necessary to make a certain decision. Consider the following when establishing DQOs for a particular project:

- Decision(s) to be made or question(s) to be answered;
- Why environmental data are needed and how the results will be used;
- Time and resource constraints on data collection;
- Descriptions of the environmental data to be collected;
- Applicable model or data interpretation method used to arrive at a conclusion;
- Detection limits for analytes of concern; and
- Sampling and analytical error.

In addition to these considerations, the quality assurance components of precision, accuracy (bias), completeness, representativeness, and comparability should also be considered. Quality assurance components are defined as follows:

- Precision -- measurement of variability in the data collection process.
- Accuracy (bias) -- measurement of bias in the analytical process. The term "bias" throughout this document refers to the QA/QC accuracy component.
- Completeness -- percentage of sampling measurements which are judged to be valid.
- Representativeness -- degree to which sample data accurately and precisely represent the characteristics of the site contaminants and their concentrations.
- Comparability -- evaluation of the similarity of conditions (e.g., sample depth, sample homogeneity) under which separate sets of data are produced.

Quality assurance/quality control (QA/QC) objectives are discussed further in Chapter 5.

2.7 ANALYTICAL SCREENING AND GEOPHYSICAL TECHNIQUES

There are two primary types of analytical data which can be generated during sampling: laboratory analytical data and analytical screening data. Analytical screening techniques (e.g., using a photoionization detector (PID), portable X-ray fluorescence (XRF) unit, and hazard categorization kits) provide real-time or direct reading capabilities. These screening methods can narrow the possible groups or classes of chemicals for laboratory analysis and are effective and economical for gathering large amounts of site data. Once an area is identified using screening techniques, a subset of samples can be sent for laboratory analysis to substantiate the screening results. Under a limited sampling budget, analytical screening (with laboratory confirmation) will generally result in more analytical data from a site than will sampling for off-site laboratory analysis alone. To minimize the potential for false negatives (not detecting on-site contamination), use only those analytical screening methods which provide detection limits below applicable action levels. It should be noted, that some analytical screening methods which do not achieve detection limits below site action levels can still detect grossly contaminated areas, and can be useful for some sampling events.

Geophysical techniques may also be utilized during a removal action to help depict locations of any potential buried drums or tanks, buried waste, and disturbed areas. Geophysical techniques include ground penetrating radar (GPR), magnetometry, electromagnetic conductivity (EM) and resistivity surveys.

2.8 PARAMETERS FOR ANALYSIS

If the historical data review yields little information about the types of waste on site, use applicable screening methods to narrow the parameters for analysis by ruling out the presence of high concentrations of certain contaminants. If the screening results are inconclusive, send a subset of samples from the areas of concern for a full chemical

characterization by an off-site laboratory. It is advised that samples from known or suspected source areas be sent to the laboratory for a full chemical characterization so that all contaminants of concern can be identified (even at low detection levels), and future sampling and analysis can then focus on those substances.

Away from source areas, select a limited number of indicator parameters (e.g., lead, PAHs) for analysis based on the suspected contaminants of concern. This will result in significant cost savings over a full chemical characterization of each sample. Utilize EPA-approved methodologies and sample preparation, where possible, for all requested off-site laboratory analyses.

2.9 SAMPLING APPROACHES

Selecting sampling locations for screening or laboratory analysis entails choosing the most appropriate sampling approach. Representative sampling approaches include **judgmental, random, stratified random, systematic grid, systematic random, search, and transect sampling**. A representative sampling plan may combine two or more of these approaches. Each approach is defined below.

2.9.1 Judgmental Sampling

Judgmental sampling is the subjective selection of sampling locations at a site, based on historical information, visual inspection, and on best professional judgment of the sampling team. Use judgmental sampling to identify the contaminants present at areas having the highest concentrations (i.e., worst-case conditions). Judgmental sampling has no randomization associated with the sampling strategy, precluding any statistical interpretation of the sampling results.

2.9.2 Random Sampling

Random sampling is the arbitrary collection of samples within defined boundaries of the area of concern. Choose random sample locations using a random selection procedure (e.g., using a random number table). Refer to U.S. EPA, 1984a, for a random number table. The arbitrary selection of sampling points requires each sampling point to be selected independent of the location of all other points, and results in all locations within the area of concern having an equal chance of being selected. Randomization is necessary in order to make probability or confidence statements about the sampling results. The key to interpreting these probability statements is the assumption that the site is homogeneous with respect to the parameters being monitored. The higher the degree of heterogeneity, the less the random sampling approach will adequately characterize true conditions at the site. Because hazardous waste sites are very rarely homogeneous, other statistical sampling approaches (discussed below) provide ways to subdivide the site into more homogeneous areas. These sampling approaches may be more appropriate for removal activities than random sampling. Refer to U.S. EPA, February 1989, pages 5-3 to 5-5 for guidelines on selecting sample coordinates for random sampling. Figure 2 illustrates a random sampling approach.

2.9.3 Stratified Random Sampling

Stratified random sampling often relies on historical information and prior analytical results (or screening data) to divide the sampling area into smaller areas called strata. Each strata is more homogeneous than the site is as a whole. Strata can be defined based on various factors, including: sampling depth, contaminant concentration levels, and contaminant source areas. Place sample locations within each of these strata using random selection procedures. Stratified random sampling imparts some control upon the sampling scheme but still allows for random sampling within each stratum. Different sampling approaches may also be selected to address the different strata at the site. Stratified random sampling is a useful and flexible design for estimating the pollutant concentration within each depth interval or area of concern. Figure 3 illustrates a stratified random sampling approach where strata are defined based on depth. In this example, soil coring devices are used to collect samples from given depths at randomly selected locations within the strata.

Figure 2: Random Sampling

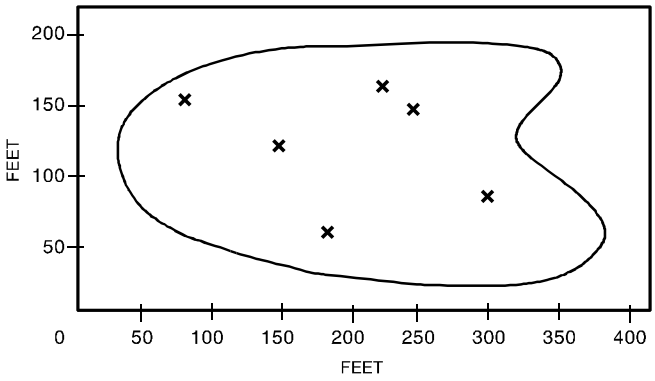


Figure 3: Stratified Random Sampling

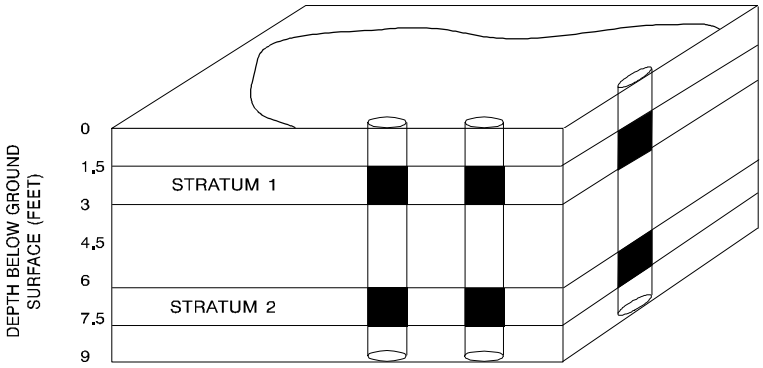
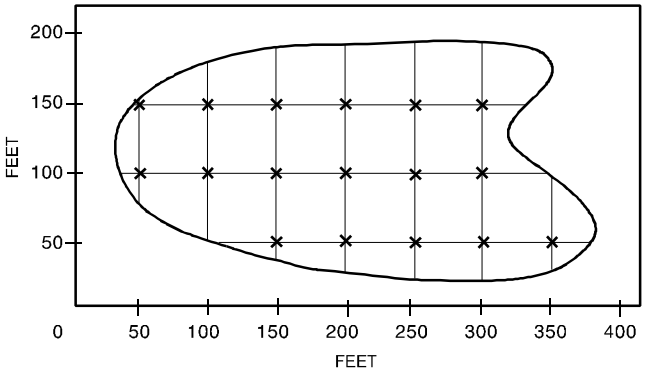


Figure 4: Systematic Grid Sampling



KEY

× SELECTED SAMPLE LOCATION

After U.S. EPA, February 1989

2.9.4 Systematic Grid Sampling

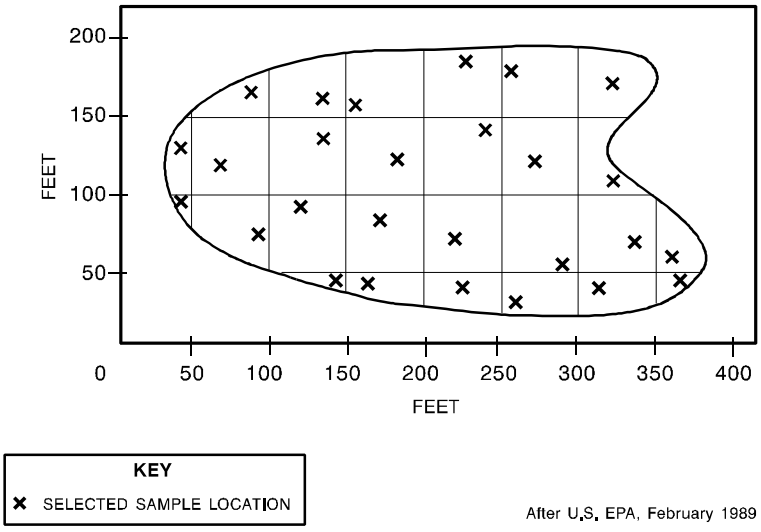
Systematic grid sampling involves subdividing the area of concern by using a square or triangular grid and collecting samples from the nodes (intersections of the grid lines). Select the origin and direction for placement of the grid using an initial random point. From that point, construct a coordinate axis and grid over the whole site. The distance between sampling locations in the systematic grid is determined by the size of the area to be sampled and the number of samples to be collected.

Systematic grid sampling is often used to delineate the extent of contamination and to define contaminant concentration gradients. Refer to U.S. EPA February 1989, pages 5-5 to 5-12, for guidelines on selection of sample coordinates for systematic grid sampling. Figure 4 illustrates a systematic grid sampling approach.

2.9.5 Systematic Random Sampling

Systematic random sampling is a useful and flexible design for estimating the average pollutant concentration within grid cells. Subdivide the area of concern using a square or triangular grid (as described in Section 2.9.4) then collect samples from within each cell using random selection procedures. Systematic random sampling allows for the isolation of cells that may require additional sampling and analysis. Figure 5 illustrates a systematic random sampling approach.

Figure 5: Systematic Random Sampling



After U.S. EPA, February 1989

2.9.6 Search Sampling

Search sampling utilizes either a systematic grid or systematic random sampling approach to search for areas where contaminants exceed applicable clean-up standards **(hot spots)**. The number of samples and the grid spacing are determined on the basis of the acceptable level of error (i.e., the chance of missing a hot spot). Search sampling requires that assumptions be made about the size, shape, and depth of the hot spots. As illustrated in Figure 6, the smaller and/or narrower the hot spots are, the smaller the grid spacing must be in order to locate them. Also, the smaller the acceptable error of missing hot spots is, the smaller the grid spacing must be. This, in effect, means collecting more samples.

Once grid spacing has been selected, the probability of locating a hot spot can be determined. Using a systematic grid approach, Table 1 lists approximate probabilities of missing an elliptical hot spot based on the grid method chosen as well as the dimensions of the hot spot. The lengths of the long and short axes (L and S) are represented as a percentage of the grid spacing chosen. The triangular grid method consistently shows lower probabilities of missing a hot spot in comparison to the block grid method. Table 1 can be used in two ways. If the acceptable probability of missing a hot spot is known, then the size of the hot spot which can be located at that probability level can be determined. Conversely, if the approximate size of the hot spot is known, the probability of locating it can be determined.

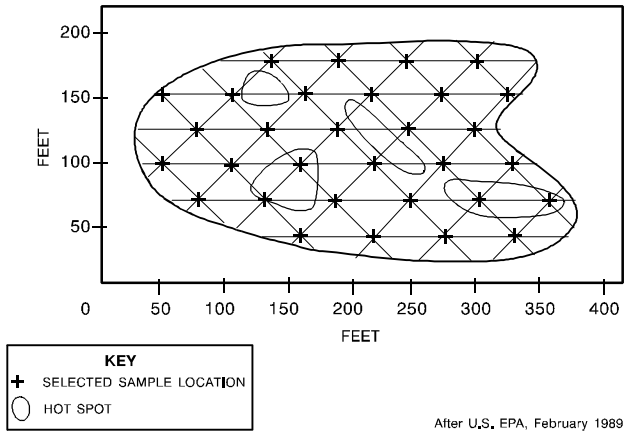
For example, suppose the block grid method is chosen with a grid spacing of 25 feet. The OSC is willing to accept a 10% chance of missing an elliptical hot spot. Using Table 1, there would be a 90% probability of locating an elliptical hot spot with L equal to 90% of the grid spacing chosen and S equal to 40% of the grid spacing chosen. Therefore the smallest elliptical hot spot which can be located would have a long axis L = 0.90 × 25ft. = 22.5 ft. and a short axis S = 0.40 × 25ft. = 10 ft.

Similarly, if the approximate size of the hot spot being searched for is known, then the probability of missing that hot spot can be determined. For example, if a triangular grid method was chosen with a 25 foot grid spacing and the approximate shape of the hot spot is known, and L is approximately 15 feet or 60% of the grid spacing, and S is approximately 10 feet or 40% of the grid spacing, then there is approximately a 15% chance of missing a hot spot of this size and shape.

2.9.7 Transect Sampling

Transect sampling involves establishing one or more transect lines across the surface of a site. Collect samples at regular intervals along the transect lines at the surface and/or at one or more given depths. The length of the transect line and the number of samples to be collected determine the spacing between sampling points along the transect. Multiple transect lines may be parallel or non-parallel to one another. If the lines are parallel, the sampling objective is sim-

Figure 6: Search Sampling



After U.S. EPA, February 1989

Table 1: Probability of Missing an Elliptical Hot Spot

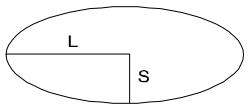
LENGTH OF SHORT AXIS AS A PERCENTAGE OF GRID SPACING

LENGTH OF LONG AXIS AS A PERCENTAGE OF GRID SPACING

	10%	20%	30%	40%	50%	60%	70%	80%	90%	100%
10%	0.97 / 0.95									
20%	0.95 / 0.92	0.88 / 0.85								
30%	0.92 / 0.87	0.83 / 0.78	0.72 / 0.66							
40%	0.88 / 0.85	0.75 / 0.71	0.65 / 0.55	0.50 / 0.41						
50%	0.85 / 0.82	0.69 / 0.63	0.54 / 0.44	0.38 / 0.27	0.21 / 0.08					
60%	0.80 / 0.80	0.62 / 0.58	0.45 / 0.35	0.27 / 0.15	0.12 / 0.03	0.06 / 0.0				
70%	0.77 / 0.77	0.56 / 0.54	0.38 / 0.29	0.18 / 0.12	0.07 / 0.01	0.03 / 0.0	0.0 / 0.0			
80%	0.75 / 0.75	0.54 / 0.50	0.32 / 0.23	0.12 / 0.08	0.05 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0		
90%	0.72 / 0.72	0.51 / 0.45	0.30 / 0.21	0.10 / 0.06	0.03 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	
100%	0.70 / 0.66	0.45 / 0.37	0.24 / 0.18	0.08 / 0.04	0.01 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0

From tables In Gilbert, 1987

L=length of long side
S=length of short side



BLOCK GRID 0.97 / 0.95 TRIANGULAR GRID

ilar to systematic grid sampling. A primary benefit of transect sampling over systematic grid sampling is the ease of establishing and relocating individual transect lines versus an entire grid. Transect sampling is often used to delineate the extent of contamination and to define contaminant concentration gradients. It is also used, to a lesser extent, in compositing sampling schemes. For example, a transect sampling approach might be used to characterize a linear feature such as a drainage ditch. A transect line is run down the center of the ditch, along its full length. Sample aliquots are collected at regular intervals along the transect line and are then composited. Figure 7 illustrates transect sampling.

Table 2 summarizes the various representative sampling approaches and ranks the approaches from most to least suitable, based on the sampling objective. Table 2 is intended to provide general guidelines, but it cannot cover all site-specific conditions encountered.

2.10 SAMPLING LOCATIONS

Once a sampling approach has been selected, the next step is to select sampling locations. For statistical (non-judgmental) sampling, careful placement of each sampling point is important to achieve representativeness.

Factors such as the difficulty in collecting a sample at a given point, the presence of vegetation, or discoloration of the soil could bias a statistical sampling plan.

Sampling points may be located with a variety of methods. A relatively simple method for locating random points consists of using either a compass and a measuring tape, or pacing, to locate samplingpoints with respect to a permanent landmark, such as a survey marker. Then plot sampling coordinates on a map and mark the actual sampling points for future reference. Where the sampling design demands a greater degree of precision, locate each sample point by means of a survey. After sample collection, mark each sample point with a permanent stake so that the survey team can identify all the locations.

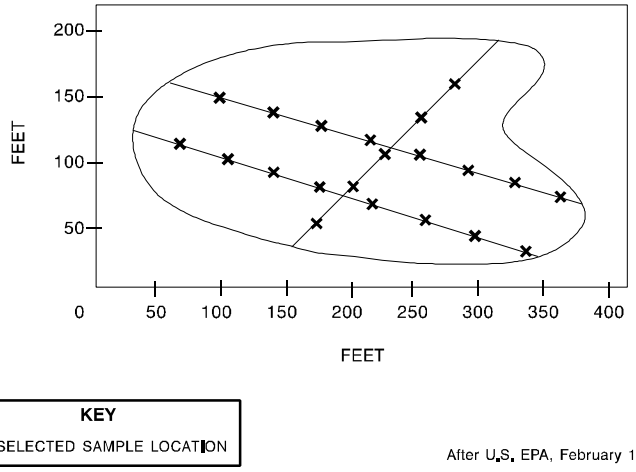
2.11 EXAMPLE SITE



2.11.1 Background Information

The ABC Plating Site is located in Carroll County, Pennsylvania, approximately 1.5 miles north of the town of Jonesville (Figure 8). The site covers approximately 4 acres, and operated as an electroplating facility from 1947 to 1982. During its years of operation, the company plated automobile and airplane parts with chromium, nickel, and copper. Cyanide solutions were used in the plating process. ABC Plating deposited electroplating wastes into two shallow surface settling lagoons in the northwest sector of the site. The county environmental health department was attempting to enforce cleanup by the site owner, when, in early 1982, a fire on site destroyed most of the process building. The owner then abandoned the facility and could not be located by enforcement and legal authorities. The county contacted EPA for an assessment of the site for a possible response.

Figure 7: Transect Sampling



After U.S. EPA, February 1989

Table 2: Representative Sampling Approach Comparison

SAMPLING OBJECTIVE	SAMPLING APPROACH: JUDGEMENTAL	RANDOM	STRATIFIED RANDOM	SYSTEMATIC GRID	SYSTEMATIC RANDOM	SEARCH	TRANSECT
ESTABLISH THREAT	1	4	3	2 [a]	3	3	2
IDENTIFY SOURCES	1	4	2	2 [a]	3	2	3
DELINEATE EXTENT OF CONTAMINATION	4	3	3	1 [b]	1	1	1
EVALUATE TREATMENT & DISPOSAL OPTIONS	3	3	1	2	2	4	2
CONFIRM CLEANUP	4	1 [c]	3	1 [b]	1	1	1 [d]

1 - PREFERRED APPROACH
2 - ACCEPTABLE APPROACH
3 - MODERATELY ACCEPTABLE APPROACH
4 - LEAST ACCEPTABLE APPROACH
a - SHOULD BE USED WITH FIELD ANALYTICAL SCREENING
b - PREFERRED ONLY WHERE KNOWN TRENDS ARE PRESENT
c - ALLOWS FOR STATISTICAL SUPPOFT OF CLEANUP VERIFICATION IF SAMPLING OVER ENTIRE SITE
d - MAY BE EFFECTIVE WITH COMPOSTING TECHNIQUE IF SITE IS PRESUMED TO BE CLEAN

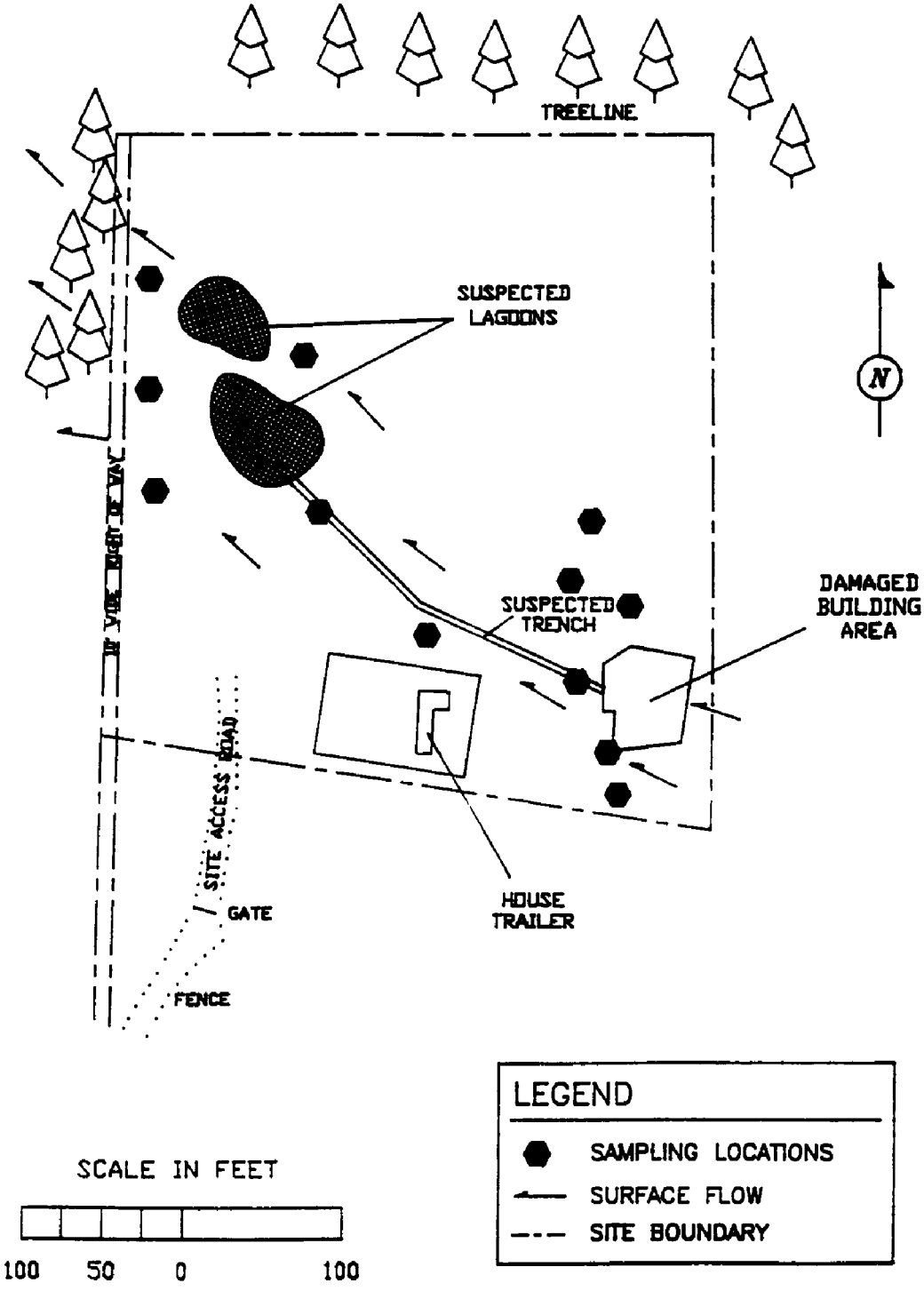
2.11.2 Historical Data Review and Site Reconnaissance

The EPA On-Scene Coordinator (OSC) reviewed the county site file, finding that in 1974, the owner was cited for violating the Clean Streams Act and for storing and treating industrial waste without a permit. The owner was ordered to file a site closure plan and to remediate the storage lagoons. The owner, however, continued operations and was then ordered to begin remediation in 90 days or be issued a cease and desist order. Soon after, a follow-up inspection revealed that the lagoons had been backfilled without removing the waste.

The OSC and response contractor arrived on site to interview local officials, fire department officers, neighboring residents (including a past facility employee), and county representatives, regarding site operating practices and other site details. A past employee sketched facility process features on a map which was obtained from the county (Figure 8). The features included two settling lagoons and a feeder trench which transported plating wastes from the process building to the lagoons. The OSC obtained copies of aerial photographs of the site area from the district office of the U.S. Soil Conservation Service. The county also provided the OSC with copies of all historical site and violation reports.

The OSC and response contractor made a site entry utilizing appropriate personal protective equipment and instrumentation. They observed 12 vats, likely containing plating solutions, on a concrete pad where the original facility building once stood.

Figure 8: Site Sketch and Phase I Soil Sampling Locations
ABC Plating Site



Measurements of pH ranged from 1 to 11. In addition, 50 drums and numerous smaller containers (some on the concrete pad, others sitting directly on the ground) were leaking and bulging, due to the fire. The response contractor noted many areas of stained soil, which indicated container leakage, poor waste handling practices, and possible illegal dumping of wastes.

2.11.3 Identification of Migration Pathways, Transport Mechanisms and Receptors

During the site entry, the OSC noted that several areas were devoid of vegetation, threatening wind erosion which could transport heavy metal- and cyanide-contaminated soil particulates off site. These particulates could be deposited on residential property downwind or be inhaled by nearby residents.

Erosion gullies located on site indicated soil erosion and fluvial transport due to storms. Surface drainage sloped towards the northwest. The response contractor observed stressed and discolored vegetation immediately off site, along the surface drainage route. Surface drainage of heavy metals and cyanide was a direct contact hazard to local residents. Further downgradient, runoff enters an intermittent tributary of Little Creek. Little Creek in turn feeds Barker Reservoir, the primary water supply for the City of Jonesville and neighboring communities, which are located 2.5 miles downgradient of the site. The site entry team observed that the site was not secure and there were signs of trespass (confirming a neighbor's claim that children play at the facility). These activities could lead to direct contact with cyanide and heavy metal contaminants, in addition to the potential for chemical burns from direct contact with strong acids and bases.

Information obtained from the historical data review and site reconnaissance was used to create a site-specific conceptual model. Sources (e.g., vats, drums), pathways (e.g., gullies) and potential receptors (e.g., local residents) were detailed on a map to assist the selection of sampling approaches, objectives, and locations.

2.11.4 Sampling Objectives

The OSC selected three specific sampling objectives, as follows:

- Phase 1 -- Determine whether a threat to public health, welfare, and the environment exists. Identify sources of contamination to support an immediate CERCLA-funded activation for containment of contaminants and security fencing.

- Phase 2 -- Define the extent of contamination at the site and adjacent residential properties. Estimate the volume of contaminated soil and the associated removal costs.

- Phase 3 -- After excavation (or treatment), document the attainment of clean-up goals. Assess that cleanup was completed to the selected level.

2.11.5 Selection of Sampling Approaches

The OSC selected a judgmental sampling approach for Phase 1. Judgmental sampling supports the Action Memorandum process by best defining on site contaminants in the worst-case scenario in order to evaluate the threat to human health, welfare, and the environment. Threat is typically established using a relatively small number of samples (less than 20) collected from source areas, or suspected contaminated areas based on the historical data review and site reconnaissance. For this site, containerized wastes were screened to categorize the contents and to establish a worst- case waste volume, while soil samples were collected to demonstrate whether a release had already occurred.

For Phase 2, a stratified systematic grid design was selected to define the extent of contamination. The grid can accommodate analytical screening and geophysical surveys and allow for contaminated soil excavation on a cell-by-cell basis. Based on search sampling conducted at similar sites, the hot spots being searched for were assumed to be elliptical in shape and 45 feet by 20 feet in size. Under these assumptions, a block grid, with a 50 foot grid spacing, was selected. This grid size ensured a no more than 10% probability of missing a hot spot (see Table 1). The grid was extended to adjacent residential properties when contaminated soil was identified at grid points near the boundary of the site.

Phase 3 utilized a systematic grid sampling approach to confirm the attainment of clean-up goals. Following cleanup, analytical screening was conducted on excavated soil areas using a

transportable X-ray fluorescence (XRF) unit mounted in a trailer (mobile laboratory instrument). Based on the results, each area was documented as clean, or was excavated to additional depth, as necessary.

2.11.6 Analytical Screening, Geophysical Techniques, and Sampling Locations

During Phase 1 operations, containerized wastes were screened using hazard categorization techniques to identify the presence of acids, bases, oxidizers, and flammable substances. Following this procedure, photoionization detector (PID) and flame ionization detector (FID) instruments, a radiation meter, and a cyanide monitor were used to detect the presence of volatile organic compounds, radioactive substances, and cyanide, respectively, in the containerized wastes. Phase 1 screening indicated the presence of strong acids and bases and the absence of volatile organic compounds. The response contractor collected a total of 12 surface soil samples (0-3 inches) during this phase and sent them to a laboratory for analysis. The soil sampling locations included stained soil areas, erosion channels and soil adjacent to leaking containers. Background samples were not collected during Phase 1 because they were unnecessary for activating funding. Phase 1 sampling locations are shown in Figure 8. Based on Phase 1 analytical results, consultation with a Regional EPA toxicologist and with the Agency for Toxic Substances and Disease Registry (ATSDR), an action level of 100 ppm for chromium was selected for cleanup.

During Phase 2 sampling activities, the OSC used a transportable XRF unit installed in an on-site trailer to screen samples for total chromium in order to limit the number of samples to be sent for off-site laboratory analysis. The transportable XRF (rather than a portable unit) was selected for analytical screening to accommodate the 100 ppm action level for chromium. Sampling was performed at all grid nodes at the surface (0-4 inches) and subsurface (36-40 inches) (Figure 9). The 36-40 inch depth was selected based on information obtained from county reports and local interviews which indicated the lagoon wastes were approximately 3 feet below ground surface. The samples were homogenized and sieved (discussed in Chapter 4), then screened for chromium using the XRF. The surface and subsurface samples from areas downgradient of the original facility (21 grid nodes) and three upgradient (background) locations were sent for off-site laboratory analysis following XRF screening. The analytical results from these samples allowed for site-specific calibration of the XRF unit. Once grid nodes with a contamination level greater than the selected action level were located, composite samples were collected from each adjoining cell. Surface aliquots were collected and then composited, sieved, thoroughly homogenized, and screened using the XRF to pinpoint contaminated cells. Additionally, four subsurface aliquots were collected at the same locations as the surface aliquots. They were also composited, sieved, thoroughly homogenized, and screened using the XRF. Figure 10 illustrates a Phase 2 sampling grid cell diagram. Based on the XRF data, each adjoining cell was either identified as clean (below action level), or designated for excavation (at or above action level).

For Phase 3 sampling, cleanup was confirmed by collecting and compositing four aliquots from the surface of each grid cell excavated during Phase 2. The surface composites were then screened (as in Phase 2), using the transportable XRF. Ten percent of the screened samples were also sent to an off-site laboratory for confirmatory sampling. Based on the Phase 3 screening and sampling results, each cell was documented as clean, or, excavated to additional depth, as necessary.

During Phase 2, the OSC conducted ground penetrating radar (GPR) and electromagnetic conductivity (EM) geophysical surveys to help delineate the buried trench and lagoon areas along with any other waste burial areas. The GPR survey was run along the north-south grid axis across the suspected locations of the trench and lagoons. Several structural discontinuities, defining possible disturbed areas, were detected. One anomaly corresponded with the suspected location and orientation of the feeder trench. Several discontinuities were identified in the suspected lagoon areas; however, the data did not conclusively pinpoint precise locations. This could be due to a disturbance of that area during the backfilling process by the PRP. The GPR survey is illustrated in Figure 11.

Figure 9: Soil Sampling and SRF Screening Locations
ABC Plating Site

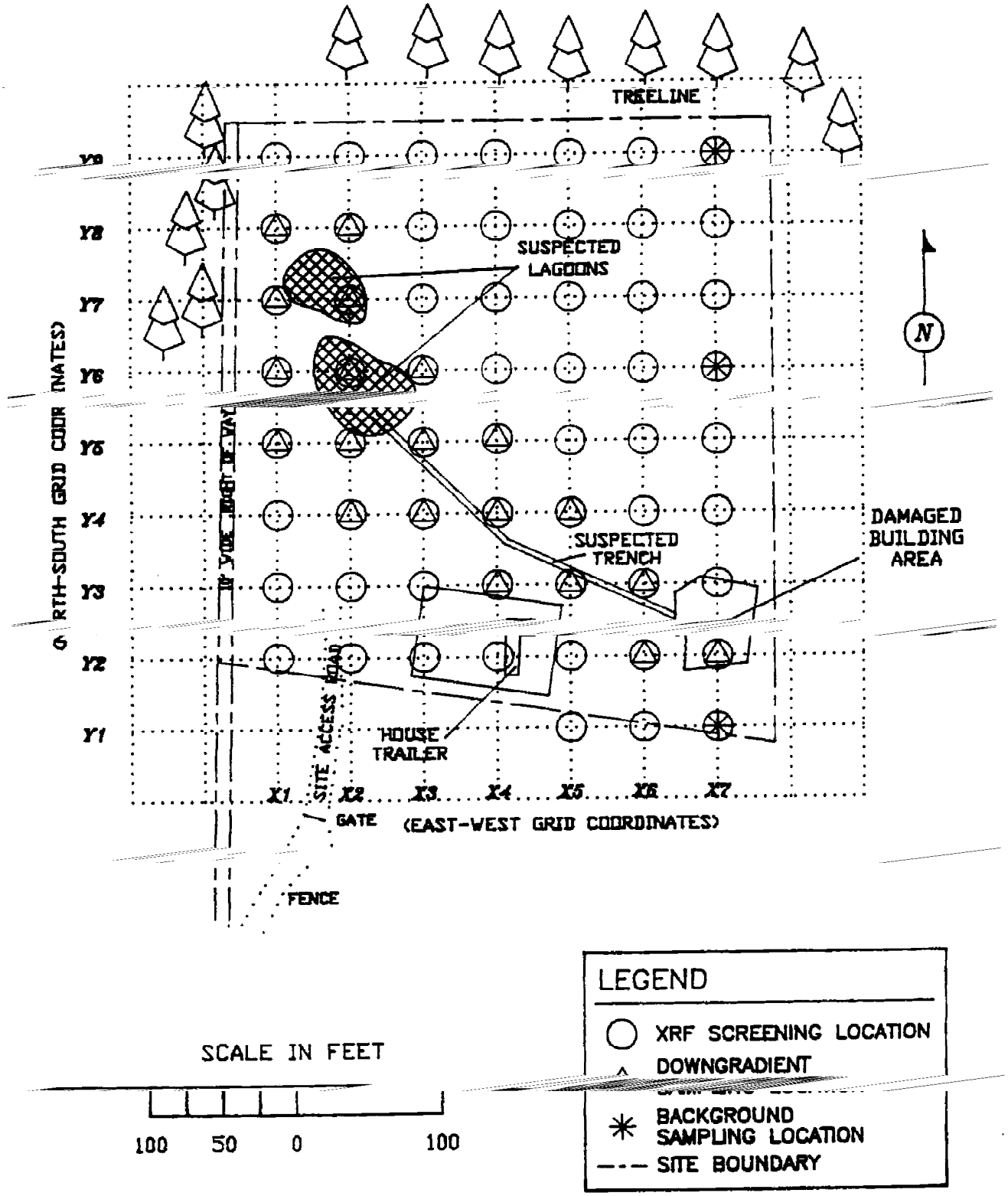
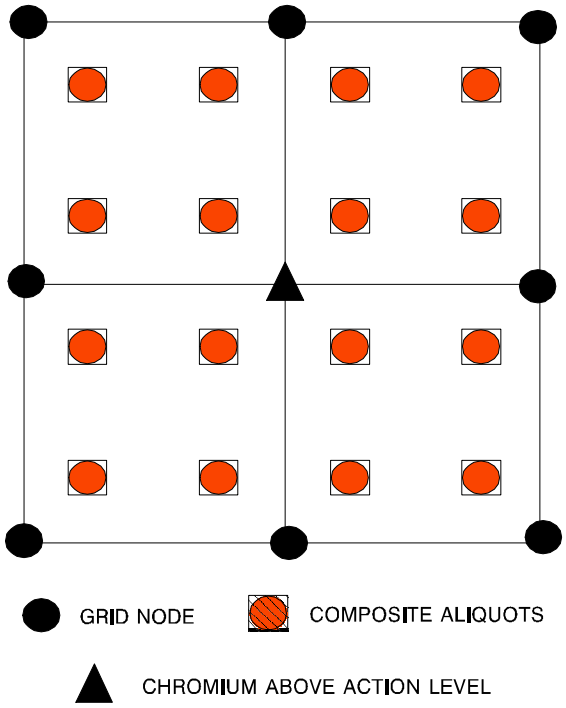


Figure 10: Phase 2 Sampling Grid Cell Diagram*



* Surface samples should be taken over a minimum area of one square foot. Sampling areas for depth sampling are limited by the diameter of the sampling equipment (e.g., auger, split spoon, or coring devices).

For the comprehensive EM survey, the original 50 foot grid spacing was decreased to 25 feet along the north-south grid axis. The EM survey was run along the north-south axes and readings were obtained at the established grid nodes. The EM survey was utilized throughout the site to detect the presence of buried metal objects (e.g., buried pipe leading to the lagoons), and potential subsurface contaminant plumes. The EM survey identified several high conductivity anomalies: the suspected feeder trench location, part of the lagoon area, and a small area west of the process building (Figure 12), which could have been an illegal waste dumping area. Several areas of interference were encountered due to the presence of large metal objects at the surface (a dumpster, surface vats and a junk car).

2.11.7 Parameters for Analysis

During Phase 1 sampling activities, full priority pollutant metals and total cyanide analyses were conducted on all samples. Since Phase 1 samples were collected from the areas of highest suspected contaminant concentration (i.e., sources and drainage pathways), Phase 2 samples were run for total chromium and cyanide, the only analytes detected during the Phase 1 analyses. During Phase 3, the samples sent to the laboratory for definitive analysis were analyzed for total chromium and cyanide. Throughout the removal, it was not possible to screen soils on site for cyanide, therefore the OSC requested laboratory cyanide analysis on the 10% confirmatory samples.

Figure 11: GPR Survey Results
ABC Plating Site

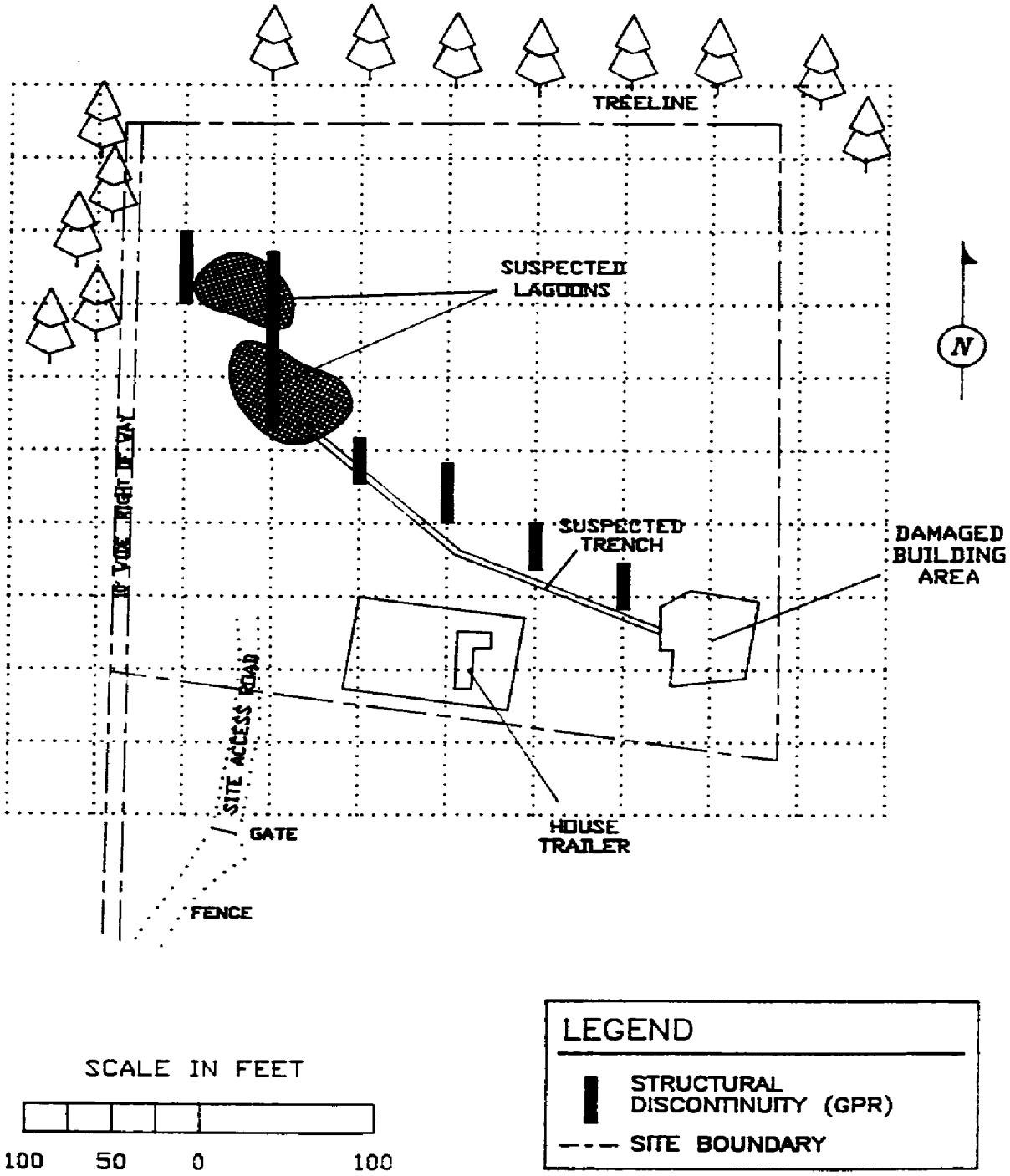
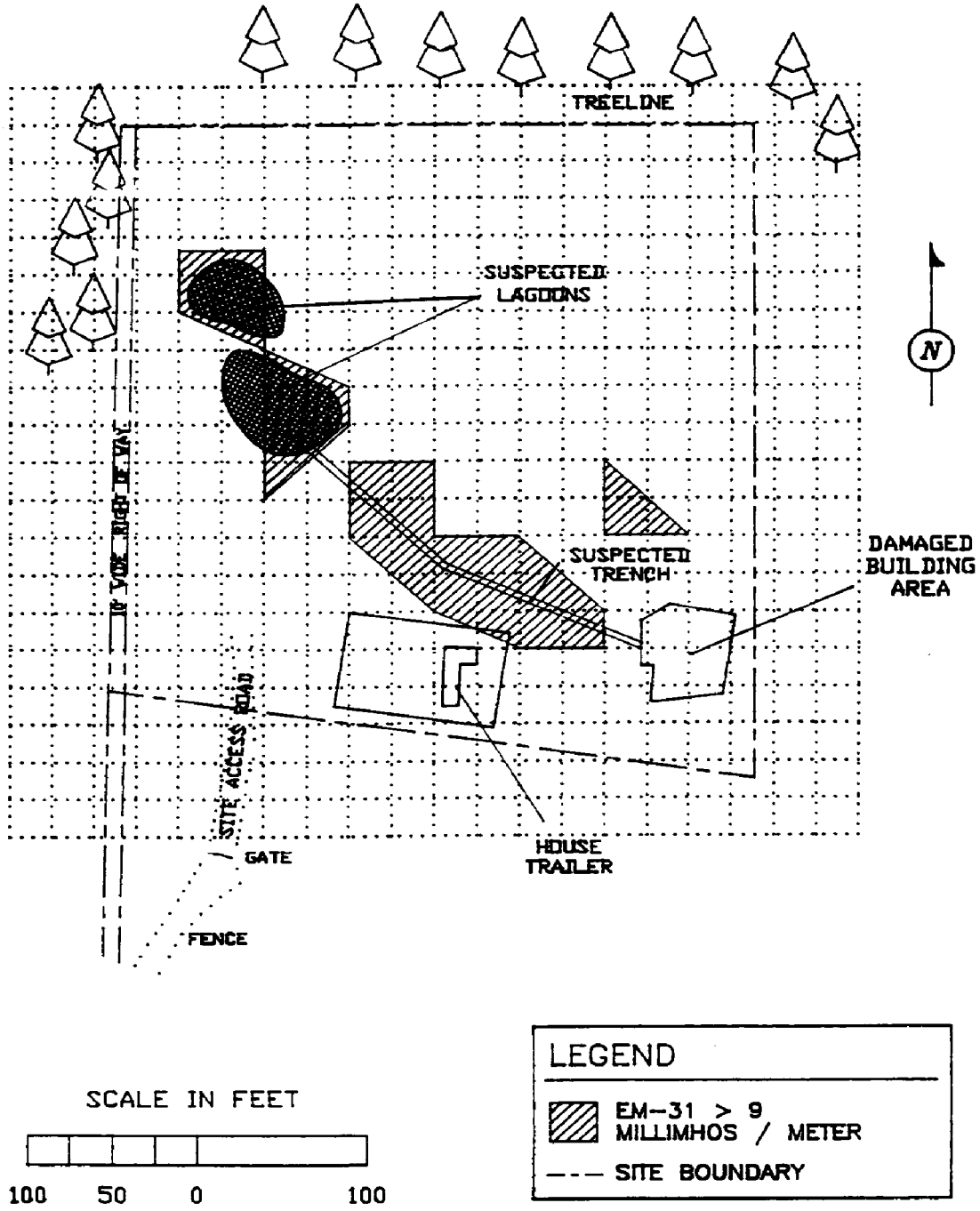


Figure 12: EM-31 Survey Results
ABC Plating Site



3.0 EQUIPMENT

3.1 INTRODUCTION

Sample collection requires an understanding of the capabilities of the sampling equipment, since using inappropriate equipment may result in biased samples. This chapter provides information for selecting sampling and screening equipment.

3.2 ANALYTICAL SCREENING EQUIPMENT

Analytical screening methods provide on-site measurements of contaminants of concern, limiting the number of samples which need to be sent to an off-site laboratory for time-consuming and often costly analysis. Screening techniques can also evaluate soil samples for indications that soil contamination exists (e.g., X-ray fluorescence (XRF) for target metals or soil gas survey for identification of buried wastes or other subsurface contamination). All screening equipment and methods described in this section are **portable** (the equipment is hand-held, and generally no external power is necessary). Examples are photoionization detectors (PID), flame ionization detectors (FID), and some XRF devices.

Screening generally provides analytical data of suitable quality for site characterization, monitoring during response activities, and on-site health and safety decisions. The methods presented here can provide rapid, cost-effective, real-time data; however, results are often not compound-specific and not quantitative.

When selecting one screening method over another, consider relative cost, sample analysis time, potential interferences or instrument limitations, detection limit, QA/QC requirements, level of training required for operation, equipment availability, and data bias. Also consider which elements, compounds, or classes of compounds the screening instrument is designed to analyze. As discussed in Section 2.7, the screening method selected should be sensitive enough to minimize the potential for false negatives. When collecting samples for on-site analysis (e.g., XRF), evaluate the detection limits and bias of the screening method by sending a minimum of 10% of the samples to an off-site laboratory for confirmation. Table 3 summarizes the advantages and disadvantages of selected portable screening equipment.

3.3 GEOPHYSICAL EQUIPMENT

Geophysical techniques can be used in conjunction with analytical screening to help delineate areas of subsurface contamination, including buried drums and tanks. Geophysical data can be obtained relatively rapidly, often without disturbing the site. Geophysical techniques suitable for emergency or removal activities include: ground penetrating radar (GPR), magnetometry, electromagnetic conductivity (EM) and resistivity. Specific advantages and disadvantages associated with geophysical equipment are summarized in Table 4. See also EPA ERT Standard Operating Procedure (SOP) #2159, General Surface Geophysics (U.S. EPA, January 1991).

3.4 SELECTING SAMPLING EQUIPMENT

The mechanical method by which a sampling tool collects the sample may impact representativeness. For example, if the sampling objective is to determine the concentrations of contaminants at each soil horizon interface, using a hand auger would be inappropriate: the augering technique would disrupt and mix soil horizons, making the precise horizon interface difficult to determine. Depth of sampling is another factor to consider in the proper selection of sampling equipment. A trowel, for example, is suitable for unconsolidated surface soils, but may be a poor choice for sampling at 12 inches, due to changes in soil consistency with depth.

All sampling devices should be of sufficient quality not to contribute contamination to samples (e.g., painted surfaces which could chip off into the sample). In addition, the sampling equipment should be either easily decontaminated, or cost-effective if considered to be expendable. Consider ease of use when selecting sampling equipment.

Table 3: Portable Field Analytical Screening Equipment

Equipment	Application to Sampling Design	Advantages and Disadvantages
X-ray fluorescence (portable)	Detects heavy metals in soils	Rapid sample analysis; may be used in situ; requires trained operator; potential matrix interferences; may be used with a generic or site-specific calibration model; detection limit may exceed action level; detects to ppm level; detection limit should be calculated on a site-specific basis.
Flame ionization detector (FID)	Semi-quantitatively detects VOCs in soils	Immediate results; can be used in GC mode to identity specific organic compounds; detects VOCs only; detects to ppm level.
Photoionization detector (PID)	Detects total concentration of VOCs and some non-volativle organics and inorganics in soils	Immediate results; easy to use; non-compound specific; results affected by high ambient humidity and electrical sources such as radios; does not respond to methane; detects to ppm level.
Field test kits	Detects specific elements, compounds, or compound classes in soils	Rapid results; easy to use; low cost; limited number of kit types available; kits may be customized to user needs; semi-quantitative; interferences by other analytes is common; colorimetric interpretation is needed; detection level dependent upon type of kit used; can be prone to error.
Radiation detector	Detects the presence of selected forms of radiation in soils or other waste materials	Easy to use; low cost; probes for one or a combination of alpha, beta or gamma forms of radiation; unit and detection limits vary greatly; detailed site surveys are time intensive and require experienced personnel to interpret results.

Sources: U.S. EPA, September 1988a; U.S. EPA, December 1987; U.S. EPA 1987.

Table 4: Geophysical Equipment

Equipment	Application to Sampling Design	Advantages and Disadvantages
Ground penetrating radar (GPR)	Detects reflection anomalies caused by lithology changes buried objects; varying depths of investigation, 15 to 30 feet, are possible.	Capable of high resolution; generates continuous measurement profile; can survey large area quickly; site specific; best results are achieved in dry, sandy soils; clay-rich and water saturated soils produce poor reflections and limit depth of penetration; data interpretation requires a trained geophysicist.
Magnetometer	Detects presence and areal extent of ferromagnetic material in subsurface soils, including buried metal containers. Single 55-gallon drums can be identified at depths up to 10 feet and large massed of drums up to 30 feet or more.	Quick and easy to operate; good initial survey instrument; readings are often affected by nearby man-made steel structures (including above-ground fences, buildings, and vehicles); data interpretation may require geophysicist.
Electromagnetic conductivity meter (EM)	Detects electrical conductivity changes in subsurface geologic lithology, pore fluids, and buried objects. Depth of investigation varies from 9 feet to 180 feet depending on instrument used, coil spacing, and coil configuration.	Rapid data collection; can delineate inorganic and large-scale organic contamination in subsurface fluids; sensitive to man-made structures (including buried cables, above-ground steel structures and electrical power lines); survey planning and data interpretation may require geophysicist.
Wadi	Detects electrical conductivity changes in surface and sub-surface materials utilizing existing very low frequency (VLF) radio waves.	Utilizes existing long-distance communication VLF radio waves (10-30 Khz range); no need to induce electrical field; directional problems can be overcome with portable transmitters.
Resistivity meter	Detects electrical resistivity variations in subsurface materials (e.g., lithology, pore fluids, buried pipelines and drums). Vertical resolution to depths of 100 feet are possible.	Detects lateral and vertical variations; instrument requires direct ground contact, making it relatively labor intensive; sensitive to outside interference; data interpretation requires a trained geophysicist.

Sources: Benson, et. al. 1988; NJDEP, 1988.

Complicated sampling procedures usually require increased training and introduce a greater likelihood of procedural errors. Standard operating procedures help to avoid such errors. Sample volume is another selection concern. Specific advantages and disadvantages of soil sampling equipment are given in Table 5. Refer also to EPA ERT SOP #2012, Soil Sampling (in U.S. EPA, January 1991) for guidance on using various types of soil sampling equipment.

3.5 EXAMPLE SITE



3.5.1 Selection of Sampling Equipment

Dedicated plastic scoops were used for Phase 1 soil sampling. For Phase 2, the OSC used bucket augers for both surface and subsurface soil sampling because of their ease of use, good vertical depth range, and uniform surface sampling volume. Standard operating procedures were followed to promote proper sample collection, handling, and decontamination. From the bucket auger, each sample was placed into a dedicated plastic pan and mixed using a dedicated plastic scoop. Samples were further prepared for XRF screening and laboratory analysis (Section 4.8).

3.5.2 Selection of Analytical Screening Equipment

Phase 1 sampling identified the sources and types of on-site contaminants in order to establish a threat. Hazard categorization techniques, organic vapor detecting instruments, and radiation and cyanide monitors were utilized to tentatively identify containerized liquid wastestreams in order to select initial judgmental soil sampling locations. During Phase 2 sampling, a portable XRF unit was used to determine the extent of contamination and to identify additional hot spots. Samples to be sent for laboratory analysis were then placed into sampling jars (as discussed in Section 4.8). Samples collected from upgradient grid nodes for XRF screening only were stored on site for later treatment/disposal. For Phase 3, the XRF was used to confirm whether contaminated areas identified during Phase 2 were sufficiently excavated.

3.5.3 Selection of Geophysical Equipment

The GPR instrument delineated buried trench and lagoon boundaries. The EM meter detected subsurface conductivity changes due to buried metal containers and contaminants. The EM-31 (a shallower-surveying instrument than the EM-34) was selected because expected contaminant depth was less than 10 feet and because of the instrument's maneuverability and ease of use.

Table 5: Soil Sampling Equipment

Equipment	Applicability	Advantages and Disadvantages
Trier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stony, dry, or sandy soil.
Scoop or trowel	Soft surface soil	Inexpensive, easy to use and decontaminate; trowels with painted surfaces should be avoided.
Tulip bulb planter	Soft soil, 0-6 in.	Easy to use and decontaminate; uniform diameter and sample volume; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; not useful for hard soils.
Soil coring device	Soft soil, 0-24 in.	Relatively easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; can be difficult to decontaminate.
Thin-wall tube sampler	Soft soil, 0-10 ft.	Easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); may be used in conjunction with bucket auger; acetate sleeve may be used to help maintain integrity of VOA samples, easy to decontaminate; can be difficult to remove cores from sampler.
Split spoon sampler	Soil, 0 in.-bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOA samples; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores.
Shelby tube sampler	Soft soil, 0 in.-bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); tube may be used to ship sample to lab undisturbed; may be used in conjunction with drill rig for obtaining deep cores and for permeability testing; not durable in rocky soils.
Bucket auger	Soft soil, 3 in.-10 ft.	Easy to use; good depth range; uniform diameter and sample volume; acetate sleeve may be used to help maintain integrity of VOA samples; may disrupt and mix soil horizons greater than 6 inches in thickness.
Hand-operated power auger	Soil, 6 in.-15 ft.	Good depth range; generally used in conjunction with bucket auger for sample collection; destroys soil core (unsuitable for VOA and undisturbed sample collection); requires 2 or more equipment operators; can be difficult to decontaminate; requires gasoline-powered engine (potential for cross-contamination).

Sources: NJDEP, 1988; U.S. EPA, January 1991.

4.0 SAMPLE COLLECTION AND PREPARATION

4.1 INTRODUCTION

In addition to sampling equipment, sample collection includes sample quantity and sample volume. Sample preparation refers to all aspects of sample handling after collection, until the sample is received by the laboratory. Sample preparation for soils may include, but is not limited to:

- removing extraneous material;
- sieving samples;
- homogenizing samples;
- splitting samples;
- compositing samples; and
- final preparation.

Sample preparation depends on the sampling objectives and analyses to be performed. Proper sample preparation and handling help to maintain sample integrity. Improper handling can result in a sample becoming unsuitable for the type of analysis required. For example, homogenizing, sieving, and compositing samples all result in a loss of volatile constituents and are therefore inappropriate when volatile contaminants are the concern.

4.2 SAMPLE COLLECTION

How a sample is collected can affect its representativeness. The greater the number of samples collected from a site and the larger the volume of each sample, the more representative the analytical results will be. However, sampling activities are often limited by sampling budgets and project schedules. The following sections provide guidelines on appropriate sample numbers and volumes.

4.2.1 Sample Number

The number of samples needed will vary according to the particular sampling approach that is being used. For example, in grid sampling, one sample is generally collected at each grid node, regardless of grid size. As discussed in Section 2.11.6, once contaminated grid node samples are located, adjoining grid cells can be sampled more thoroughly to define areas of contamination. Four aliquots from each grid cell, situated equidistant from the sides of each cell and each other (as illustrated in Figure 10), are recommended for grid cells measuring up to 100 x 100 feet. One additional aliquot may be collected from the center of each cell, making a total of five aliquots per cell. For grid sizes greater than 100 feet x 100 feet, nine aliquots, situated equidistant from the sides of each cell and each other (as illustrated in Figure 13), are recommended. Depending on budget and other considerations, grid cell aliquots can be analyzed as separate samples or composited into one or more samples per cell.

4.2.2 Sample Volume

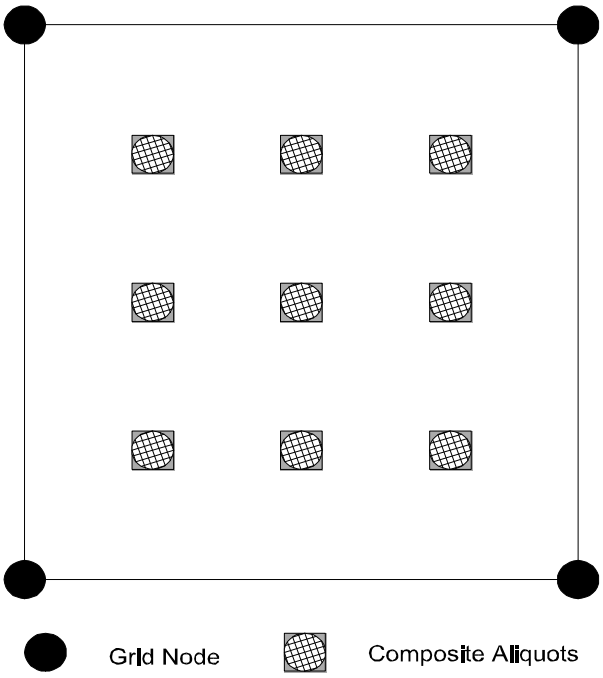
Both sample depth and area are considerations in determining appropriate sample volume. Depending on the analytes being investigated, samples are collected at the surface (0-3 in.), extended surface (0-6 in.), and/or at one-foot depth intervals. Non-water soluble contaminants such as dioxin and PCBs are often encountered within the first six inches of soil. Water-soluble contaminants such as metals, acids, ketones, and alcohols will be encountered at deeper depths in most soils except clays. Contaminants in solution, such as PCPs in diesel fuel and pesticides in solvents, can penetrate to great depths (e.g., down to bedrock), depending on soil type.

For surface samples, collect soil over a surface area of one square foot per sample. A square cardboard template measuring 12 in. x 12 in., or a round template with a 12 in. diameter can be used to mark sampling areas. For subsurface samples, one of several coring devices may be used (see Table 5). Using a coring device results in a smaller diameter sampling area than a surface template, and therefore somewhat lessens the representativeness of the sample.

4.3 REMOVING EXTRANEOUS MATERIAL

Identify and discard materials in a sample which are not relevant or vital for characterizing the sample or the site, since their presence may introduce an error in the sampling or analytical procedures. Examples of extraneous material in soil samples include pieces glass, twigs or leaves. However, not all non-soil material is extraneous. For example, when sampling at a junkyard, lead-contaminated battery casing pieces should not be removed from a sample if the casing composes more than 10% of the sample composition. For a sample to be representative, it must also incorporate the lead from the casing. Collect samples

Figure 13: Phase 2 Sampling Grid Cell Diagram (Grid Sizes > 100 x 100 ft.)



of any material thought to be a potential source of contamination for a laboratory extraction procedure. Discuss any special analytical requirements for extraneous materials with project management, geologists, and chemists and notify the laboratory of any special sample handling requirements.

4.4 SIEVING SAMPLES

Sieving is the process of physically sorting a sample to obtain uniform particle sizes, using sieve screens of predetermined size. For example, the sampler may wish to sieve a certain number of samples to determine if particle size is related to contaminant distribution. Sieving is generally only conducted when preparing soil samples for XRF screening. For this purpose, a 20-mesh screen size is recommended.

Be aware of the intent of the sampling episode, when deciding whether to sieve a sample prior to analysis. Prior to sieving, samples may need to be oven-dried. Discarding non-soil or non-sieved materials, as well as the sieving process itself, can result in physical and chemical losses. Sieving is not recommended where volatile compounds are of concern. Analyze the discarded materials, or a fraction thereof, to determine their contribution to the contamination of the site being investigated.

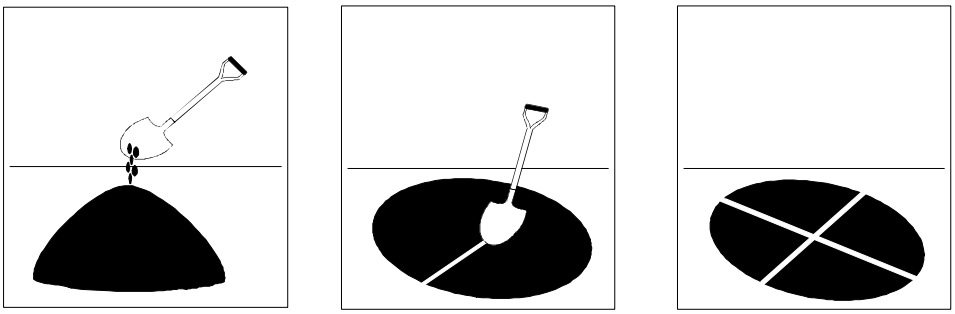
4.5 HOMOGENIZING SAMPLES

Homogenization is the mixing or blending of a soil sample in an attempt to provide uniform distribution of contaminants. (Do not homogenize samples for volatile compound analysis). Ideally, proper homogenization ensures that portions of the containerized samples are equal or identical in composition and are representative of the total soil sample collected. Incomplete homogenization will increase sampling error. All samples to be composited or split should be homogenized after all aliquots have been combined. Manually homogenize samples using a stainless steel spoon or scoop and a stainless steel bucket, or use a disposable scoop and pan. Quarter and split the sample as illustrated in Figure 14, repeating each step a minimum of 5 times until the sample is visually homogenized. Samples can also be homogenized using a mechanically-operated stirring device as depicted in ASTM standard D422-63.

4.6 SPLITTING SAMPLES

Splitting samples after collection and field preparation into two or more equivalent parts is performed when two or more portions of the same sample need to be analyzed separately. Split samples are most often collected in enforcement actions to compare sample results obtained by EPA with those obtained by the potentially responsible party (PRP). Split samples also provide a measure of the sample variability, and a measure of the analytical and extraction errors. Before splitting, follow homogenization techniques outlined above. Fill two sample collection jars simultaneously with alternate spoonfuls (or scoopfuls) of homogenized sample. To simultaneously homogenize and split a sample, **quarter** (as illustrated in Figure 14) or **mechanically** split the sample using a riffle sample splitter. The latter two techniques are described in detail in ASTM Standard C702-87.

Figure 14: Quartering to Homogenize and Split Samples



Step 1:

- Cone sample on hard, clean surface
- Mix by forming new cone

Step 2:

- Flatten cone
- Divide sample into quarters

Step 3: (not shown)

- Remix opposite quarters
- Reform cone
- Repeat a minimum of 5 times

4.7 COMPOSITING SAMPLES

Compositing is the process of physically combining and homogenizing several individual soil aliquots. Compositing samples provides an average concentration of contaminants over a certain number of sampling points, which reduces both the number of required lab analyses and the sample variability. Compositing can be a useful technique, but must always be implemented with caution. Compositing is not recommended where volatile compounds are of concern.

Specify the method of selecting the aliquots that are composited and the compositing factor in the sampling plan. The compositing factor is the number of aliquots to be composited into one sample (e.g., 3 to 1; 10 to 1). Determine this factor by evaluating detection limits for parameters of interest and comparing them with the selected action level for that parameter. Compositing also requires that each discrete aliquot be the same in terms of volume or weight, and that the aliquots be thoroughly homogenized. Since compositing dilutes high concentration aliquots, the applicable detection limits should be reduced accordingly. If the composite value is to be compared to a selected action level, then the action level must be divided by the number of aliquots that make up the composite in order to determine the appropriate detection limit (e.g., if the action level for a particular substance is 50 ppb, an action level of 10 ppb should be used when analyzing a 5-aliquot composite). The detection level need not be reduced if the composite area is assumed to be homogeneous in concentration (for example, stack emission plume deposits of particulate contamination across an area, or roadside spraying of waste oils).

4.8 FINAL PREPARATION

Select sample containers on the basis of compatibility with the material being sampled, resistance to breakage, and volume. For soil sampling, use wide-mouth glass containers with Teflon-lined lids. Appropriate sample volumes and containers will vary according to the parameter being analyzed. Keep low and medium concentration soil samples to be analyzed for organic constituents at 4EC. Actual sample volumes, appropriate containers, and holding times are specified in the *QA/QC Guidance for Removal Activities* (U.S. EPA, April 1990), in 40 CFR 136, and in the Compendium of ERT Soil Sampling and Surface Geophysics (U.S. EPA, January 1991). Package all samples in compliance with Department of Transportation (DOT) or International Air Transport Association (IATA) requirements.

It is sometimes possible to ship samples to the laboratory directly in the sampling equipment. For example, the ends of a Shelby tube can be sealed with caps, taped, and sent to the laboratory for analysis. To help maintain the integrity of VOA samples, collect soil cores using acetate sleeves and send the sleeves to the laboratory. To ensure the integrity of the sample after delivery to the laboratory, make laboratory sample preparation procedures part of all laboratory bid contracts.

4.9 EXAMPLE SITE



After placing each sample in a dedicated pan and mixing (as discussed in Section 3.5.1), plant matter, stones, and broken glass were removed. Soil samples were oven-dried (at 104E C) and sieved using a 20-mesh screen in preparation for XRF analysis. Samples were then homogenized and split using the quartering technique. Opposite quarters were remixed and quartering was repeated five times to ensure thorough homogenization. A portion of each sample was placed into XRF analysis cups for screening. The remainder of each sample was placed into 8-ounce, wide-mouth glass jars with Teflon-lined lids and sent to a laboratory for inorganic analysis. The samples were packaged in compliance with IATA requirements. Chain-of-custody paperwork was prepared for the samples. Laboratory paperwork was completed as appropriate and the samples were shipped to the predesignated laboratories for analysis.

5.0 QUALITY ASSURANCE/QUALITY CONTROL EVALUATION

5.1 INTRODUCTION

The goal of representative sampling is to collect samples which yield analytical results that accurately depict site conditions during a given time frame. The goal of quality assurance/quality control (QA/QC) is to identify and implement correct methodologies which limit the introduction of error into the sampling and analytical procedures, ultimately affecting the analytical data.

QA/QC samples evaluate the degree of site variation, whether samples were cross-contaminated during sampling and sample handling procedures, or if a discrepancy in sample results is due to laboratory handling and analysis procedures. The QA/QC sample results are used to assess the quality of the analytical results of waste and environmental samples collected from a site.

5.2 DATA CATEGORIES

EPA has established a process of data quality objectives (DQOs) which ensure that the precision, accuracy, representativeness, and quality of environmental data are appropriate for their intended application. Superfund DQO guidance defines two broad categories of analytical data: *screening* and *definitive*.

Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, rather than elaborate extraction/digestion and cleanup. At least 10 percent of the screening data are confirmed using the analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be data of known quality. To be acceptable, screening data must include the following: chain of custody, initial and continuing calibration, analyte identification, and analyte quantification. Streamlined QC requirements are the defining characteristic of screening data.

Definitive data are generated using rigorous analytical methods (e.g., approved EPA reference methods). These data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. QC measures for definitive data contain all of the elements associated with screening data, but also may include trip, method, and rinsate blanks; matrix spikes; performance evaluation samples; and replicate analyses for error determination.

For further information on these QA/QC objectives, please refer to EPA's *Quality Assurance/Quality Control Guidance for Removal Activities* or EPA's *Data Quality Objectives Process for Superfund*.

5.3 SOURCES OF ERROR

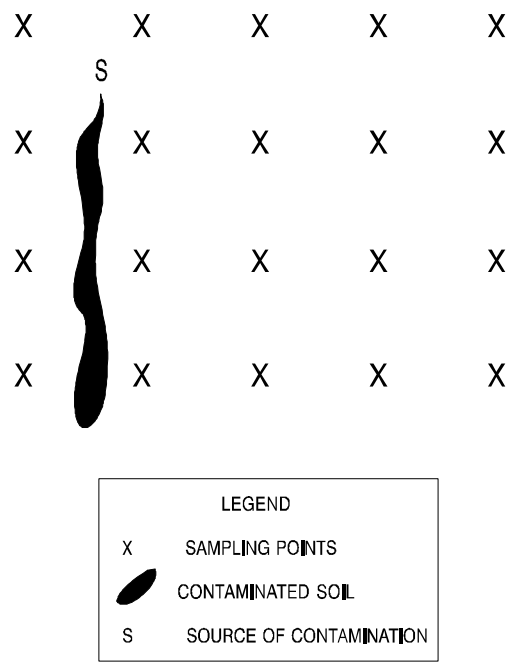
Identifying and quantifying the error or variation in sampling and laboratory analysis can be difficult. However, it is important to limit their effect(s) on the data. Four potential sources of error are:

- sampling design;
- sampling methodology;
- sample heterogeneity; and
- analytical procedures.

5.3.1 Sampling Design

Site variation includes the variation both in the types and in the concentration levels of contaminants throughout a site. Representative sampling should accurately identify and define this variation. However, error can be introduced by the selection of a sampling design which "misses" site variation. For example, a sampling grid with relatively large distances between sampling points or a biased sampling approach (i.e., judgmental sampling) may allow significant contaminant trends to go unidentified, as illustrated in Figure 15.

Figure 15: Sampling Error Due to Sampling Design



5.3.2 Sampling Methodology

Error can be introduced by the sampling methodology and sample handling procedures, as in cross-contamination from inappropriate use of sample collection equipment, unclean sample containers, improper sampling equipment decontamination and shipment procedures, and other factors. Standardized procedures for collecting, handling, and shipping samples allow for easier identification of the source(s) of error, and can limit error associated with sampling methodology. The use of standard operating procedures ensures that all sampling tasks for a given matrix and analyte will be performed in the same manner, regardless of the individual sampling team, date, or location of sampling activity. Trip blanks, field blanks, replicate samples, and rinsate blanks are used to identify error due to sampling methodology and sample handling procedures.

5.3.3 Sample Heterogeneity

Sample heterogeneity is a potential source of error. Unlike water, soil is rarely a homogeneous medium and it exhibits variable properties with lateral distance and with depth. This heterogeneity may also be present in the sample container unless the sample was homogenized in the field or in the laboratory. The laboratory uses only a small aliquot of the sample for analysis; if the sample is not properly homogenized, the analysis may not be truly representative of the sample and of the corresponding site. Thoroughly homogenizing samples, therefore, can limit error associated with sample heterogeneity.

5.3.4 Analytical Procedures

Error which may originate in analytical procedures includes cross-contamination, inefficient extraction, and inappropriate methodology. Matrix spike samples, replicate samples, performance evaluation samples, and associated quality assurance evaluation of recovery, precision, and bias, can be used to distinguish analytical error from error introduced during sampling activities.

5.4 QA/QC SAMPLES

This section briefly describes the types and uses of QA/QC samples that are collected in the field, or prepared for or by the laboratory. QA/QC samples are analyzed in addition to field samples and provide information on the variability and usability of environmental sample results. They assist in identifying the origin of analytical discrepancies to help determine how the analytical results should be used. They are used mostly to validate analytical results. Field replicate, collocated, background, and rinsate blank samples are the most commonly collected field QA/QC samples. Performance evaluation, matrix spike, and matrix spike duplicate samples, either prepared for or by the laboratory, provide additional measures of control for the data generated. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet site-specific quality assurance objectives. Refer to data validation procedures in U.S. EPA, April 1990, for guidelines on utilizing QA/QC analytical results. The following paragraphs briefly describe each type of QA/QC sample.

5.4.1 Field Replicates

Field replicates are field samples obtained from one location, homogenized, divided into separate containers and treated as separate samples throughout the remaining sample handling and analytical processes. These samples are used to assess error associated with sample heterogeneity, sample methodology and analytical procedures. Use field replicates when determining total error for critical samples with contamination concentrations near the action level. For statistical analysis to be valid in such a case, a minimum of eight replicate samples would be required.

5.4.2 Collocated Samples

Collocated samples are collected adjacent to the routine field sample to determine local variability of the soil and contamination at the site. Typically, collocated samples are collected about one-half to three feet away from the selected sample location. Analytical results from collocated samples can be used to assess site variation, but only in the immediate sampling area. Due to the non-homogeneous nature of soil at sites, collocated samples should not be used to assess variability across a site and are not recommended for assessing error. Determine the applicability of collocated samples on a site-by-site basis. Collecting many samples (more than 50 samples/acre), is sufficient to demonstrate site variation.

5.4.3 Background Samples

Background samples are collected upgradient of the area(s) of contamination (either on or off site) where there is little or no chance of migration of the contaminants of concern. Background samples determine the natural composition of the soil (especially important in areas with high concentrations of naturally-occurring metals) and are considered "clean" samples. They provide a basis for comparison of contaminant concentration levels with samples collected on site. At least one background soil sample should be collected; however, more are warranted when site-specific factors such as natural variability of local soil, multiple on-site contaminant source areas, and presence of off-site facilities potentially contributing to soil contamination exist. Background samples may be collected for all QA objectives, in order to evaluate potential error associated with sampling design, sampling methodology, and analytical procedures.

5.4.4 Rinsate Blanks

Rinsate blanks are samples obtained by running analyte-free water over decontaminated sampling equipment to test for residual contamination. The blank is placed in sample containers for handling, shipment, and analysis identical to the samples collected that day. A rinsate blank is used to assess cross-contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank, per type of sampling device, per day.

5.4.5 Performance Evaluation Samples

Performance evaluation (PE) samples evaluate the overall bias of the analytical laboratory and detect any error in the analytical method used. These samples are usually prepared by a third party, using a quantity of analyte(s) which is known to the preparer but unknown to the laboratory, and always undergo certification analysis. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. Laboratory procedural error is evaluated by the percentage of analyte identified in the PE sample (percent recovery). Even though they are not available for every single analyte, analysis of PE samples is required to obtain definitive data.

5.4.6 Matrix Spike Samples

Matrix spike and matrix spike duplicate samples (MS/MSDs) are environmental samples that are spiked in the laboratory with a known concentration of a target analyte(s) to verify percent recoveries. MS/MSDs are primarily used to check sample matrix interferences. They can also be used to monitor laboratory performance. However, a dataset of at least three or more results is necessary to distinguish between laboratory performance and matrix interference.

MS/MSDs can also monitor method performance. Again, a dataset is helpful to assess whether a method is performing properly. Generally, interference and poor method performance go together.

MS/MSDs can also evaluate error due to laboratory bias and precision (when four or more pairs are analyzed). Analyze one MS/MSD pair to assess bias for every 20 soil samples. Use the average percent recovery for the pair. To assess precision, analyze at least 8 matrix spike replicates from the same sample, determine the standard deviation and the coefficient of variation. See pages 9 - 10 of the *QA/QC Guidance*

for Removal Activities (U.S. EPA, April 1990) for procedures on calculating analytical error. MS/MSDs are optional when the goal is to obtain screening data and required to obtain definitive data as one of several methods to determine analytical error.

5.4.7 Field Blanks

Field blanks are samples prepared in the field using certified clean sand or soil and are then submitted to the laboratory for analysis. A field blank is used to evaluate contamination error associated with sampling methodology and laboratory procedures. If available, submit field blanks at a rate of one per day.

5.4.8 Trip Blanks

Trip blanks are samples prepared prior to going into the field. Trip blanks consist of certified clean sand or soil and are handled, transported, and analyzed in the same manner as the other volatile organic samples acquired that day. Trip blanks are used to evaluate error associated with sampling methodology and analytical procedures by determining if any contamination was introduced into samples during sampling, sample handling and shipment, and/or during laboratory handling and analysis. If available, utilize trip blanks for volatile organic analyses.

5.5 EVALUATION OF ANALYTICAL ERROR

The percentage and types of QA/QC samples needed to help identify the error and confidence in the data is based on the sampling objectives and the corresponding QA/QC objectives. The acceptable level of error is determined by the intended use of the data and the sampling objectives, including such factors as: the degree of threat to public health, welfare, or the environment; selected action levels; litigation concerns; and budgetary constraints.

The use of replicate samples is one method to evaluate error. To evaluate the total error of samples with contaminant concentrations near the selected action level, prepare and analyze a minimum of eight replicates of the same sample. Analytical data from replicate samples can also be used for a quick check on errors associated with sample heterogeneity, sample methodology and analytical procedures. Differing analytical results from two or more replicate samples could indicate improper sample preparation (e.g., incomplete homogenization), or that contamination was introduced during sample collection, preparation, handling, shipment, or analysis.

It may be desirable to try to quantify confidence; however, quantification or analytical data correction is not always possible. A 95% confidence level (i.e., 5% acceptable error) should be adequate for most sampling activities. Experience will provide the best determination of whether to use a higher (e.g., 99%) or lower (e.g., 90%) level of confidence. It must be recognized that the use of confidence levels is based on the assumption that a sample is homogeneous. See also Section 6.8 for information on total error.

5.6 CORRELATION BETWEEN SCREENING RESULTS AND DEFINITIVE RESULTS

One cost-effective approach for delineating the extent of site contamination is to correlate inexpensive screening data and other field measurements (e.g., XRF, soil-gas measurements) with laboratory results. The relationship between the two methods can then be described by a regression analysis and used to predict laboratory results based on screening measurements. In this manner, cost-effective screening results may be used in addition to, or in lieu of, off-site laboratory sample analysis.

Statistical regression involves developing a model (equation) that relates two or more variables at an acceptable level of correlation. When screening techniques, such as XRF, are used along with laboratory methods (e.g., atomic absorption (AA)), a regression equation can be used to predict a laboratory value based on the results of the screening device. The model can also be used to place confidence limits around predictions. Additional discussion of correlation and regression can be found in most introductory statistics textbooks. A simple regression equation (e.g., linear) can be developed on many calculators or computer databases; however, a statistician should be consulted to check the accuracy of more complex models.

Evaluation of the accuracy of a model in part relies on statistical correlation. Statistical correlation involves computing an index called the correlation coefficient (r) that indicates the degree and nature of the relationship between two or more sets of values. The correlation coefficient ranges from !1.0 (a perfect inverse or negative relationship), through 0 (no relationship), to +1.0 (a perfect direct, or positive, relationship). The square of the correlation coefficient, called the coefficient of determination, or simply R^2, is an estimate of the proportion of variance

in one variable (the dependent variable) that can be accounted for by the independent variables. The R^2 value that is acceptable depends on the sampling objectives and intended data uses. As a rule of thumb, statistical relationships should have an R^2 value of at least 0.6 to determine a reliable model; however, for health or risk assessment purposes, the acceptable R^2 value may be made more stringent (e.g., 0.8). Analytical calibration regressions have an R^2 value of 0.98 or better.

Once a reliable regression equation has been derived, the screening data can be used to predict laboratory results. These predicted values can then be located on a base map and contoured (mapping methods are described in Chapter 6). These maps can be examined to evaluate the estimated extent of contamination and the adequacy of the sampling program.

5.7 EXAMPLE SITE



The screening of containerized liquid wastes was performed to quickly obtain data indicating general chemical class. Definitive analysis was run on 10% of the samples in order to verify screening results. The definitive analyses provided were analyte and concentration specific. Recoveries of matrix spike and matrix spike duplicate samples indicated no matrix interferences. Dedicated equipment was used during Phase 1 sampling, making rinsate blanks unnecessary. Phase 2 screening was performed using XRF. During Phase 2, samples were collected at 30% of the nodes screened with the XRF. These samples were sent for laboratory AA analysis. A correlation was established by plotting the Phase 2 AA and XRF data. This allowed the XRF data from the other 70% of the nodes to be used to evaluate the chromium levels across the site.

For Phase 2 and 3 sampling, 10% of the data were confirmed by running replicate analyses to obtain an estimate of precision. The results indicated good correlation. Matrix spikes and matrix spike duplicate samples indicated no matrix interferences. During Phase 2, the OSC included performance evaluation (PE) samples for metals to evaluate the overall laboratory bias. The laboratory achieved 92% recovery, which was within the acceptable control limits.

During Phases 2 and 3, a rinsate blank was collected each day. Following the decontamination of the bucket augers, analyte-free water was poured over the augers and the rinsate was placed into 1-liter polyethylene bottles and preserved. The rinsate blanks were analyzed for total metals and cyanide to determine the effectiveness of the decontamination procedures and the potential for cross-contamination. All rinsate blank samples were "clean", indicating sufficient decontamination procedures.

The correlation analysis run on Phase 2 laboratory (AA) data and corresponding XRF values resulted in r values of 0.97 for both surface and subsurface data, which indicated a strong relationship between the AA and XRF data. Following the correlation analyses, regression analyses were run and equations to predict laboratory values based on the XRF data were developed. The resulting equation for the surface data was: AA = 0.87 (XRF) + 10.16. The resulting regression equation for the subsurface data was: AA = 0.94 (XRF) + 0.30.

6.0 DATA PRESENTATION AND ANALYSIS

6.1 INTRODUCTION

Data presentation and analysis techniques are performed with analytical, geophysical, or screening results. The techniques discussed below can be used to compare analytical values, to evaluate numerical distribution of data, to determine and illustrate the location of hot spots and the extent of contamination across a site, and to assess the need for removal of contaminated soil with concentrations at or near the action level. The appropriate methods to present and analyze sample data depend on the sampling objectives, the number of samples collected, the sampling approaches used, and a variety of other considerations.

6.2 DATA POSTING

Data posting involves placement of sample values on a site basemap. Data posting is useful for displaying the spatial distribution of sample values to visually depict extent of contamination and to locate hot spots. Data posting requires each sample to have a specific location (e.g., X and Y coordinates). Ideally, the sample coordinates would be surveyed values to facilitate placement on a scaled map.

6.3 GEOLOGIC GRAPHICS

Geologic graphics include cross-sections and fence diagrams, which are two- and three-dimensional depictions, respectively, of soils and strata to a given depth beneath the site. These types of graphics are useful for posting subsurface analytical data as well as for interpreting subsurface geology and contaminant migration.

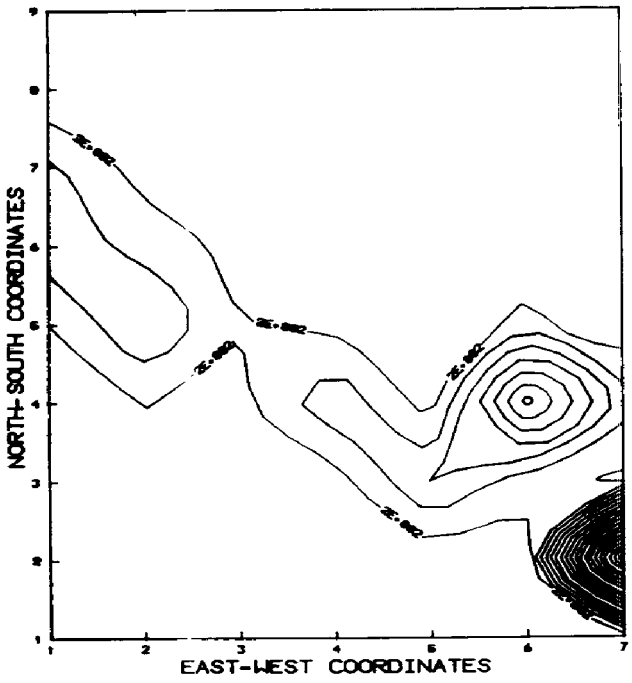
6.4 CONTOUR MAPPING

Contour maps are useful for depicting contaminant concentration values throughout a site. Contour mapping requires an accurate, to-scale basemap of the site. After data posting sample values on the basemap, insert contour lines (or isopleths) at a specified contour interval, interpolating values between sample points. Contour lines can be drawn manually or be generated by computer using contouring software. Although the software makes the contouring process easier, computer programs have a limitation: they may interpolate between all data points, attempting to fit a contour interval to the full range of data values. This can result in a contour map that does not accurately represent general site contaminant trends. Typical emergency or early action sites have low concentration/non-detect areas and hot spots. Computer contouring programs may represent these features as in Figure 16 which illustrates a site that has a 4000 mg/kg hot spot. Because there is a large difference in concentration between the hot spot and the surrounding area, the computer contouring program used a contour interval that eliminated most of the subtle site features and general trends. However, if that same hot spot concentration value is posted at a reduced value, then the contouring program can select a more appropriate contour interval to better illustrate the general site trends. Figure 17 depicts the same site as in Figure 16, but the hot spot concentration value has been arbitrarily posted at 1400 mg/kg. The map was recontoured and the contouring program selected a contour interval that resulted in a map which enhanced the subtle detail and general site contaminant trends.

6.5 STATISTICAL GRAPHICS

The distribution or spread of the data set is important in determining which statistical techniques to use. Common statistical analyses such as the t-test relies on normally distributed data. The histogram is a statistical bar graph which displays the distribution of a data set. A normally distributed data set takes the shape of a bell curve, with the mean and median close together about halfway between the maximum and minimum values. A probability plot depicts cumulative percent against the concentration of the contaminant of concern. A normally distributed data set, when plotted as a probability plot, would appear as a straight line. Use a histogram or probability plot to see trends and anomalies in the data prior to conducting more rigorous forms of statistical analysis.

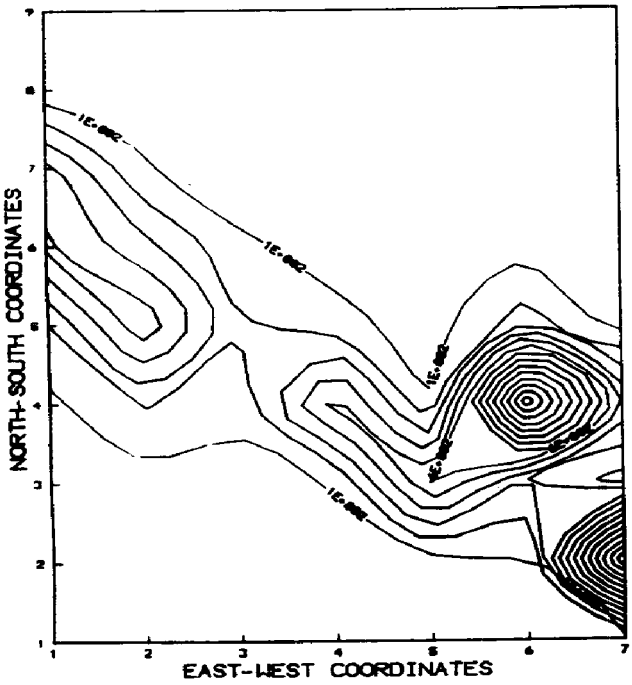
Figure 16: Computer-Generated Contour Map (4000 mg/kg Hot Spot)
ABC Plating Site



Total Chromium Concentration
Units = mg/kg
Contour Interval = 100 mg/kg

Includes 4000 mg/kg Hot Spot

Figure 17: Computer-Generated Contour Map (1400 mg/kg Hot Spot)
ABC Plating Site



Total Chromium Concentration
Units = mg/kg
Contour Interval = 100 mg/kg

Includes 1400 mg/kg Hot Spot*

* 1400 mg/kg hot spot is substituted for 4000 mg/kg hot spot

6.6 GEOSTATISTICS

Geostatistical methods are useful for data analysis and presentation. The characteristic feature of geostatistics is the use of variograms to quantify and model the spatial relationship between values at different sampling locations and for interpolating (e.g., kriging) estimated values across a site. The geostatistical analysis can be broken down into two phases. First, a model is developed that describes the spatial relationship between sample locations on the basis of a plot of spatial variance versus the distance between pairs of samples. This plot is called a variogram. Second, the spatial relationship modeled by the variogram is used to compute a weighted-average interpolation of the data. The result of geostatistical mapping by data interpolation is a contour map that represents estimates of values across a site, and maps depicting potential error in the estimates. The error maps are useful for deciding if additional samples are needed and for calculating best or worst-case scenarios for site cleanup. More information on geostatistics can be found in U.S. EPA, September 1988b and U.S. EPA, 1990. Geo-EAS and GEOPACK, geostatistical environmental assessment software packages developed by U.S. EPA, can greatly assist with geostatistical analysis methods.

6.7 RECOMMENDED DATA INTERPRETATION METHODS

The data interpretation method chosen depends on project-specific considerations, such as the number of sampling locations and their associated range in values. A site depicting extremely low data values (e.g., non-detects) with significantly higher values (e.g., 5,000 ppm) from neighboring hot spots, with little or no concentration gradient in-between, does not lend itself to contouring and geostatistics, specifically the development of variograms. However, data posting would be useful at such a site to illustrate hot spot and clean areas. Conversely, geostatistics and contour mapping, as well as data posting, can be applied to site data with a wide distribution of values (i.e., depicting a "bell shaped" curve) with beneficial results.

6.8 UTILIZATION OF DATA

When conducting search sampling to determine the locations of hot spots (as discussed in Section 2.9), analyze the data using one of the methods discussed in this chapter. For each node that is determined to be close to or above the action level, the following procedure is recommended.

Investigate all neighboring grid cells to determine which areas must be excavated and/or treated. From each grid cell, take a composite sample consisting of four or more aliquots, using the procedure described in Section 2.11.6. Grid cells with contaminant concentrations significantly above the action level (e.g., 20%) should be marked for removal. Grid cells with contaminant concentrations significantly less than the action level should be designated as clean. For grid cells with contaminant concentrations close to the action level, it is recommended that additional sampling be done within that grid cell to determine whether it is truly a hot spot, or whether the analytical result is due to sampling and/or analytical procedural error. If additional sampling is to be performed, one of the following methods should be considered:

- Collect a minimum of four grab samples within the grid cell in question. Use these samples to develop a 95% confidence interval around the mean concentration. If the action level falls within or below this confidence interval, then consider removal/treatment of the soil within that grid cell. More information on confidence intervals and standard deviation can be found in Gilbert, 1987.

- Collect additional composite samples from the grid cells in question using the technique discussed in Section 2.11.6. From these additional samples, determine the need for removal/treatment.

These two practical approaches help to determine the total error associated with collecting a sample from a non-homogeneous site. Total error includes design error, sampling error, non-homogeneous sampling error, and analytical error.

If additional sampling is being considered, weigh the cost-effectiveness of collecting the additional samples versus removing the soil from the areas in question. This decision must be made on a site-by-site basis.

After removal/treatment of the contaminated soil, re-investigate the grid cells to verify cleanup below the action level. Each grid cell that had soil removed must either be composite sampled again, or have multiple grab samples collected with a 95% confidence interval set up again. Again, this decision must be made on a site-by-site basis. The methodology should be repeated until all grid cells are determined to have soil concentrations below the action level.

6.9 EXAMPLE SITE



The Phase 2 XRF/atomic absorption (AA) data were examined to determine the appropriate data interpretation method to use. A histogram was generated to illustrate the distribution of the data as depicted in Figure 18. The histogram showed an uneven distribution of the data with most values less than 50 (approximately 4 on the LN scale of the histogram). Also, the presence of a single data point of 4000 (8 on the LN scale) was shown on the histogram. The data were initially posted as illustrated in Figures 19 and 20. Data posting was performed manually to give the OSC a quick depiction of the general site contamination trends. A contour mapping program was used to generate contours based on the posted data. Figure 16 illustrates the results of contouring with the 4000 mg/kg hot spot included. This contour map exaggerated the hot spot while eliminating the subtle site features and contaminant trends. Figure 17 depicts the same site data with the hot spot arbitrarily reduced to 1400 mg/kg. The resulting contour map enhanced more of the subtle site features and trends while reducing the effects of the hot spot.

AA concentrations predicted by the regression equations were kriged and contoured using Geo-EAS (Figures 21 and 22). Both the kriged contours and the data posting showed the same general site contaminant trends. However, data posting gave a more representative depiction of actual levels of contamination and the OSC used data posting for decision-making.

For each node with chromium concentrations close to or above the 100 ppm action level, the adjacent grid cells were further investigated. Composite samples consisting of four aliquots of soil were taken from within each grid cell in question and analyzed. If the soil concentration level was significantly below 100 ppm of chromium, the cell was designated as clean. Each cell that had a soil concentration level well above the action level was marked for removal/treatment. Any cells having soil concentrations close to the action level were sampled further using the compositing method to better quantify the actual contaminant concentration. Since the surrounding area is residential, on-site landfilling was not considered a viable treatment option. To expedite treatment/disposal, all excavated soil from contaminated cells was stockpiled on site until treatment/disposal could be accomplished under a fixed-price contract. The stockpile, placed in the area of the most highly contaminated grid cells (where the lagoons were located), was covered until treatment/disposal could be arranged. Cleanup was verified with composite sampling in the excavated cells. Results of the composite sampling were compared with the action level to verify cleanup. All action levels were met. The excavation pits were filled with stone and clean soil, covered with topsoil, graded and seeded.

Figure 18: Histogram of Surface Chromium Concentrations
ABC Plating Site

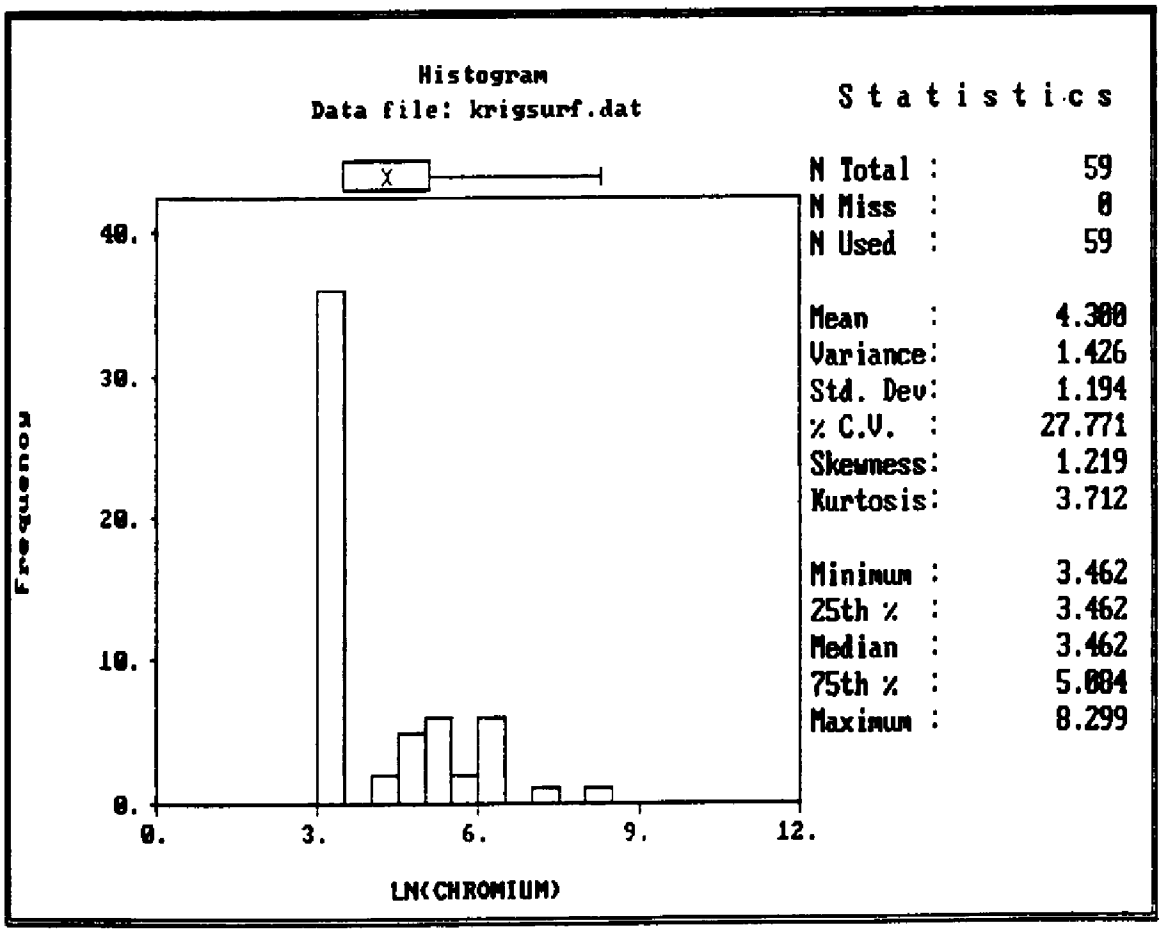


Figure 19: Phase 2 Surface Data Posting for Chromium
ABC Plating Site

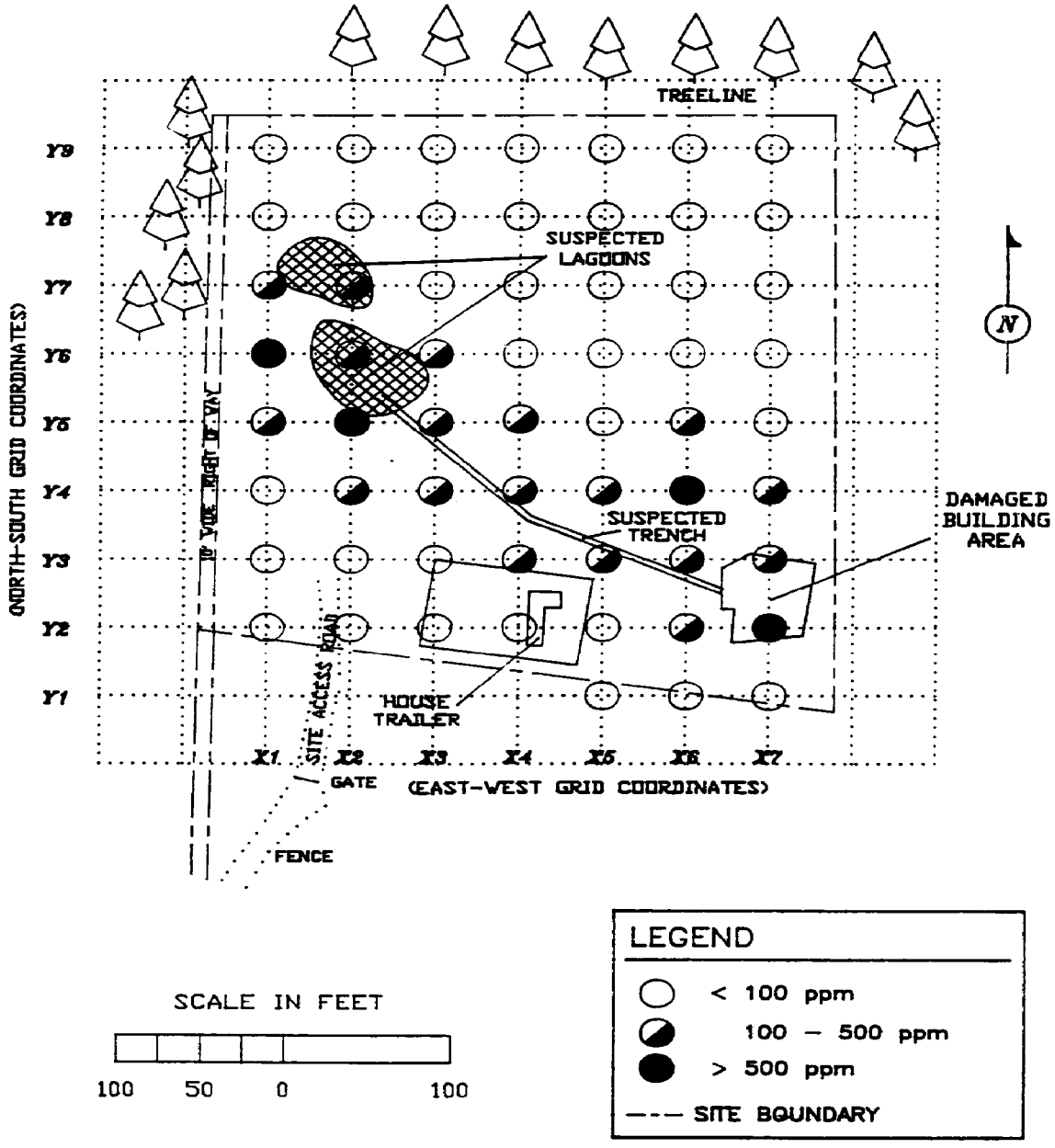


Figure 20: Phase 2 Subsurface Data Posting for Chromium
ABC Plating Site

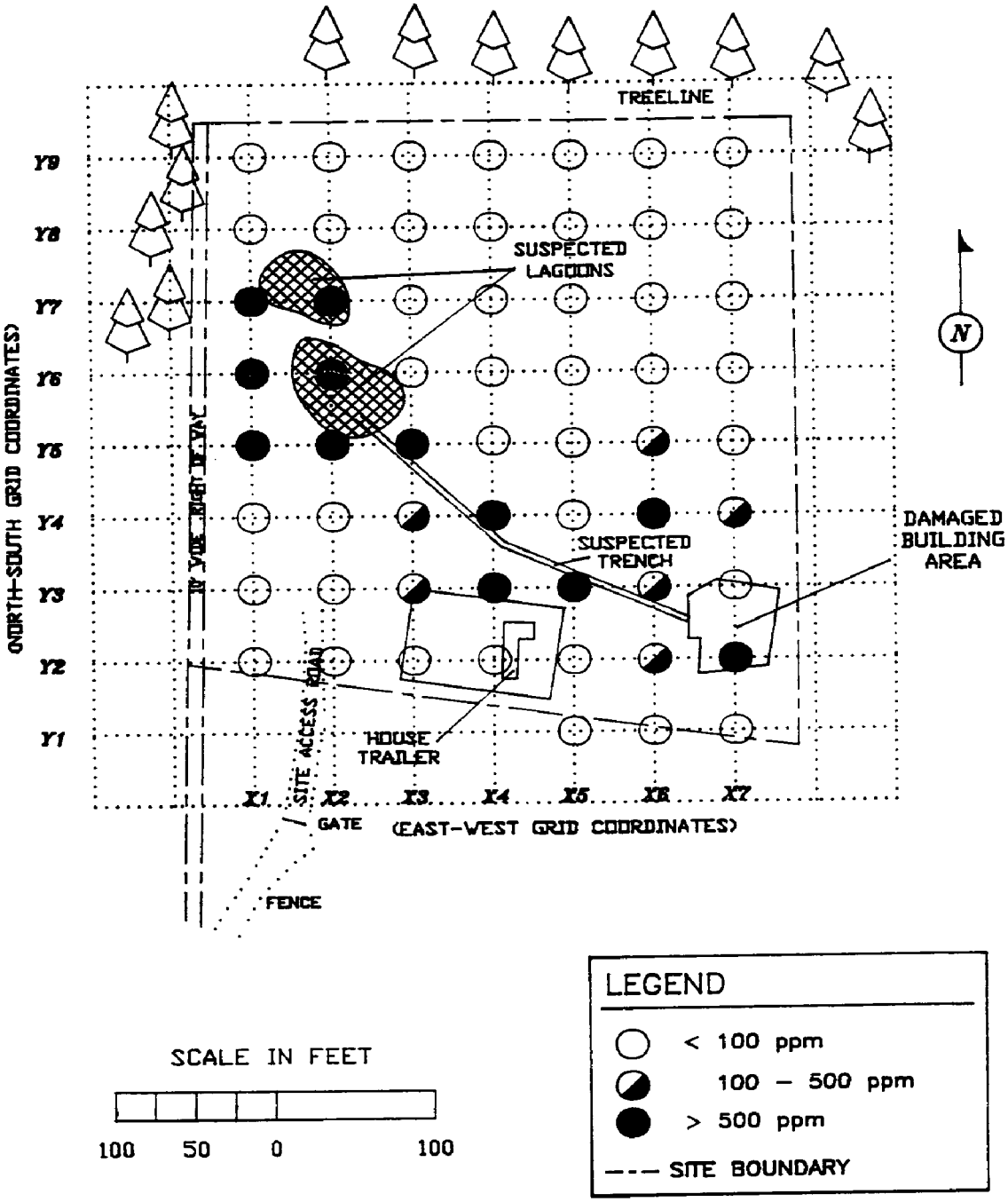


Figure 21: Contour Map of Surface Chromium Data (ppm)
ABC Plating Site

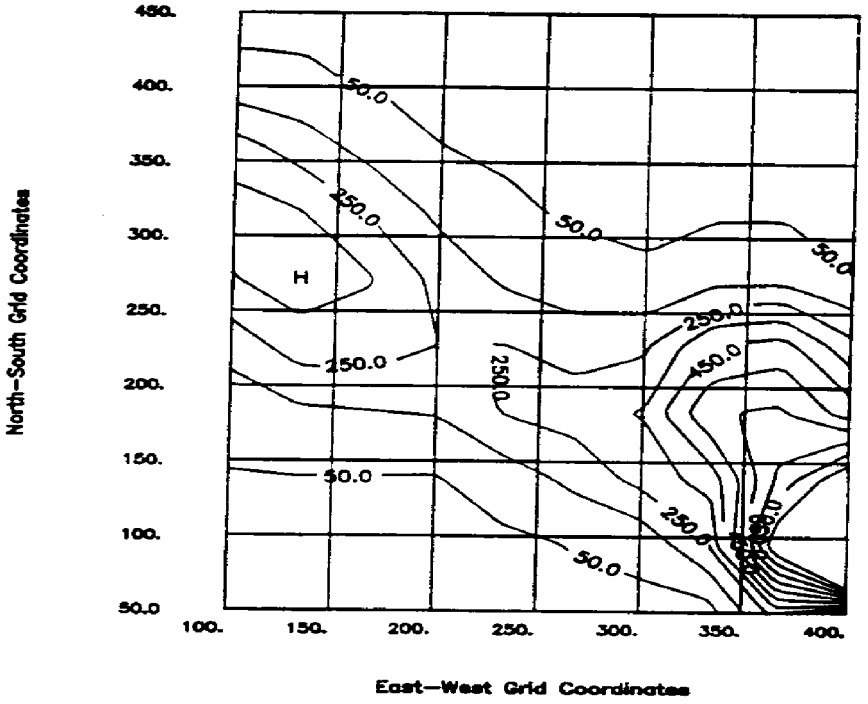
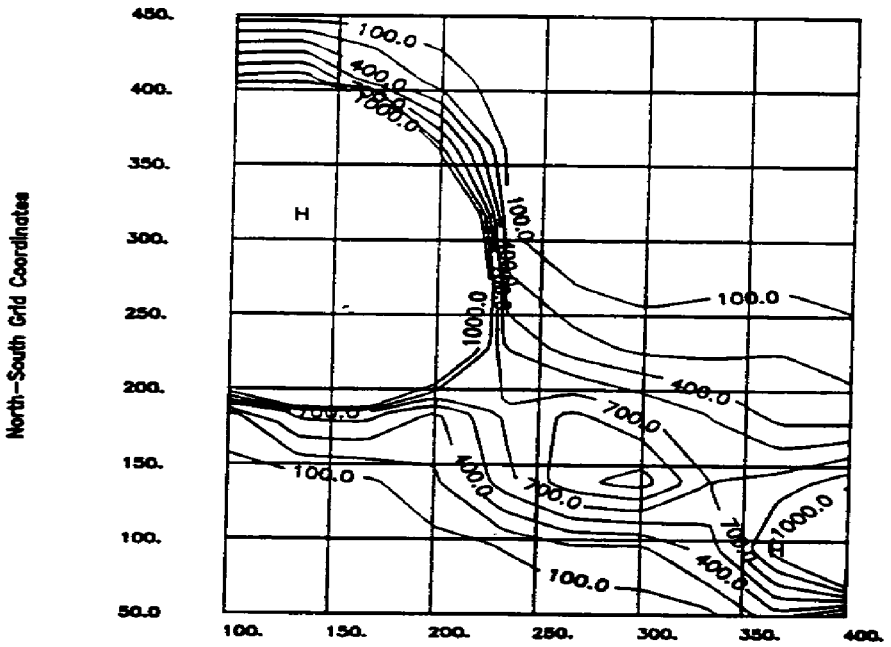
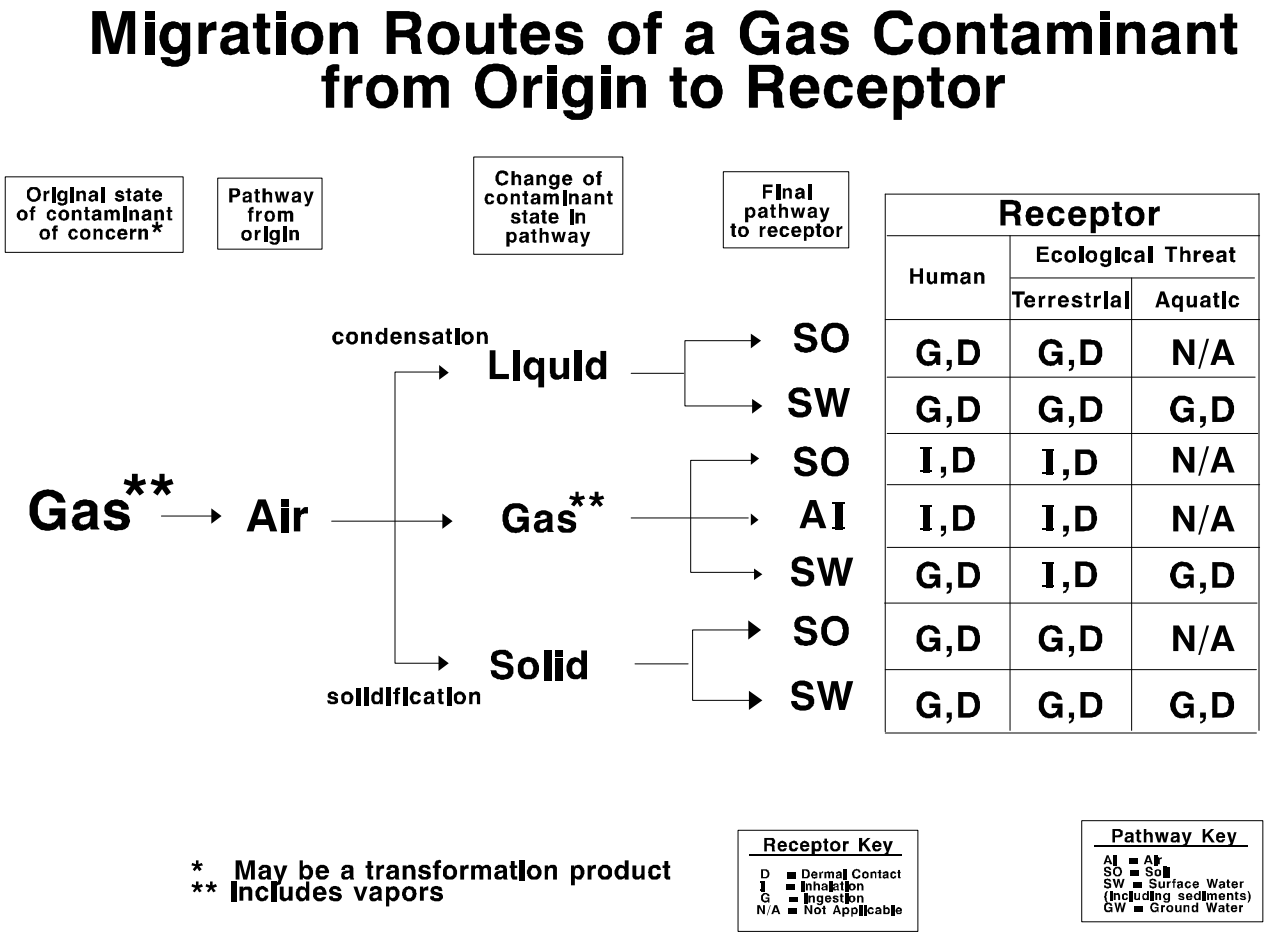


Figure 22: Contour Map of Subsurface Chromium Data (ppm)
ABC Plating Site



APPENDIX A -- EXAMPLE OF FLOW DIAGRAM FOR CONCEPTUAL SITE MODEL

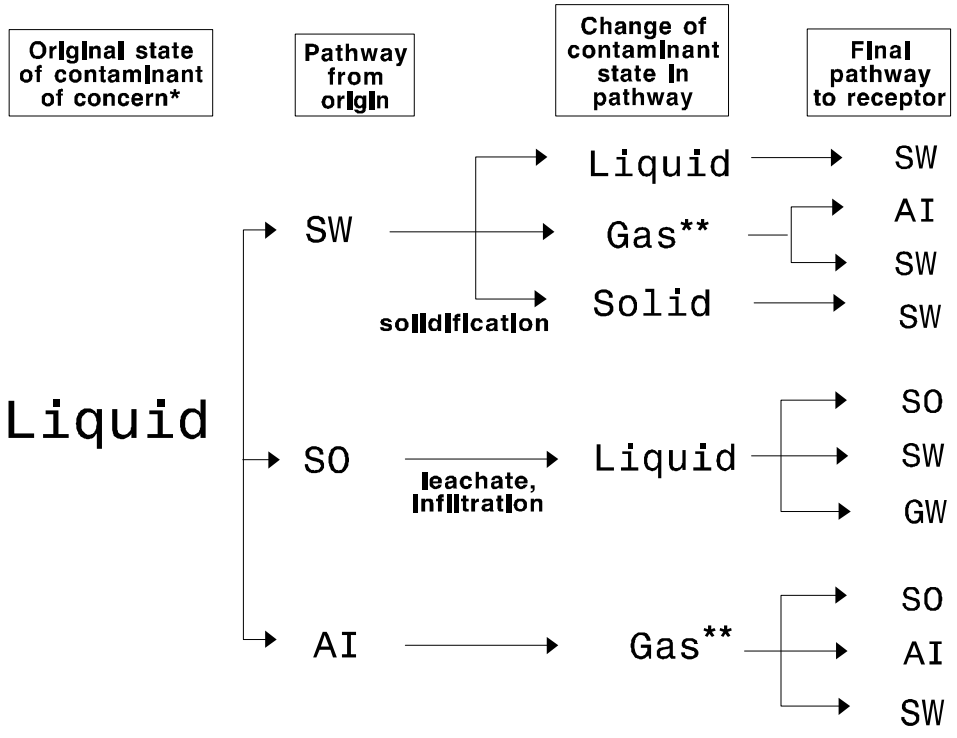
Figure A-1



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
G,D	G,D	N/A
G,D	G,D	G,D
I,D	I,D	N/A
I,D	I,D	N/A
G,D	I,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D

Figure A-2

Migration Routes of a Liquid Contaminant from Origin to Receptor



Original state of contaminant of concern*	Pathway from origin	Change of contaminant state in pathway	Final pathway to receptor	Receptor: Human	Receptor: Ecological Threat: Terrestrial	Receptor: Ecological Threat: Aquatic
Liquid	SW	Liquid	SW	G,D	G,D	G,D
		Gas**	AI	I,D	I,D	N/A
			SW	G,D	G,D	G,A
		Solid (solidification)	SW	G,D	G,D	G,D
	SO	Liquid (leachate, infiltration)	SO	G,D	G,D	N/A
			SW	G,D	G,D	G,D
			GW	G,D	N/A	N/A
	AI	Gas**	SO	G,D	G,D	N/A
			AI	I,D	I,D	N/A
			SW	G,D	G,D	G,D

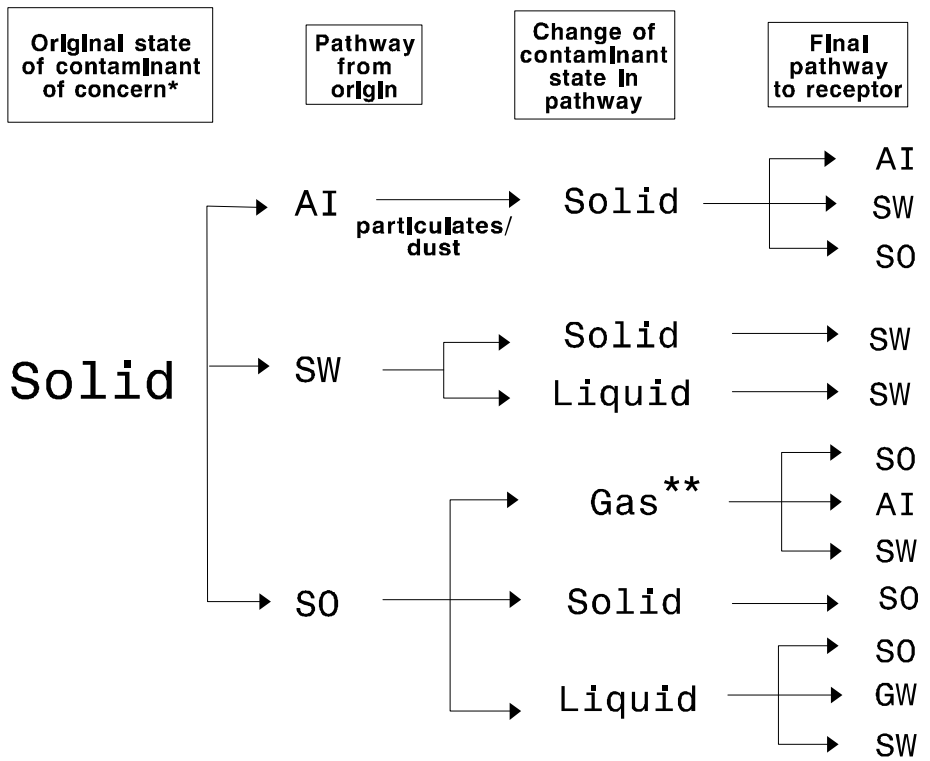
* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

Figure A-3

Migration Routes of a Solid Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D
G,D	G,D	G,D
G,D	G,D	N/A
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	N/A
G,D	N/A	N/A
G,D	G,D	G,D

* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

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PBxx-xxxxxx
December 1995

SUPERFUND PROGRAM

REPRESENTATIVE SAMPLING GUIDANCE

VOLUME 5: WATER AND SEDIMENT

PART II -- Ground Water

Interim Final

Environmental Response Team

Office of Emergency and Remedial Response
Office of Solid Waste and Emergency Response

U.S. Environmental Protection Agency
Washington, DC 20460

Notice

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication.

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For more information on Ground-Water Sampling procedures, refer to the U.S. EPA *Compendium of ERT Ground-Water Sampling Procedures*, OSWER Directive 9360.4-06. Topics covered in this compendium include: sampling equipment decontamination; ground-water monitoring well installation, development, and sampling; soil gas sampling; water level measurement; controlled pump testing; slug testing.

Please note that the procedures in this document should be used only by individuals properly trained and certified under a 40-hour hazardous waste site training course that meets the requirements set forth in 29 CFR 1910.120(e)(3). This document should not be used to replace or supersede any information obtained in a 40-hour hazardous waste site training course.

Questions, comments, and recommendations are welcomed regarding the *Superfund Program Representative Sampling Guidance, Volume 5 -- Water and Sediment, Part II -- Ground Water*. Send remarks to:

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1.0 INTRODUCTION

1.1 OBJECTIVE AND SCOPE

This is Part II of the fifth volume in a series of guidance documents that assist Superfund Program Site Managers, On-Scene Coordinators (OSCs), Remedial Project Managers (RPMs), and other field staff in obtaining representative samples at Superfund sites. The objective of representative sampling is to ensure that a sample or a group of samples accurately characterizes site conditions.

Most hazardous waste site investigations utilize some form of a ground-water sampling or monitoring program to fully characterize the nature and extent of contamination. Because site conditions may differ, experienced hydrogeologists and geochemists should be consulted to establish the most suitable types of sampling and monitoring for each site.

The purpose of this document is to address representative ground-water sampling. Ground-water modeling and monitoring well installation are briefly introduced but are not addressed in detail in this document. References on these topics are provided in Section 1.6

The representative ground-water sampling principles discussed in this document are applicable throughout the Superfund Program. The following chapters will help field personnel to assess available information, select an appropriate sampling approach and design, select and utilize field analytical/geophysical screening methods and sampling equipment, incorporate suitable types and numbers of quality assurance/quality control (QA/QC) samples, and interpret and present the site analytical data.

As the Superfund Program has developed, the emphasis of the response action has expanded beyond addressing emergency response and short-term cleanups. Each planned response action must consider a variety of sampling objectives, including identifying threat, delineating sources of contamination, and confirming the achievement of clean-up standards. Because many important and potentially costly decisions are based on the sampling data, Site Managers and other field personnel must characterize site conditions accurately. To that end, this document emphasizes the use of cost-effective field analytical and geophysical screening techniques to characterize the site and aid in the selection of sampling locations.

1.2 UNIQUE CHARACTERISTICS OF GROUND WATER

The following are media-specific variables of ground water that should be considered when performing representative ground-water sampling:

- Homogeneity - Ground water, as a medium, is usually homogeneous, especially when compared to other media such as soil, air, or waste.

- Seasonal and Localized Variation in Flow - Seasonal and localized variations in ground-water flow should be considered when developing a ground-water assessment program. Seasonal variations are generally controlled by weather. Surface streams gain or lose water to the subsurface when flood or drought conditions are present. Localized variations in flow are caused by nearby, outside influences, as when a production well creates a cone of depression in the water table.

- Inaccessibility for Investigation - Ground water is often inaccessible to standard grab sampling techniques. Because ground water is subsurface, wells must often be drilled and completed for sampling if no existing wells are available. Sampling ground water is generally more complicated, labor-intensive, time-consuming, and expensive than sampling other media.

- Natural Background Composition - Knowledge of the natural background composition is necessary in order to determine the effects of a site on the ground water. Background or control monitoring wells are necessary to determine ambient composition.

- Water Treatment - Ground-water samples are often extracted from existing residential or commercial wells that have been treated with softeners or have been filtered or altered in other ways. Sampling (times, parameters, methods, preservatives, etc.) may have to be altered in order to compensate for or avoid treatment variables.

- Reproducibility of Sampling Results - Ground water is a flowing water body below the earth's surface. Physical and chemical characteristics may vary over time and space because of the

factors listed above (e.g., seasonal variation). Contaminants tend to flow through ground water in a plume or plug of varying concentration; contamination sources may discharge in pulses or as a continuous flow; and contaminants may react with ground water to chemically transform over time. Because of this flowing nature, contaminant or natural constituent concentrations can vary. This variation could affect duplicating sample results over an extended time period. Contaminants will most often continue to be detected in ground water, but sample concentration ranges may be altered, either by an increase or a decrease, or contaminant by-products may be detected.

1.3 REPRESENTATIVE SAMPLING

Representative ground-water sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration throughout a site.

In addition to the variables introduced due to the characteristics of the sample media (as discussed in Section 1.2), this document concentrates on those that are introduced in the field. These latter variables relate to site-specific conditions, the sampling design approach, and the techniques for collection and preparation of samples. The following variables affect the representativeness of samples and subsequent measurements:

- Media variability - The physical and chemical characteristics of ground water.

- Contaminant concentration variability - Variations in the contaminant concentrations throughout the site and/or variables affecting the release of site contaminants into ground water on or away from the site.

- Collection and preparation variability - Deviations in analytical results attributable to bias introduced during sample collection, preparation, and transportation (for analysis).

- Analytical variability - Deviations in analytical results attributable to the manner in which the sample was stored, prepared, and analyzed by the on-site or off-site laboratory. Although analytical variability cannot be corrected through representative sampling, it can lead to the false conclusion that error is due to sample collection and handling procedures.

1.4 REPRESENTATIVE SAMPLING OBJECTIVES

Representative sampling objectives for ground water include the following:

- Identify the presence of contamination, including source, composition, and characteristics. Determine if it is hazardous.

- Establish the existence of an imminent or substantial threat to public health or welfare or to the environment.

- Establish the existence of potential threat requiring long-term actions.

- Develop containment and control strategies.

- Evaluate treatment options.

Note: Clean-up goals are generally established for ground water and are not considered a sampling objective.

1.4.1 Identify Contamination and Determine Hazard

One of the first objectives during a response action at a site is to determine the presence, identity, and potential threat of any hazardous materials. Field screening techniques can be used for rapid detection of contaminants. Upon confirming the presence of hazardous materials, sample and/or continue screening to identify their compositions and determine their concentrations.

1.4.2 Establish Imminent or Substantial Threat

Establishing threat to the public or environment is a primary objective during any response action. The data obtained from characterizing the contaminants will help the Site Manager to determine whether an imminent or substantial threat exists and whether a response action is necessary. The type and degree of threat determines the rate at which a response action is taken.

1.4.3 Determine Long-Term Threat

Site conditions may support a long-term threat that is not imminent or substantial. Characterization of the contaminants can assist the Site Manager to determine the need for long-term remediation and response. Samples should be collected in a manner that enables their use for evaluating the site under the Hazard Ranking System.

1.4.4 Develop Containment and Control Strategies

Once the chemical constituents and threat have been determined, many strategies for ground-water containment and control are available. Analytical data indicating the presence of chemical hazards are not in themselves sufficient to select a containment or control strategy. Site reconnaissance and historical site research provide information on site conditions and the physical state of the contaminant sources; containment and control strategies are largely determined by this information. For example, trenching and pump and treat systems can prevent spread of contamination in an aquifer.

1.4.5 Evaluate Treatment Options

The contaminants should be identified, quantified, and compared to action levels (e.g., maximum contaminant levels (MCLs) for drinking water). Where regulatory action levels do not exist, site-specific clean-up levels are determined by the Region (often in consultation with the Agency for Toxic Substances and Disease Registry (ATSDR)) or by State identification of Applicable or Relevant and Appropriate Requirements (ARARs). If action levels are exceeded, a series of chemical and physical tests may be required to evaluate possible treatment options.

1.5 CONCEPTUAL SITE MODEL

A conceptual site model is a useful tool for selecting sampling locations. It helps ensure that sources, migration pathways, and receptors throughout the site have been considered before sampling locations are chosen. The conceptual model assists the Site Manager in evaluating the interaction of different site features. Risk assessors use conceptual models to help plan for risk assessment activities. Frequently, a conceptual model is created as a site map (see Figure 1) or it may be developed as a flow diagram which describes potential migration of contaminants to site receptors (See Appendix A).

A conceptual site model follows contaminants from their sources, through migration pathways (e.g., air, ground water), and eventually to the assessment endpoints. Consider the following when creating a conceptual site model:

- The state(s) of each contaminant and its potential mobility
- Site topographic features
- Meteorological conditions (e.g., wind direction/speed, average precipitation, temperature, humidity)
- Human/wildlife activities on or near the site

The conceptual site model in Figure 1 is an example created for this document. The model assists in identifying the following site characteristics:

Potential Sources: Site (waste pile, lagoon); drum dump; agricultural activities.

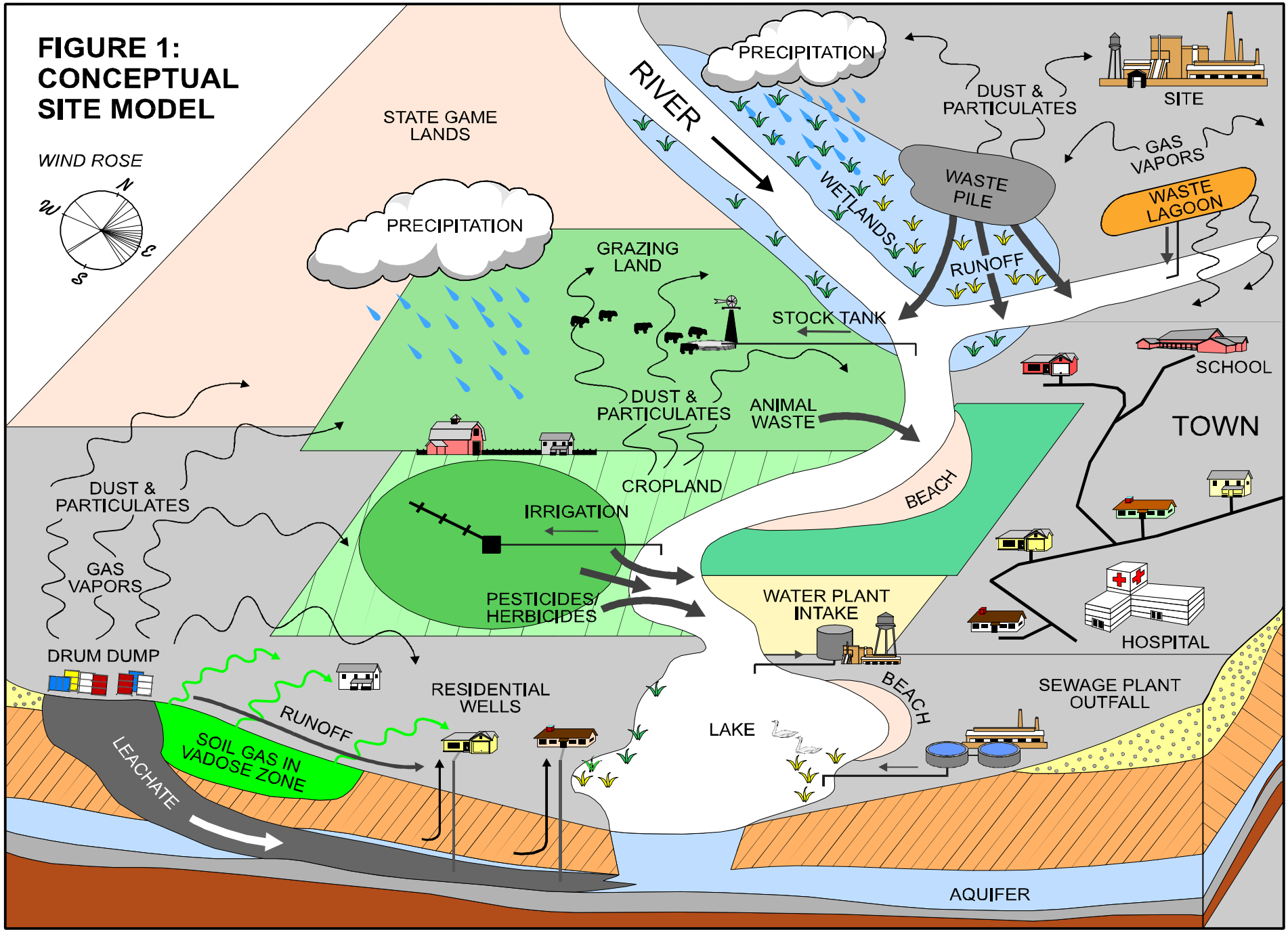
Potential Migration Pathway (Ground Water): Leachate from the waste pile, lagoon, drum dump, or agricultural activities.

Potential Migration Routes: Ingestion or direct contact with water from the aquifer (e.g., ingestion of drinking water, direct contact when showering).

Potential Receptors of Concern:

Human Population (Residents/Workers): Ingestion or direct contact with contaminated water from the aquifer.

Preliminary site information may provide the identification of the contaminant(s) of concern and the level(s) of the contamination. Develop a sampling plan based upon the receptors of concern and the suspected sources and pathways. The model may assist in the selection of on-site and off-site sampling locations.


FIGURE 1:
CONCEPTUAL
SITE MODEL
WIND ROSE
N
W
E
S
STATE GAME
LANDS
RIVER
PRECIPITATION
DUST &
PARTICULATES
SITE
GAS
VAPORS
WETLANDS
WASTE
PILE
WASTE
LAGOON
RUNOFF
PRECIPITATION
GRAZING
LAND
STOCK TANK
SCHOOL
TOWN
DUST &
PARTICULATES
ANIMAL
WASTE
CROPLAND
BEACH
DUST &
PARTICULATES
IRRIGATION
GAS
VAPORS
PESTICIDES/
HERBICIDES
WATER PLANT
INTAKE
HOSPITAL
DRUM DUMP
RESIDENTIAL
WELLS
BEACH
SEWAGE PLANT
OUTFALL
RUNOFF
SOIL GAS IN
VADOSE ZONE
LEACHATE
LAKE
AQUIFER

1.6 OVERVIEW OF GROUND-WATER MONITORING WELL INSTALLATION AND GROUND-WATER MODELING

Ground-water monitoring well installation and ground-water modeling are complex issues which fall outside the scope of this document. Many standard operating procedures (SOPs) covering ground-water monitoring well installation techniques have been published. Monitoring well installation and ground-water modeling are briefly introduced here with several specific items for consideration. Refer to existing SOPs and other reference documents for more in-depth study.

1.6.1 Ground-Water Monitoring Well Installation

For most Superfund response actions where ground-water sampling is performed, existing ground-water production wells (commercial or residential) are used, if available, to obtain samples. Chemical data obtained from this type of well depict the general quality of water that is being delivered to the user community. Ground water is usually a composite of multiple aquifer strata which may mask the presence of narrow or small contaminant plumes from a single stratum. For this reason, production wells are not suitable for detailed source, case-preparation, or research types of monitoring. Such detailed monitoring efforts require wells designed to determine the geologic and hydrologic quality at specific locations and depths. The following items must be considered for ground-water sampling from monitoring wells:

- Drilling method
- Monitoring well components
- Monitoring well location
- Well diameter
- Well depth
- Well screen location

Refer to the U.S. EPA *A Compendium of Superfund Field Operations Methods*, OSWER Directive 9355.0-14; *Compendium of ERT Ground-water Sampling Procedures*, OSWER Directive 9360.4-06; *RCRA Ground-Water Monitoring Technical Enforcement Guidance Document*, OSWER Directive 9950.1; and *RCRA Ground-Water Monitoring: Draft Technical Guidance*, EPA/530-R-93-001, for specific details on monitoring well installation. The latter two documents should be referenced for information on locating, installing, and developing monitoring wells.

Locating Monitoring Wells

Often, one well is sited near the center of the contaminant plume just downgradient from the contamination source. Another well is installed downgradient of the contaminant source, outside the limits of the plume. For background data, one well may be placed outside of the contaminant plume, upgradient of the contaminant source. Additional wells may be installed to track the amount of contaminant dispersion taking place.

Determining the depth to sample is critical for successful ground-water monitoring. Sampling depth depends on the contaminant density, the aquifer characteristics, and the slope of the water table or potentiometric surface. The number of wells necessary to monitor ground water varies depending on many factors. For example, if an impoundment contamination source is higher than the surrounding landscape, leachate may flow locally in all four downgradient directions. In this case, at least four wells are needed to monitor plume movement, plus a background well may be desired in an unaffected area. In addition, some wells may be installed at more than one depth in a contaminant plume to verify vertical flow or spread of contamination at different depths.

See Driscoll, 1986, pp. 715-16 for more information on locating monitoring wells.

Well Casing and Well Screen

Select a well casing material based on water quality, well depth, cost, borehole diameter, drilling procedure, and Federal, state, and local regulations. Types of casing materials include: steel, polyvinyl chloride (PVC), fiberglass, and Teflon®. Common well casing diameters range from 2 inches to 12 inches or greater, and depend on well type, well size, well depth, and subsurface geology. Often a series of progressively smaller-diameter well casings are used from the ground surface to the well depth.

A well screen is a filtering device which permits water to enter the well from the saturated aquifer while preventing sediment from entering the well. A well screen has slots or perforations and attaches to the well casing. It can be constructed of metal, plastic, or other material. Important criteria for selecting a well screen include: a large percentage of

open area, nonclogging slots, resistance to corrosion, and a sufficient column and collapse strength.

See Driscoll, 1986, pp. 413-431, and Fetter, 1993, pp. 339-344 for more information regarding well casing. See Driscoll, 1986, pp. 395-405, and Fetter, 1993, pp. 345-346 for more information regarding well screens. See U.S. EPA, November 1992, pp. 6-16 - 6-38 for advantages and disadvantages of selecting well casing and screen materials.

1.6.2 Ground-Water Modeling

Ground-water models, like conceptual site models, can be useful when selecting sampling approaches, objectives, and locations. Ground-water models developed for Superfund sites attempt to provide an estimation of how the actual ground-water system functions.

There are many types of ground-water models available (e.g., physical, analog, mathematical). The International Ground-Water Modeling Center (IGWMC) has developed a ground-water model definition which emphasizes the importance of describing a ground-water system mathematically. The IGWMC defines a ground-water model as "a non-unique, simplified, mathematical description of an existing ground-water system, coded in a programming language, together with a quantification of the ground-water system the code simulates in the form of boundary conditions, system parameters, and system stresses."

A ground-water model may be useful throughout site investigation activities because it can be adjusted as conditions in the actual ground-water system become better defined. The data which are generated by the model can be used to refine sampling approaches and locations as necessary. Typically, a ground-water modeling report will include data (results), along with a discussion of activities such as model calibration and conceptual model development. A suggested format for a ground-water modeling report can be found in U.S. EPA *Ground-Water Issue: Fundamentals of Ground-Water Modeling* (EPA/540/S-92/005).

1.7 EXAMPLE SITE

An example site, presented at the end of each chapter, illustrates the development of a representative ground-water sampling plan that meets Superfund Program objectives for early actions or emergency responses.

2.0 GROUND-WATER SAMPLING DESIGN

2.1 INTRODUCTION

The purpose of ground-water sampling is to provide technical information relative to the nature and condition of subsurface water resources at a specific time and place. Designs to monitor the status of ground water range from the studies of naturally occurring geochemical constituents to the detection or assessment of contamination within a ground-water system.

Ground-water sampling objectives include identifying threats, delineating sources and extent of contamination, determining treatment and disposal options, and confirming the attainment of targeted clean-up levels. Representative sampling designs are developed to most accurately characterize the hydrogeologic system and its interaction with the environment. Sampling protocols must integrate detailed sampling methodology, techniques and practices to ensure valid assessment. Sampling methodology and practice may be the most common source of assessment error. Consequently, sampling methodology and practice collectively demand careful preparation, execution, and evaluation to accurately characterize the hydrogeologic system or its subsystems. (For additional information see: U.S. EPA *Ground Water, Volume II: Methodology,* EPA/625/6-90/016b; and Palmer, Christopher M., *Principles of Contaminant Hydrogeology*.)

There are many methods and types of equipment useful for site characterization and sample collection. Selection of these factors is a critical component of a site-specific sampling design.

A properly developed ground-water sampling design defines the sampling purpose, protects site worker health and safety, effectively utilizes resources, and minimizes errors. The sampling design will vary according to the characteristics of the site. When developing a sampling design, consider:

- Prior actions at the site (e.g., sampling events, compliance inspections)
- Regional ground-water properties and characteristics
- Potential on-site waste sources (e.g., impoundments, waste piles, drums)
- Topographic, geologic, hydrologic, and meteorologic conditions of the site
- Flora, fauna, and human populations in the area

2.1.1 Pre-Sampling Plan Investigation

The pre-sampling plan investigation provides the planner with information critical to the development of a sound ground-water sampling design. Integration of all pertinent facts regarding the site history, the population(s) affected, and concentrations of substances on a site must be reviewed. After all of the pertinent information has been processed and incorporated into a thorough site pre-evaluation, the sampling plan can be developed. Considerations for sampling plan modification should be reviewed as necessary in light of the complex nature of ground-water resource dynamics.

Site History

Review of the site's history helps assess the natural and man-made impacts on a site. Geographic, geologic, tax, and fire insurance maps can indicate the status of the site. These maps can usually be found at local and collegiate libraries or municipal and county tax offices. Aerial photographs are helpful in reviewing operational use of the site. Archival aerial photographs may show changes in operation and site condition over time. This information can be correlated with information from potentially responsible parties.

Hydrogeologic information is critical to developing a sampling plan. A ground-water system is site specific, depending upon local geology, land and subsurface use, precipitation and water use, proximity to water bodies, and hydrogeologic parameters affecting contaminant transport. Hydrologic and hydrogeologic information can be found in libraries or requested from the U.S. Geological Survey (USGS), Water Resources Division, or state geological agencies and their water branches. Inspection histories can be used to determine prior health status of the site in view of possible trends. Local, state, and federal agencies dealing with health or environmental inspection can provide such historical information about a site.

Affected Populations

Human population statistics for the selected area can establish the number of people threatened by the contaminant exposure. Include populations affected

by projected migration of contaminants within the ground-water system. Knowing the interaction of the contaminant within a ground-water system and the potential regional populations exposed to the contaminant will focus the sampling plan to the source and possible pathways of the contaminant. Wildlife populations in the area must be studied as well. Wildlife in ponds, lakes, streams, rivers, and bays is often affected by contaminants transported by ground water discharging into surface water. Information regarding regional wildlife populations and susceptibility to hazardous substances can be obtained from federal and state wildlife and conservation agencies.

Detection Levels versus Maximum Contaminant Levels

Sampling plan development must also address the concentration level of the contaminant within the ground-water system in relation to the maximum contaminant levels (MCLs) allowed within a public water system. Refer to the Federal Register for the levels requiring enforceable action. Knowledge of the chemical contaminant interaction within the ground-water system can add insight into the fate of the contaminant (soluble or insoluble in water; less or more dense than water; the nature of reactivity with sediment or geology of the subsurface). Correlate the concentration level versus the location of these concentrations. A sequence of order can then be applied to the locations. Ideally, a pattern may develop that can be related to the ground-water system and its dynamics. In the case of a single location, investigate potential sources in the surrounding area either by working backwards from an identified contaminant spot to a potential source, or from a potential source to an identified contaminant spot. Also consider source-to-current-location pathways and projected pathways when developing a sampling plan.

2.1.2 Types of Information Provided by Ground-Water Sampling Assessment

There are several types of information that a ground-water sampling assessment provides. These include but are not limited to: measure of ground-water quality, contaminant concentrations compared to action levels, selection of the appropriate response action, and determination of ground-water flow and contaminant plume movement.

Measure of Ground-Water Quality

Ground-water sampling assessments provide information concerning measure of ground-water quality of a site or region. Water quality is classified according to many categories and its intended use. Drinking water is especially subject to guidelines. A sampling assessment of ground water can determine whether the quality of the water has been maintained, upgraded, or allowed to degrade. The natural and artificially induced characteristics of ground water from a specific site or region can be established by ground-water sampling assessments -- specifically, the chemical, biological, and physical characteristics of the ground water.

Contaminant Concentrations Compared to Action Levels

Ground-water sampling assessments provide a single contamination level for a particular sampling location, or a set of contamination levels for several sampling locations within a site. Comparison to action levels in ARARs determines the basis for further action. Thus, sampling can evaluate potential hazards and represent a condition of ground-water character requiring enforceable action procedures.

Selection of Appropriate Response Action

The level of contaminant concentration as determined through sampling assessments is a critical factor in selecting a site response action. Depending upon the degree or level of contaminant concentration, contaminant frequency, or number of locations established as contaminated, and the site's potential threat to human health or the environment, a rapid or extensive clean-up program can be formulated, as well as temporary or short-term responses (e.g., provision of bottled water).

A sampling assessment may not always indicate contamination of the site. Careful examination of sampling protocol must consider the range of explanations. A miscalculation of suspected source sites; gross procedure error in sampling, laboratory analysis, or documentation; or error at many other points in sampling protocol could be the source of assessment error. These errors are addressed more extensively in Chapter 5.

If quality assurance/quality control (QA/QC) procedures have been followed for ground-water sampling assessment, then it is possible that sources

of contamination may originate from above ground systems (e.g., lead entering tap water in the proximity of the facility). In any case, a sampling assessment at the least can characterize the natural ground-water conditions, which can be used as a control or comparison.

Determination of Ground- Water Flow and Contaminant Plume Movement

Knowing the direction of ground-water flow is important when evaluating a contaminated aquifer. When contamination enters the ground at a higher head (gradient) than exists at nearby shallow wells, these wells may become contaminated. Ground water flows from higher head to lower head. The direction of water movement may be determined using water-elevation data from a minimum of three wells. See Driscoll, 1986, pp. 79-85 and Freeze and Cherry, 1979, pp. 168-236 for more information regarding ground-water flow.

Ground-water tracers, such as dye or salt may be used to track ground-water flow velocities and contaminant plume movement. A tracer is placed in one well and the time of its arrival in a second well downgradient from the first well is noted. The dilution of the tracer detected in the second well can indicate the contaminant dilution rate and help determine the contaminant source concentration as well as the width, depth, and spreading velocity of the plume. Tracers also may be used to help determine aquifer porosity, hydraulic conductivity, and dispersivity.
The tracer selected must be detectable in extremely low concentrations and must not react chemically or physically with the ground-water or aquifer composition. See Driscoll, 1986, pp. 84-85 for more information regarding ground-water tracers.

2.1.3 Site Reconnaissance

A site reconnaissance can be conducted at an earlier date or immediately prior to sampling activities. It allows field personnel to assess actual, current site conditions, evaluate areas of potential contamination, evaluate potential hazards associated with sampling, and finalize a sampling plan. Site reconnaissance activities for a ground-water assessment include: observing and photographing the site; noting site access and potential evacuation routes; noting potential safety hazards; inventorying and recording label information from drums, tanks, or other containers; mapping process and waste disposal areas such as landfills, impoundments, and effluent pipes; mapping potential contaminant migration routes such as drainage, streams, and irrigation ditches; noting the condition of animals and/or vegetation; noting topographic and/or structural features; noting and mapping existing ground-water monitoring or other types of wells for potential sampling; and siting potential locations for new monitoring wells if necessary. Field personnel should use appropriate personal protective equipment when engaged in any on-site activities. Consider the following site-specific factors while performing a site reconnaissance:

- Sampling Objectives - Sampling is conducted typically to comply with regulations for the detection or assessment of suspected contamination within the subsurface. The information gathered aids in the identification of known and unknown substances present within the site and the level and extent of contamination of the environment. The information is used to document the condition of the ground-water system as an initial assessment, a record of development, or as evidence of remediation efficiency and compliance.

- Sample Collection and Toxicity - The samples collected are intended to document the absence or measure the presence of contaminants. The measure of acute or chronic toxicity is evaluated by assessing the site's extent of contamination, the time period in relation to the extent, and health hazards associated with the contaminant exposure time frame.

- Statistical Concerns - A site visit will familiarize the sampling planner with the environment to be sampled. Conspicuous indicators of potential contamination sources or contamination effects may suggest use of a judgmental or bias sampling design. A geostatistical sampling method can be cost-effective and time-efficient when compared to strictly random or random-stratified procedures. When using less random methods, the choice of sampling locations should be documented and justified. Employ random sampling in addition to bias sampling and include background or control samples for a thorough representation of the ground-water character. (See Section 2.3 for a discussion of sampling approaches.) (For additional information see Keith, Lawrence H., *Principles of Environmental Sampling*.)

- Timing of the Response - Consider seasonal variation when evaluating a site. Predictions of bad weather can influence technique and design. The urgency of the action weighed against

seasonal constraints may dictate the options available within the targeted budget.

- Site-Specific Factors Affecting Ground-Water Flow Many factors of a site control the path or direction of ground-water flow. A combination of geologic survey information with the site reconnaissance can better familiarize the planner with the dynamics of the hydrogeologic system. The local geology of a site can determine the direction and rate of ground-water movement by means of its orientation and composition (e.g., horizontal, tilted or vertical structures, and confining clay versus unconfining sand and gravel). The degree of development of a site and its local topography can affect the ground-water flow (e.g., parking lot runoff disproportionally delivers water quantities to the subsurface and greater slopes afford less infiltration of water to the subsurface). The extent and type of vegetation can affect the amount of rainfall that actually recharges an aquifer system. Dense vegetation and high evapo-transpiration from vegetation allows very little water to descend to the subsurface. Seasonal variations can cause reversal of ground-water flow direction. This is usually associated with water bodies such as streams, rivers, ponds, and lakes. Water may flow to or from streams depending upon its surface elevation in relation to adjacent water table surfaces. During flood conditions, water usually flows from rivers toward the surrounding subsurface. During drought, water moves toward the lower level of the stream surface from higher ground-water surfaces. (Consult U.S. EPA *Handbook, Ground Water*, EPA/625/6-87/016, Chapter 4: Basic Hydrogeology.)

- Analytical Parameters - The site reconnaissance can help develop the list of analytical parameters. For example, a reconnaissance may indicate the presence of battery casings. Lead would then be a substance of concern. The site may contain constraints that may or may not allow a variety of tests to be performed. The cost-effectiveness of testing within the site's constraints can lead to limited options available to properly analyze the ground-water system. Testing methods may vary within one site (e.g., monitoring well sampling, hydroprobe extraction, etc.) in order to evaluate multiple criteria vital to the site assessment.

- Degradation (or Transformation) Products - Sites may contain degradation (or transformation) products, or by-products, of the contaminant that are detectable and potentially as hazardous as the contaminant itself. Sampling for the product can lead to clues of the source substance location and its reactive status within the subsurface.

- Sampling Order - The sampling plan should address a specific order of sampling locations (and depths at a single location) to be developed. In order to use equipment efficiently, the plan should attempt to sample from "clean" to "dirty" locations, reducing the potential for contaminants to affect relatively less contaminated locations. Typically, the background or "clean" location of a site is hydrologically upgradient from the suspected contaminant "hot spot." Depending upon the nature of the contaminant (e.g., a "sinker" or "floater"), the sampling at different depths within a column of water in a monitoring well should also follow a sequence.

2.2 PARAMETERS OF CONCERN, DATA QUALITY OBJECTIVES, AND QUALITY ASSURANCE MEASURES

2.2.1 Parameters of Concern

Drinking water populations, contaminants, and migration pathways are additional parameters that should be considered when developing a sampling plan. Often, ground-water contamination goes undetected because it is not directly visible. Drinking water odor or taste complaints by residents close to the site are usually the initial indication of ground-water contamination and potential health hazards. The sampling data should accurately delineate the extent of ground-water contamination, determine the impact on drinking water populations, and indicate potential migration pathways to such populations. It is important to design the sampling plan to determine where contaminants are most highly concentrated, and to locate areas of decreasing detectable concentrations and those not yet contaminated.

2.2.2 Data Quality Objectives

Data quality objectives (DQOs) state the level of uncertainty that is acceptable for data collection activities and define the certainty of the data necessary to make decisions. The overall goal of DQOs for a representative ground-water sampling plan are to acquire thorough and accurate information about subsurface water conditions at a site. DQOs are unique and site specific and should address the

contaminant's interaction with the immediate site environment. When establishing DQOs for a particular project, consider:

- Decision(s) to be made or question(s) to be answered
- Why analytical data are needed and how the results will be used
- Time and resource constraints on data collection
- Descriptions of the analytical data to be collected
- Applicable model or data interpretation method used to arrive at a conclusion
- Detection limits for analytes of concern
- Sampling and analytical error

2.2.3 Quality Assurance Measures

To ensure that analytical samples are representative of site conditions, quality assurance measures must be associated with each sampling and analysis event. The sampling plan must specify QA measures, which include, but are not limited to, sample collection, laboratory SOPs, sample container preparation, equipment decontamination, field blanks, replicate samples, performance evaluation samples, sample preservation and handling, and chain-of-custody requirements. Quality assurance components are defined as follows:

- Precision - Measurement of variability in the data collection process

- Accuracy (bias) - Measurement of bias in the analytical process; the term "bias" throughout this document refers to (QA/QC) accuracy measurement

- Completeness - Percentage of sampling measurements which are judged to be valid

- Representativeness - Degree to which sample data accurately and precisely represent the characteristics and concentrations of the source/site contaminants

- Comparability - Evaluation of the similarity of conditions (e.g., sample depth, sample homogeneity) under which separate sets of data are produced

Refer to Chapter 5, Quality Assurance/Quality Control (QA/QC), for more detailed ground-water QA/QC information.

2.3 REPRESENTATIVE GROUND-WATER SAMPLING APPROACHES AND SAMPLE TYPES

Judgmental sampling is the primary representative sampling approach used for ground water. Other representative sampling approaches for ground water such as random, systematic grid, and systematic random sampling are described below. For information on the other types of sampling approaches, refer to U.S. EPA, *Superfund Program Representative Sampling Guidance*, Volume 1 -- *Soil*, OSWER Directive 9360.4-10.

2.3.1 Judgmental Sampling

Judgmental sampling is the biased selection of sampling locations at a site, based on historical information, visual inspection, sampling objectives, and professional judgment. A judgmental approach is best used when knowledge of the suspected contaminant(s) or its origins is available. Judgmental sampling includes no randomization in the sampling strategy, precluding statistical interpretation of the sampling results. Criteria for selecting sampling locations are dependent on the particular site and level of contamination expected.

Once a contaminant has been detected in the ground water, the source and extent must be identified. To do this, an understanding of the contaminant characteristics and the local geologic and hydrogeologic conditions is needed. Characteristics of the contaminant and any daughter (degradation) products must be known in order to understand how the material may be transported (both vertically and laterally) from the contamination source. Knowledge of the local hydrogeology is needed in order to identify areas or zones that would facilitate contaminant migration, such as water bodies and gravelly or sandy soils. The permeability of the underlying rock type should be analyzed, as well as its depth, which will help to narrow the potential sampling area. For example, if the underlying bedrock strikes northeast to southwest, then sampling of an aquifer should also be in this direction, unless cross-contamination between aquifers has already been identified.

When appropriate (based on sampling objectives, availability, sampling parameters, and budget), sample available local residential or commercial wells following a relatively systematic pattern based on the

geology of the area. In the example given in the paragraph above, wells would be sampled along a line northeast to southwest. If the number of wells available is not sufficient to adequately identify the extent of contamination, then additional monitoring wells could be installed.

During a ground-water assessment, the selection of locations for monitoring well installation is done with a judgmental approach. This is generally because monitoring wells are complex, expensive, and time-consuming to install. In order to best determine the nature of a suspected contaminant plume, monitoring wells need to be placed in areas most likely to intercept the plume. Using a random, systematic grid or a systematic random approach would likely result in too many wells that miss the contaminant plume. Even placement of background or control monitoring wells favors a judgmental approach. Locations are selected based on the site reconnaissance and the planner's knowledge of the suspected contaminants, site geology, and hydrology.

2.3.2 Random, Systematic Grid, and Systematic Random Sampling

Random, systematic grid, and systematic random sampling are generally not used for ground-water sampling because sampling points are pre-determined from either existing wells or monitoring wells which are placed by judgment. However, these approaches may be useful for soil gas testing to assist in the siting of new monitoring wells. They can also be useful for conducting Geoprobe® sampling, if necessary. For additional information on these sampling approaches, refer to U.S. EPA, *Superfund Program Representative Sampling Guidance*, Volume 1 -- *Soil*, OSWER Directive 9360.4-10.

2.3.3 Grab versus Composite Sample Types

Grab samples are essentially the only type of samples collected for ground water. Unlike surface water, ground water is not composited. Each ground-water sample is representative of a discrete location and horizon in the subsurface.

2.4 SAMPLING PLAN

To develop a successful and practical representative ground-water sampling plan, the following site-specific information is required:

- Site Location - The location of the site will often influence the size of the sampling area and whether sampling should be conducted on or off site or a combination of both.

- Local Geology and Hydrology - Local geology and hydrology can determine whether off-site sampling is necessary and defines ground-water sampling boundaries and locations. For example, if an aquifer is very deep or there is a confining layer between the ground surface and the aquifer, then sampling within a small area may be all that is necessary in order to determine the extent of contamination within that aquifer.

- Topography - Topography will control the direction of surface runoff and may give clues to subsurface conditions. For example, wells in valleys may not be of the same aquifer as wells on a hill.

- Analytical Parameters - If contaminants are initially unknown, then a broad spectrum of analytical parameters is usually collected. As more information about the site becomes available (through screening or laboratory analysis), the number of parameters can be streamlined or altered in order to more effectively characterize the site. If the contaminant is known, then concentrate on sampling for it and its degradation products.

- Sampling Budget - Budget constraints inevitably affect operations. A combination of screening and analytical techniques minimizes expenses while still providing an acceptable level of quality for the sampling data.

- Physiochemical Nature of Suspected Contamination When designing the sampling plan, take into account the physical and chemical nature of the suspected contaminants, then design the sampling plan to facilitate efficient detection of the contaminants through sampling methodology, equipment, and analyses. For example, the water density or solubility of a contaminant may provide an indication of the contaminant's physical location within the water column.

  Water has a specific gravity of one. Some chemical compounds, such as many complex petrochemicals, have a specific gravity of greater than one, and are therefore more dense than water. These substances tend to sink and include chlorinated solvents, wood preservatives, other

coal tar wastes, and pesticides. These compounds are referred to as *dense nonaqueous phase liquids* (DNAPLs), or "sinkers". On the other hand, a specific gravity of less than one will allow a contaminant to float on or near the water table, and includes many fuel oil products and byproducts (e.g., gasoline, benzene, toluene, ethyl benzene, xylene (BTEX), and other straight chain hydrocarbons). These compounds are referred to as *light nonaqueous phase liquids* (LNAPLs), or "floaters". Nonaqueous phase liquids (NAPLs) tend to exist as separate layers in the water column. A substance with a specific gravity value near to or equal to one will generally dissolve in the water column (e.g., acetone, phenols, and creosote). Because of the potential stratification in the water column due to NAPL substances, sampling location with respect to the suspected contaminant location within the well should always be considered.

LNAPLs commonly occupy the capillary fringe zone above the water table. In a confined aquifer, these compounds are found along the upper surface of the permeable unit and also within the overlying confining layer.

DNAPLs cause additional representative sampling concerns. These compounds move downward under the influence of gravity until reaching a less permeable formation where they may either accumulate, move downslope along the bedrock, or penetrate fractures. Special precautions should be taken during drilling at sites suspected of DNAPL contamination; ensure that the drilling does not induce the spread of free-phase DNAPL contaminants. Monitoring well installation should be suspended when a DNAPL or low permeability lithogic unit is encountered. Fine-grained aquitards (e.g., silt or clay) should be assumed to permit downward DNAPL migration. For guidance on sites with potential DNAPL contamination, see U.S. EPA *Estimating the Potential for Occurrence of DNAPL at Superfund Sites*, OSWER Directive 9355.4-07.

Additional elements which should be addressed in a representative ground-water sampling plan include:

- Sample Number - The number of samples collected depends on the number of sample locations. Normally one sample is taken at each location, except for QA/QC requirements (e.g., replicates, and matrix spike/matrix spike duplicates). If there are multiple, discreet aquifers at the site, then samples of each may be necessary. Splitting samples also requires an increase in the number of samples.

- Sample Volume - The sample volume is dependent on the analytical parameters. It is also dependent on whether the contaminant is known or unknown. A greater volume is generally needed when the contaminant is unknown because a larger suite of parameters is usually selected.

- Sample Location - Sample location is generally dictated by the availability of existing monitoring, residential, or commercial wells. New monitoring wells are located by judgmental methods.

- Sample Depth - Sampling depth is typically the bottom or screened zone of a well. However, there may be times when certain stratigraphic horizons within the water column may need to be discreetly sampled (e.g., capturing "floaters" or "sinkers"). (Procedures for addressing stratified samples are discussed in Section 4.4.)

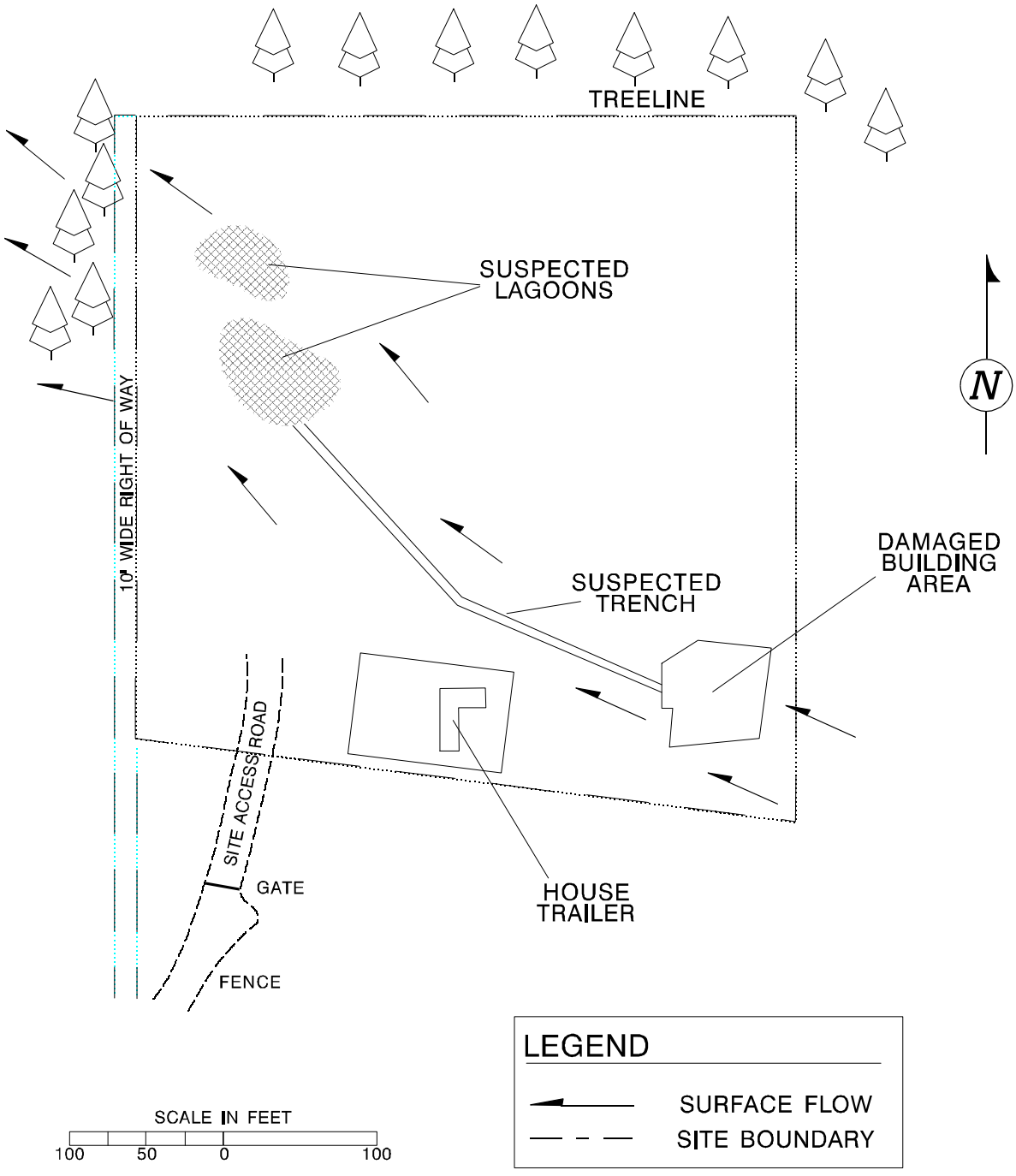
- Sample Order - Sampling order is from the least contaminated to the most contaminated wells or areas (if known).

2.5 EXAMPLE SITE

2.5.1 Background

The ABC Plating Site is located in northeastern Pennsylvania approximately 1.5 miles north of the town of Jonesville. Figure 2 provides a layout sketch of the site and surrounding area. The site covers approximately four acres and operated as a multi-purpose specialty electroplating facility from 1947 to 1982. During its years of operation, the company plated automobile and airplane parts with chromium, nickel, and copper. Cyanide solutions were used in the plating process. ABC Plating deposited electroplating wastes into two unlined shallow surface settling lagoons in the northwest portion of the site. Pennsylvania Department of Environmental Resources (PADER) personnel cited the owner/operator for the operation of an unpermitted treatment system and ordered the owner to submit a remediation plan for state approval. Before PADER could follow up on the order, the lagoons were partially backfilled with the wastes in place. The process building was later destroyed by a fire of suspicious origin. The owner abandoned the facility and could not be located by

Figure 2: Site Sketch
ABC Plating Site



enforcement and legal authorities. Several vats, drums, and containers were left unsecured and exposed to the elements. The state contacted EPA for an assessment of the site for a possible federally funded response action; an EPA On-Scene Coordinator (OSC) was assigned to the task.

2.5.2 Site History and Reconnaissance

The EPA OSC reviewed the PADER site file. In 1974 the owner was cited for violating the Clean Streams Act and for storing and treating industrial waste without a permit. The owner was ordered to file a site closure plan and to remediate the settling lagoons. The owner, however, continued operations and was then ordered to begin remediation in 90 days or be issued a cease and desist order. Soon after, a follow- The OSC obtained copies of aerial photographs of the site area from the local district office up inspection revealed that the lagoons had been backfilled without removing the waste.

The OSC and a sampling contractor (Team) arrived on site to interview local and county officials, fire department officers, neighboring residents (including a former facility employee), and PADER representatives regarding site operating practices and other site details. The former employee sketched facility process features on a map copied from state files. The features included two settling lagoons and a feeder trench which transported plating wastes from the process building to the lagoons. The OSC obtained copies of aerial photographs of the site area from the local district office of the U.S. Soil Conservation Service. The state provided the OSC with copies of all historical site and violation reports. These sources indicated the possible presence and locations of chromium, copper, and zinc plating process areas.

The Team mobilized to the site with all the equipment needed to perform multi-media sampling. The OSC and Team made a site entry, utilizing appropriate personal protective equipment and instrumentation, to survey the general site conditions. They observed 12 vats, likely containing plating solutions, on a concrete pad where the original facility process building once stood. Measurements of pH ranged from 1 to 11. Fifty drums and numerous smaller containers (some on the concrete pad, others sitting directly on the ground) were leaking and bulging because of the fire. Some rooms of the process building could not be entered due to unsafe structural conditions caused by the fire. The Team noted many areas of stained soil, which indicated container leakage, poor waste handling practices, and possible illegal dumping of wastes.

2.5.3 Identification of Parameters of Concern

During the site entry, the OSC and Team noted that several areas were devoid of vegetation, threatening wind erosion which could transport heavy metal- and cyanide-contaminated soil particulates off site. These particulates could be deposited on residential property downwind or be inhaled by nearby residents.

Erosion gullies located on site indicated surface soil erosion and stormwater transport. Surface drainage gradient was toward the west and northwest. The Team observed stressed, discolored, and necrotic vegetation immediately off site along the surface drainage route. Surface drainage of heavy metals and cyanide was a direct contact hazard to local residents. Surface water systems were also potentially affected. Further downgradient, site runoff entered an intermittent tributary of Little Creek, which in turn feeds Barker Reservoir. This reservoir is the primary water supply for the City of Jonesville and neighboring communities, which are located 2.5 miles downgradient of the site.

The site entry team observed that the site was not secure and there were signs of trespass (confirming a neighbor's claim that children play at the facility). These activities could lead to direct contact with cyanide and heavy metal contaminants, in addition to the potential for chemical burns from direct contact with strong acids and bases as might be found in leaking or unsecured drums or containers.

After interviewing residents, it was established that the homes located to the south and nearest to the site rely upon private wells for their primary drinking water supply. Ground water is also utilized by several small community production systems which have wells located within 2 miles of the site. The on-site settling lagoons were unlined and therefore posed a threat to ground water, as did precipitation percolating through contaminated soils. Contamination might have entered shallow or deeper aquifers and potentially migrated to off-site drinking water wells.

During Phase 1 sampling activities, full priority pollutant metals and total cyanide analyses were conducted on all soil and ground-water samples sent to the laboratory. These parameters were initially selected based on a study of plating chemistry:

plating facilities generally use either an acid or basic cyanide bath to achieve the desired coating on their metal products. Since Phase 1 samples were collected from the areas of highest suspected contaminant concentration (i.e., sources and drainage pathways), Phase 2 samples (all media types) were analyzed for total chromium, hexavalent chromium (in water only), and cyanide, the only analytes detected consistently during the Phase 1 analyses. During Phase 3, the samples sent to the laboratory for definitive analysis were analyzed for total chromium and cyanide.

2.5.4 Sampling Objectives

The OSC initiated an assessment with a specific sampling objective, as follows:

- Phase 1 -- Determine whether a threat to public health, welfare, and the environment exists. Identify sources of contamination to support an immediate CERCLA-funded activation for containment of contaminants and security fencing (site stabilization strategies) to reduce direct contact concerns on site. Sample the nearby drinking water wells for immediate human health concerns.

Once CERCLA funding was obtained and the site was stabilized:

- Phase 2 -- Define the extent of contamination at the site and adjacent residential properties. Estimate the costs for early action options and review any potential long-term remediation objectives. For example, install and sample soil borings and monitoring wells on site to evaluate potential impact on subsurface soils and ground water.

- Phase 3 -- After early actions are completed, document the attainment of goals. Assess that the response action was completed to the selected level and is suitable for long-term goals.

2.5.5 Selection of Sampling Approaches

The OSC, Team, and PADER reviewed all available information to formulate a sampling plan. The OSC selected a judgmental sampling approach for Phase 1. Judgmental sampling supports the immediate action process by best defining on-site contaminants in the worst-case scenario in order to evaluate the threat to human health, welfare, and the environment. Threat is typically established using a relatively small number of samples (fewer than 20) collected from source areas or suspected contaminated areas based on the historical data review and site reconnaissance. For this site, containerized wastes were screened to categorize the contents and to establish a worst-case waste volume, while soil samples were collected to demonstrate whether a release had already occurred, and nearby residential drinking water wells were sampled for immediate human health concerns.

For Phase 2, a stratified systematic grid design was selected to define the extent of contamination in soils. The grid could accommodate analytical screening and geophysical surveys. Based on search sampling conducted at sites similar to ABC Plating, a block grid with a 50-foot grid spacing was selected. This grid size ensured a 10 percent or less probability of missing a "hot spot" of 45 feet by 20 feet. The grid was extended to adjacent residential properties when contaminated soil was identified at grid points near the boundary of the site.

Based on the results of soil sampling and geophysical surveys, a judgmental approach was used to select locations for installation of 15 monitoring wells: at "hot spots"; along the perimeter of the suspected plume established from analytical results and geophysical survey plots; and at background ("clean") locations. Subsurface soil and ground- water samples were collected from each of the 15 monitoring well locations for laboratory analysis to establish the presence and, if applicable, the degree of contamination at depth.

2.5.6 Sampling Plan

During Phase 1, containerized wastes were evaluated using field analytical screening techniques. Phase 1 wastes-screening indicated the presence of strong acids and bases and the absence of volatile organic compounds. The Team collected a total of 12 surface soil samples (0-3 inches) and 3 ground-water samples during this phase and sent them to a laboratory for analysis. The soil sampling locations included stained soil areas, erosion channels, and soil adjacent to leaking containers. Background samples were not collected during Phase 1 because they were unnecessary for activating immediate action response funding. Ground-water samples were collected from three nearby residential wells. Based on Phase 1 analytical results, chromium was selected as the target compound for determination of extent of contamination in soil and ground water.

During Phase 2 sampling activities, the OSC used a transportable X-ray fluorescence (XRF) unit installed in an on-site trailer to screen soil samples for total chromium in order to limit the number of samples to be sent for laboratory analysis. Soil sampling was performed at all grid nodes at the surface (0-4 inches) and subsurface (36-40 inches). The 36-40 inch depth was selected based on information obtained from state reports and local interviews, which indicated that lagoon wastes were approximately 3 feet below ground surface. Once grid nodes with a contamination level greater than a selected target action level were located, composite samples were collected from each adjoining grid cell. Based on the XRF data, each adjoining grid cell was either identified as "clean" (below action level) or designated for response consideration (at or above action level).

Also during Phase 2, the OSC oversaw the performance of ground penetrating radar (GPR) and electromagnetic conductivity (EM) geophysical surveys to help delineate the buried trench and lagoon areas, any conductive ground-water plume, and any other waste burial areas. The GPR and comprehensive EM surveys were conducted over the original grid. Several structural discontinuities, defining possible disturbed areas, were detected. One GPR anomaly corresponded with the suspected location and orientation of the feeder trench. The EM survey identified several high conductivity anomalies: the suspected feeder trench location, part of the lagoon area, and a small area west of the process building, which may have been an illegal waste dumping area. (Field analytical screening and geophysical techniques are further discussed in Chapter 3.)

Using the data obtained during soil sampling and the geophysical surveys, a ground-water assessment plan for Phase 2 was prepared. The Team collected depth soundings and water level measurements of the nearby residential wells to assess aquifer usage and location (depth). With these data and the analytical results from Phase 1, a work plan for monitoring well installation and testing on site was developed. The plan consisted of:

- Installation of overburden, bedrock contact and bedrock (open borehole) monitoring wells in order to evaluate the shallow water table and aquifer conditions
- Analysis of subsurface soils retrieved during borehole/well drilling in order to evaluate the extent of contamination in overlying soils
- Collection of depth soundings and water level measurements of the newly installed monitoring wells to map aquifer and water table gradients
- Collection of ground-water samples from each monitoring well
- Performance of hydraulic tests in order to evaluate aquifer characteristics

The monitoring wells were located in areas shown, during soil sampling, to be heavily contaminated; along the outer perimeter of a contaminant plume based on soil XRF results and the geophysical surveys; and an apparent upgradient location for background conditions comparison. Fifteen wells were located at grid nodes corresponding to the above results. (Section 4.6.1 provides details on the performance of well installation (drilling), testing and surveying, and ground-water sampling procedures.)

Upon monitoring well installation and sampling, a hydraulic (pumping) test was completed of the bedrock monitoring wells to gather information about aquifer characteristics. These data characterize contaminant transport through the ground-water aquifer. The hydraulic test provided transmissibility, hydraulic conductivity, and storativity values. Utilizing these values with ground-water level data, the estimated vertical and horizontal ground-water gradient and velocity could be calculated. All monitoring wells installed were surveyed for elevation above mean sea level, needed to determine accurate depth to ground water (piezometric surface) and relative gradients.

Phase 3 activities are discussed in Section 6.8.

3.0 FIELD ANALYTICAL SCREENING, SAMPLING EQUIPMENT, AND GEOPHYSICAL TECHNIQUES

3.1 FIELD ANALYTICAL SCREENING

Field analytical screening techniques can provide valuable information in ground-water sampling. Field analytical screening for ground water is used primarily as a tool for siting monitoring wells and for on-site health and safety assessment during well drilling activities. When used correctly, screening techniques can help to limit the number of "non-detect" monitoring wells installed. Some of the commonly used screening methods for ground-water assessment are presented in this chapter in the general order that they would initially be used at a site, although site-specific conditions may mandate a different sequence. For more information on ground-water field screening devices, refer to the U.S. EPA *Compendium of ERT Field Analytical Procedures*, OSWER Directive 9360.4-04, and *Compendium of ERT Ground-Water Sampling Procedures*, OSWER Directive 9360.4-06. Refer to Standard Operating Safety Guides for each instrument, and the U.S. Department of Health and Human Services *Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities* (NIOSH Pub. 85-115) for site entry information.

3.1.1 Flame Ionization Detector

The flame ionization detector (FID) detects and measures the level of total organic compounds (including methane) in the ambient air in proximity to a well or in a container headspace. The FID uses the principle of hydrogen flame ionization for detection and measurement. It is especially effective as an ethane/methane detector when used with an activated charcoal filter because most organic vapors are absorbed as the sample passes through the filter, leaving only ethane and methane to be measured.

The FID operates in one of two modes: the survey mode, or the gas chromatography (GC) mode. In the survey mode, the FID provides an approximate total concentration of all detectable organic vapors and gases measured relative to the calibration gas (usually methane). The GC mode identifies and measures specific components, some with detection limits as low as a few parts per million (ppm), using known standards analyzed concurrently in the field. Since the GC mode requires standards to identify classes of compounds, it is necessary to have an idea of which compounds might be present on site before sampling. Advantages of the FID are that it is portable, relatively rugged, and provides real-time results.

During a ground-water assessment, the FID is used in the survey mode for monitoring the borehole during drilling and in the survey or GC mode for health and safety screening.

The FID does not respond to inorganic substances. It has positive or negative response factors for each compound depending on the selected calibration gas standard. Ambient air temperatures of less than 40 degrees Fahrenheit will cause slower responses; relative humidity of greater than 95 percent can cause inaccurate and unstable responses. Interpretation of readings (especially in the GC mode) requires training and experience with the instrument.

3.1.2 Photoionization Detector

Another portable air monitoring instrument frequently used for field screening during ground-water assessments is the photoionization detector (PID). Like the FID, the PID provides data for real-time total organic vapor measurements, identifying potential sample locations and extent of contamination, and supporting health and safety decisions. The PID is useful in performing soil gas screening, health and safety monitoring during well drilling activities, and headspace screening analysis. The PID works on the principle of photoionization. Unlike the FID, the PID can be used to detect gross organic and some inorganic vapors, depending on the substance's ionization potential (IP) and the selected probe energy. It is portable and relatively easy to operate and maintain in the field.

The PID detects total concentrations and is not generally used to quantify specific substances. PIDs cannot detect methane; however, methane is an ultraviolet (UV) light absorber, and false negative instrument readings may register in methane-rich environments. The PID cannot detect substances with IPs greater than that of the UV light source. (Interchangeable UV lamps are available.) Readings can be affected by high wind speeds, humidity, condensation, dust, power lines, and portable radios. Dust particles and water droplets (humidity) in the sample may collect on the light source and absorb or

deflect UV energy, causing erratic responses in PIDs not equipped with dust and moisture filters.

3.1.3 Gas Chromatograph

Although many FIDs are equipped with a GC mode, an independent, portable GC (gas chromatograph) can also be used on site to provide a chromatographic profile of the occurrence and intensity of unknown volatile organic compounds (VOCs) in ground water. The GC is useful as a soil gas screening tool to determine "hot spots" or plumes, potential interferences, and semi-quantitation of VOCs and semi-volatile organic compounds (semi-VOCs) in ground-water samples. For example, when installing a monitoring well, the GC might be used to screen water samples during drilling in order to indicate when a target contaminated aquifer zone is encountered.

Compounds with high response factors, such as benzene and toluene, produce large response peaks at low concentrations, and can mask the presence of compounds with lower response factors. However, recent improvements in GCs, such as pre-concentrator devices for lower concentrations, pre-column detection with back-flush capability for rapid analytical time, and the multi-detector (PID, FID, and electron capture detector (ECD)), all enable better compound detection. The GC is highly temperature-sensitive. It requires set-up time, many standards, and operation by trained personnel.

3.1.4 Hydraulic Probe

The hydraulic probe (Geoprobe® is one brand) is a truck-mounted device used to collect screening ground-water, soil, and soil gas samples at relatively shallow depths. The probe is mounted on the back of a small truck or van and is operated hydraulically using the vehicle's engine. Small diameter hardened steel probes are driven to depths of up to 40 feet or more, depending on soil conditions. Soil gas samples can then be collected using a vacuum pump. Soil or water samples can also be collected using a small-diameter shelby tube or slotted well point and foot valve pump.

The hydraulic probe can be used in ground-water investigations to assess vertical and horizontal extent of contamination. Shallow samples can be collected relatively quickly and easily. It is useful in a ground-water assessment to assist in siting monitoring wells and to install shallow wells if necessary. It can also collect undisturbed ground-water samples without installing wells. The hydraulic probe is only effective in unconsolidated geologic materials, however. In general, probing is possible under conditions amenable to hollow stem auger drilling.

3.1.5 Soil Gas Technique

Soil gas testing is a quick method of site evaluation. For ground-water assessments, soil gas testing is used to track contaminant plumes and determine appropriate locations for installing monitoring wells. For this technique, a thin stainless steel probe is inserted into a hole made in the soil with a special slam bar. The hole is sealed around the probe and a sampling pump is attached. Samples are then collected in Tedlar bags, sorbent cartridges, or SUMMA canisters. The samples are analyzed using an FID, PID, or GC. A disadvantage of the soil gas technique is that its ability to detect contaminants diminishes the further it is from the source (as contaminant concentration diminishes).

3.1.6 Field Parameter Instruments

Field parameters measured during ground-water sampling include pH, specific conductivity, temperature, salinity, and dissolved oxygen. Specific conductivity, pH, and temperature are often used as standard indicators of water quality. Instruments that measure these three indicators are used during ground-water assessments to determine if a well has been purged sufficiently (stabilized) prior to sampling (see Section 4.3).

3.1.7 X-Ray Fluorescence

Field analytical screening using X-ray fluorescence (XRF) is a cost-effective and time-saving method to detect and classify lead and other heavy metals in a sample. XRF screening provides immediate semi-quantitative results. The principle behind XRF is the detection and measurement of the X-rays released from an atom when it is excited by the absorption of source X-rays. The energy released (fluorescent X-rays) are characteristic of the atoms present.

Results of XRF analysis help determine the presence of metals and are often used to assess the extent of soil contamination at a site. For ground-water assessment, XRF may be used on subsurface soil samples collected during drilling or with surface soils when selecting locations for monitoring well installation. XRF use requires a trained operator and may require numerous site-specific calibration samples.

3.2 GROUND-WATER SAMPLING EQUIPMENT

Conducting representative ground-water sampling requires an understanding of the capabilities of the equipment used for sampling, since the use of inappropriate equipment may result in biased samples. Select appropriate sampling equipment based on the sampling objectives, the analytical parameters, the type of well being sampled (e.g., monitoring well or drinking water well), and other site-specific conditions. Follow SOPs for the proper use and decontamination of sampling equipment. This section presents various types of ground-water sampling equipment and information to assist in selecting appropriate materials.

The ground-water sampling devices discussed below are covered in greater detail in many SOPs and references on the various types of available ground-water sampling devices. Refer to U.S. EPA *A Compendium of Superfund Field Operations Methods*, OSWER Directive 9355.0-14, and *Compendium of ERT Ground-Water Sampling Procedures*, OSWER Directive 9360.4-06, for details on the equipment listed. Also refer to Driscoll, Fletcher G., *Ground-Water and Wells*, 2nd ed., and the 1985 "Proceedings of the Fifth National Symposium and Exposition on Aquifer Restoration and Ground-Water Monitoring," for additional comparisons of the various types of sampling equipment.

3.2.1 Bailer

A bailer is a simple purging device for collecting samples from monitoring wells. It usually consists of a rigid length of tube with a ball check-valve at the bottom. A line is used to mechanically lower the bailer into the well to retrieve a volume of water. Because bailers are portable and inexpensive, they can be dedicated to monitoring wells at a site, thus avoiding the need to use a bailer for sampling more than one well (and avoiding cross-contamination). Bailers are available in a variety of sizes and construction materials (e.g., polyvinyl chloride (PVC), Teflon®, and stainless steel).

Bailers are best suited for purging shallow or narrow diameter monitoring wells. Deeper, larger diameter, and water supply wells generally require mechanical pumps to evacuate a large volume of water.

For VOC analysis, a positive-displacement volatile sampling bailer is most effective. Bottom-fill bailers, which are more commonly used, are suitable provided that care is taken to preserve volatile constituents. Fill sample containers directly from the bailer, filling samples for VOC analysis first.

3.2.2 Hydraulic Probe

The hydraulic probe can be used to collect shallow (generally 40 feet or less) ground-water samples using a mill-slotted well point or retractable screen drive point. After the well point is driven to the desired depth, the probe rod is connected to a vacuum pump for purging. (Since ground water is sampled in situ and is not exposed to the atmosphere, extensive purging is not required.)

Water samples are collected using dedicated polypropylene tubing fitted with a small diameter foot-valve pump. Samples are collected in 40-ml vials or other containers for laboratory analysis. See Section 3.1.4 for more information on the hydraulic probe.

3.2.3 Air-Lift Pump

An air-lift pump operates by releasing compressed air via an air line lowered into the well. The air mixes with the water in the well to reduce the specific gravity of the water column and lift the water to the surface.

Air-lift pumping is used in well development and for preliminary testing. For sampling, air-lift pumping is less efficient than other pumping methods which follow; it may be selected for use when aeration is needed to remove gas or corrosive water which can be destructive to a well pump. Because an air-lift pump aerates the water, it is not applicable for VOC sample collection.

3.2.4 Bladder Pump

A bladder pump consists of a stainless steel or Teflon® housing that encloses a Teflon® bladder. The bladder pump is operated using a compressed gas source (bottled gas or an air compressor). Water enters the bladder through a lower check valve; compressed gas moves the water through an upper check valve and into a discharge line. The upper check valve prevents back flow into the bladder.

The bladder pump can be used to purge and sample to a depth of approximately 100 feet. It is recommended for VOC sampling because it causes minimal alteration of sample integrity as compared with other

ground-water sample methods. The bladder pump requires a power supply and a compressed gas supply or air compressor. The pump is somewhat difficult to decontaminate and should thus be dedicated to a well (or dedicated tubing should be used).

3.2.5 Rotary Pump

A rotary pump is a positive displacement pump which discharges the same volume of water regardless of the water pressure. The rate of discharge is the same at both low and high pressure, but the input power varies in direct proportion to the pressure. The rotary pump consists of a housing with inlet and outlet ports and rotating gears or vanes. As water is discharged from the pump, a replacement supply of equal volume is taken in.

Rotary pumps are useful for well purging and general sample collection at shallow to deep sampling depths. Because of water agitation, they may not be suitable for sampling VOCs, and they are difficult to decontaminate between sampling stations.

3.2.6 Peristaltic Pump

A peristaltic pump is a suction lift pump consisting of a rotor with ball-bearing rollers. Dedicated Teflon® tubing is threaded around the rotor. Additional lengths of dedicated Teflon® tubing are attached to both ends of the rotor tubing: one end is inserted into the well; the other end is a discharge tube. The sample makes contact with the tubing only, not with the pump. The tubing should be equipped with a foot valve to avoid having aerated water from the tubing fall back into the well.

A peristaltic pump is suitable for sampling small diameter wells (e.g., 2 inches). Cross-contamination is not of concern because dedicated tubing is used and the sample does not come into contact with the pump or other equipment. The peristaltic pump has a depth limitation of 25 feet and its use can result in a potential loss of the volatile fraction due to sample aeration.

3.2.7 Packer Pump

A packer pump is used to isolate portions of a well or water column for sampling. The pump consists of two expandable parts that isolate a sampling unit between them. The parts deflate for vertical movement within the well and inflate when the desired sampling depth is reached. The packers are constructed of rubber and can be used with various types of pumps.

An advantage of the packer pump is it allows the isolation of a portion of the water column in order to sample at a discrete depth. Disadvantages relate to the rubber construction of the packers which may deteriorate over time allowing cross contamination. The rubber also poses potential contaminant compatibility concerns. A packer pump should not be used if the contaminants are unknown, or where well casing or contaminant characteristics interfere or interact with the pump construction materials.

3.2.8 Syringe Sampler

Syringe samplers are a relatively new and less commonly available sampling device. Syringe samplers were developed by research groups to obtain ground water samples over a period of time. The device consists of a syringe (15 to 1500 ml in volume) which is lowered into the well to the desired sampling depth. The syringe plunger is then pulled open by a remote method, either mechanical or pneumatic, allowing the syringe to fill.

The remote operation allows the collection of a sample at a discrete depth. In addition, the interior of the sampler (i.e., the syringe) is not exposed to the water column. Disadvantages to this device include the small volume of sample that can be collected, it cannot be adapted for evacuation/purging uses, and it is not readily commercially available.

3.2.9 Ground-Water Sampling Equipment Selection Factors

The following factors should be considered when selecting ground-water sampling equipment.

- Composition - Select the composition of the sampling equipment based on the sampling parameters and objectives. For example, use samplers made of Teflon®, glass, or stainless steel instead of PVC when sampling for VOCs. Consider well composition when selecting sampling equipment. For example, select a stainless steel bailer when bailing a well with stainless steel casing to avoid the introduction of organic constituents. When sampling a PVC-cased well, PVC, stainless steel, or Teflon® bailers may be used.

- Physical Constraints - Physical constraints of the monitoring well location, power availability, and topography are factors that affect selection of ground-water sampling equipment. For example, a small diameter or particularly deep well may

require the use of different purging and sampling equipment than that used for other wells at the site. Site accessibility may hinder the use of large or vehicle-mounted equipment.

- Sample Analysis - Equipment should be chosen based on its impact on the samples. For example, sampling equipment selected for collecting VOCs should agitate the water as little as possible. This is not as critical for metals or other non-volatile analyses.

- Ease of Use - Generally, the more complicated the sampling equipment is, the greater the chance for some form of failure in the field. Utilize the simplest effective sampling devices available. Adequate training in equipment safety and use is critical to personnel safety as well as to sample representativeness. Consider ease of decontamination when using non-dedicated equipment.

3.3 GEOPHYSICAL METHODS

Geophysical methods can be useful in conjunction with screening and sampling activities to help delineate subsurface features and boundaries, contaminant plumes, and bedrock types. Geophysical data can be obtained relatively rapidly, often without disturbing the site. The data are helpful in selecting well locations and screen depths. The following sections discuss surface and borehole geophysics and preferable geophysical techniques for ground-water investigations.

3.3.1 Surface Geophysics

The following surface geophysical techniques may be useful in ground-water investigations. As implied by the name, these techniques are performed above ground. For more detailed information on each of these techniques (with the exception of gravimetric surveys), see ERT SOP #2159 and Driscoll, 1986. For more information on gravimetric surveys, see Driscoll, 1986.

- Ground Penetrating Radar (GPR) - Uses a high frequency transmitter that emits radar pulses into the subsurface. These waves are scattered at points of change in the dielectric permittivity of the subsurface material and are reflected back to an antenna. (Dielectric permittivity is a function of bulk density, clay content, and water content of the subsurface.) The returning energy wave is then plotted as a function of time on an analog plot. Interpretation of the analog plot identifies anomalies, clay layers, and water content in the substrate.

  GPR works best in dry, sandy soil above the water table, and at depths between 1 and 10 meters (although the full instrument depth range is less than one meter to tens of meters). When properly interpreted, GPR data can indicate changes in soil horizons, fractures, and other geological features, water-insoluble contaminants, man-made buried objects, and hydrologic features such as water table depth. Uneven ground surfaces or cultural noise affect GPR results.

- Electromagnetic Conductivity (EM) - Relies on the detection of induced electrical current flow through geologic strata. This method measures bulk conductivity (inverse of resistivity) of subsurface materials below the transmitter and receiver. EM is commonly used in the detection of ground-water pollution, as well as to locate pipes, utility lines, cables, trenches, buried steel drums, and other buried waste.

  EM has limited applications in areas of cultural noise, including above-ground power lines and metal fences, and lateral geologic variations which might be misinterpreted as contaminant plumes.

- Electrical Resistivity - Used to map subsurface structures through differences in their resistance to electrical current. Material resistivities are measured as functions of porosity, permeability, water solution, and concentrations of dissolved solids in pore fluids. Bulk resistivity is measured in the subsurface by measuring electrical currents injected through electrodes placed in the soil.

  Electrical resistivity surveys are limited by electrical noise, such as occur in industrial areas. Resistivity surveys should ideally be conducted in areas removed from pipelines and grounded metallic structures such as metal fences and railroad tracks. This requirement precludes use of electrical resistivity surveys on many sites. Resistivity can often be used off site to map area stratigraphy. Resistivity surveys are labor intensive, requiring ground setup and removal of electrodes for each station measurement. Use extreme care during rain or wet ground conditions.

- Seismic Investigations - Conducted by two methods: refractive and reflective. In the refractive method, the travel time of acoustic waves is measured as they move through and are refracted along an interface of the subsurface. The reflective method measures travel time of acoustic waves as they are reflected off an interface. Seismic refraction is typically used when bedrock is within 500 feet of the ground surface.

  Seismic refraction is useful for mapping discrete stratigraphic layers and therefore can help in selecting monitoring well locations and depths. A seismic refraction survey can provide subsurface stratigraphic and structural data in areas between existing wells or boreholes. Seismic reflection is used less often in ground-water investigations, but is more commonly used for deeper and larger-scale stratigraphic mapping (e.g., petroleum exploration).

- Magnetic Investigations - Rely on local variations in the earth's magnetic field to detect ferrous or magnetic objects. By mapping variations in the concentrations of the local magnetic fields, detection of buried objects such as drums or tanks may be accomplished. Magnetic surveys are limited by cultural noise such as power lines, utilities, and metal structures.

- Gravimetric Surveys - Measure small localized differences in the earth's gravity field caused by subsurface density variations, which may be produced by changes in rock type (porosity and grain type), saturation, fault zones, and varying thickness of unconsolidated sediments overlying bedrock. This method is useful in identifying buried valleys, particularly in glaciated areas.

  Gravimetric surveys use a portable gravity meter which can survey a large area relatively quickly. The accuracy of the readings is dependent upon the accuracy of the elevation determination of each station. (Most altimeters are accurate only to plus or minus 2 ft (0.6 m), so gravity stations should be surveyed.) A gravimetric survey can provide a quick preliminary screening of an area. Other geophysical methods or test drilling can then be used to help identify stratigraphy and aquifer characteristics.

Table 1 illustrates the applicability of various surface geophysical techniques to ground-water investigations. Table 2 lists some advantages and disadvantages of surface geophysical techniques to ground-water investigations.

3.3.2 Borehole Geophysics

The following borehole geophysical techniques may be useful in ground-water investigations. Borehole geophysics may be used alone or to supplement surface geophysical techniques. Site terrain is an important factor when conducting borehole geophysical surveys. Much of the equipment is mounted or housed inside a truck but can be carried to well locations if necessary. Some borehole logs can be run in a cased as well as open hole.

Often several of the following tests are run at the same time for comparative purposes. Borehole geophysical logs can be interpreted to determine the lithology, geometry, resistivity, formation factor, bulk density, porosity, permeability, moisture content, and specific yield of water-bearing formations as well as the chemical and physical characteristics of ground water. The operating principles of the various borehole geophysical techniques are similar. A sonde (a cylindrical tool containing one or more sensors) is lowered to the bottom of the borehole, activated, and slowly withdrawn. Signals or measurements at various depths are recorded at the surface. Instruments vary from hand-held portable gear to truck-mounted, power-driven equipment. For more detailed information on each of these techniques, see Driscoll, 1986.

- Resistance Logs - Electric logs measuring the apparent resistivity of the rock and fluid surrounding a well. They are good indicators of subsurface stratigraphy and water quality. Electric current is measured as it flows from electrodes in the probe to other electrodes in the probe or on the ground surface.

  Resistance logs have a small radius of investigation and are very sensitive to conductivity of borehole fluid and changes in borehole diameter. Increases in formation resistivity produce corresponding increases in resistance measurements on the log. Deflections on the log are interpreted as changes in lithology. Because of its excellent response to lithology changes, the resistance log is very useful for geological correlation. Formation fluids are perhaps the most important variable in interpreting resistance logs. For example, dry sands and clays have high resistivities, but their resistivities decrease with water saturation.

Dissolved minerals also affect resistivity. (Fresh water is a poor conductor whereas salt water is a good conductor; water in saturated clays contains dissolved minerals from the clay, which results in high conductivities.)

A limitation of resistance logs is that they can be run only in uncased boreholes that are filled with drilling fluid and water. Resistance logging is therefore most appropriately conducted before monitoring well completion.

- Spontaneous Potential (SP) Logs - Used in conjunction with resistivity logs to show the naturally occurring electric potentials of the chemical and physical changes at contacts between differing types of geologic materials. The electric current is measured between an electrode placed in an uncased borehole and one placed at the surface.

  SP response is due to small voltage differences caused by chemical and physical contacts between the borehole fluid and the surrounding formation. Voltage differences appear at lithology changes or bed boundaries and their response is used to quantitatively determine bed thickness or formation fluid resistivity. Qualitative interpretation of the data can be used to identify permeable beds.

  Buried cables, pipelines, magnetic storms, and the flow of ground water can all cause anomalous readings. Caution must be exercised when using SP data in a quantitative fashion. Mathematical formulas are structured for oil well logging and incorporate assumptions which may not apply to fresh water wells. As with resistance logs, SP logs can be run only in uncased, liquid-filled boreholes.

- Gamma Logs - Measure the naturally occurring gamma radiation emitted from the decay of radioisotopes normally found in the substrate. Elements that emit natural gamma radiation are potassium-40 and daughter products of the uranium and thorium decay series. Changes in radiation levels are commonly associated with differences in substrate composition.

  Gamma logs can be run in open or cased boreholes filled with water or air. The sensing device can be part of the same sonde that conducts SP and resistance logs. Gamma rays or photons are measured and plotted as counts per minute. This method is useful in identifying clay layers or other naturally radioactive geologic units.

  Gamma logging is used to identify the lithology of detrital sediments, where the finer-grained units have higher gamma intensity. (Fine-grained materials also tend to have lower permeability and effective porosity, important for evaluating aquifer zones.) A limitation with gamma and other nuclear logs is that they are affected by changes in borehole diameter and borehole media (e.g., air, water, or mud). Gamma logs record the sum of the radiation emitted from the formation and do not distinguish between radioactive elements. For use in stratigraphic correlation however, specific element identification is not critical. Interpretation of gamma logs is difficult where sandstone and other strata contain volcanic rock fragments with radioactive minerals (e.g., rhyolite). Interpretation is also difficult in sandstone containing a large proportion of feldspar (which contains radioactive potassium-40).

- Gamma-Gamma Logs - Similar to gamma logs except that a radioactive gamma source is attached to the gamma sonde and the gamma particles reflected back from the geologic formation are measured. Gamma-gamma logs measure the differing bulk densities of geologic materials. They can be used to identify lithology and also to calculate porosity when fluid and grain density are known.

- Neutron Logs - Also utilize a radiation source in the sonde. The neutron source is a europium-activated, lithium iodide crystal enriched in lithium-6. The neutron logging tool bombards the formation with neutrons and measures the returning radiation. Neutrons, when ejected from a nucleus, have great penetrating power and may travel through several feet of subsurface formation. All free neutrons are eventually captured by the nuclei of some element. Neutron logs respond primarily to hydrogen density. The high energy neutrons from the source are slowed by collision with hydrogen ions in the formation. This response to hydrogen ion content is then cross-calibrated to porosities for water-saturated rocks. Neutron logs respond to the hydrogen content in the borehole and surrounding formation and indicate the porosity of the various geologic units in the survey. Neutron logs can be run in cased or open holes which are dry or filled with fluid.

Neutron logs are typically used to determine moisture content above the water table and total porosity below the water table. Neutron logs are effective for identifying perched water tables. Neutron log information can also be used to determine lithology and conduct stratigraphic correlation of aquifers and associated formations as well as to help determine the effective porosity and specific yield of unconfined aquifers.

- Acoustic (Sonic) Logs - Measure the travel time and attenuation of an acoustic signal created by an electromechanical source in the borehole. A transmitter in the borehole converts the electrical energy to acoustic (sound) energy which travels through the formation as an acoustic pulse to one or more receivers. The acoustic energy is then converted back to electrical energy, which is measured at the surface. The acoustic wave velocity is affected by the type of material through which it passes (rock or sediment is more conductive than is pore fluid), hence it is useful in determining porosity.

  Acoustic logs can help determine fracture patterns within semiconsolidated and consolidated bedrock such as sandstone, conglomerate, and igneous rocks. Knowledge of fracture patterns in an aquifer is helpful in estimating ground-water flow, and thereby estimating the rate of plume movement. Acoustic logs can be used to locate the static water level and to detect perched water tables.

- Temperature Logs - Used to measure the thermal gradient of the borehole fluid. The sonde measures changes in temperature of the fluid surrounding it, and the log records resistivity as a function of temperature. Borehole fluid temperature is influenced by fluid movement in the borehole and adjacent strata. In general, the temperature gradient is greater in low permeability rocks than in high permeability rocks, likely due to ground-water flow. Temperature logs provide information regarding ground-water movement and water table elevation. Temperature logs are useful for detecting seasonal recharge and subsurface infiltration of irrigation and industrial wastewater runoff, and quantitative interpretation of resistivity logs.

  Temperature logs are designed to be operated from the top to the bottom of the borehole, in order to channel water past the sensor. Repeat temperature logs should be delayed until the borehole fluid has had time to reach thermal equilibrium.

Table 3 illustrates the applicability of various borehole geophysical techniques to ground-water investigations. Table 4 lists some advantages and disadvantages of borehole geophysical techniques to ground-water investigations.

3.3.3 Geophysical Techniques for Ground-Water Investigations

The following situations illustrate uses for geophysical techniques in ground-water assessment.

- To define the location, extent, and the movement of a contaminant plume, several geophysical techniques may be utilized, including EM, electrical resistivity, and possibly GPR. Resistivity and spontaneous potential (SP) logs could also be utilized as borehole geophysical methods.

- To locate faults and fracture systems, seismic refraction and reflection and EM are the preferred methods, but GPR, electrical resistivity and acoustic logs could also be used.

- The mapping of grain size distribution in unconsolidated sediments is not possible with any geophysical technique. It is possible, however, to identify different soil types of different grain sizes (e.g., sand, silt, and clay). Seismic reflection and refraction, GPR, and gravimetric surveys may be used to identify differing formations. Several borehole geophysical techniques could also be utilized in this type of analysis, including gamma, gamma-gamma, neutron porosity, resistivity, and SP logs.

- Definition of lithologic boundaries may be accomplished with seismic reflection and refraction and with GPR techniques. When using borehole geophysics, resistivity, SP, and acoustic logs are useful.

- For mapping water tables, GPR and electrical resistivity are preferred but seismic refraction and reflection and gravimetric surveys may also be used. If using borehole geophysics, direct measurement or temperature logs would be the method of choice. Resistivity and SP logs could also be used.

- To define the bedrock topography, seismic refraction and reflection, GPR and gravimetric surveys may be used.

- For delineating stratigraphic layers or subsurface features, such as buried stream channels and lenses, seismic refraction and reflection, electrical resistivity, gravimetric surveys, and possibly GPR could be used.

3.4 EXAMPLE SITE

3.4.1 Selection of Field Analytical Screening Techniques

Phase 1 sampling identified the sources and types of on-site contaminants in order to establish a threat. Hazard categorization techniques, organic vapor detecting instruments (FID and PID), and radiation and cyanide monitors were utilized to tentatively identify containerized liquid wastestreams in order to select initial judgmental soil sampling locations. During Phase 2 sampling, a portable XRF unit was used to determine the extent of soil contamination and to identify additional "hot spots." A FID and PID continued to be utilized throughout all field activities for health and safety monitoring during Phases 1 through 3.

The portable XRF for soil screening was also used during monitoring well installation. Continuous split spoon samples were collected during advancement of the boreholes. Each spoon was sampled and screened in the field using the XRF unit. Selected samples (one per borehole location) were submitted to the laboratory for confirmation analysis. One off-site sample was selected by the field geologist based on field observations and professional judgment.

Ground-water samples were screened in the field for pH, specific conductivity, and temperature using a three-in-one monitoring instrument. The instrument probe was placed into a clean glass jar containing an aliquot of the ground-water sample. The instrument was decontaminated prior to and after each sample screening.

3.4.2 Selection of Sampling Equipment

Dedicated plastic scoops were used for Phase 1 soil sampling. Phase 1 ground-water samples were collected directly from the residential taps into sample containers. For Phase 2, soils were collected from the near surface (0-4 inches) and at depth. Stainless steel trowels were used to retrieve shallow soil samples. Subsurface samples were collected by advancing boreholes using a hand-operated power auger to just above the sampling zone and then using a stainless steel split spoon to retrieve the soil. The split spoon was advanced with a manual hammer attachment.

Monitoring wells were installed using a dual-tube, air percussion drill rig. Borehole soil samples were retrieved using 2-foot stainless steel split spoon samplers. Soil from the split spoons was transferred to sample containers using disposable plastic scoops. Monitoring well installation is described further in Section 4.6.1.

Ground water was sampled in Phase 2 from the monitoring wells installed on site. First, monitoring wells were purged using a 1.5 gallon per minute (gpm) submersible rotary pump with flexible PVC outflow hose and safety cable. The pump and hose were decontaminated between well locations by pumping deionized water through the system. A similar pump and hose system was used to perform the hydraulic (pumping) test. The pumps are operated by a gas-powered generator placed near the well location.

The ground-water samples were obtained using dedicated bottom-fill Teflon® bailers. The bailer was attached to nylon rope, which was selected because less material would be adsorbed onto the nylon and brought out of the well. Residential ground-water samples were collected directly into the sample containers from the kitchen sink tap. Water level and depth measurements were obtained from monitoring wells using decontaminated electronic measuring equipment.

3.4.3 Selection of Geophysical Methods

The GPR instrument delineated buried trench and lagoon boundaries. The EM meter detected subsurface conductivity changes, thereby identifying buried metal containers and contaminants. The EM-31D, a shallower-surveying instrument than the EM-34, was selected because of the instrument's maneuverability and ease of use, and because the expected contaminant depth was less than 10 feet.

Table 1: Applicability of Surface Geophysical Techniques to Ground-Water Investigations

	Seismic Reflection	Seismic Refraction	Electromagnetic Conductivity	Magnetic Investigations	Ground Penetrating Radar	Electrical Resistivity	Gravi-metric Surveys
Contaminant Plume Delineation			P		A	P	
Faults/Fracture Detection	P		P		A	A	A
Lithologic Boundary Delineation	P	P			A		
Bedrock Topography Delineation	P	P			A		A
Stratigraphic Mapping	P	P			A	P	P
Water Table Mapping	A	A			P	P	A
Soil Type of Unconsolidated Sediments	P	P			P		P
Metallic Detection			P	P	P	A	
Non-Metallic Detection						P	
Seepage Detection			A		P	A	
Buried Structure Detection				A	A		
P - Preferred Method				A - Applicable Method (in most cases)			

Table 2: Advantages and Disadvantages of Surface Geophysical Techniques to Ground-Water Investigations

	Advantages	Disadvantages
Seismic Reflection	• Ability to discern discrete layers • Less offset space is required than for refraction	• Velocities 10-20% of true velocities • Data collection and interpretation are more labor intensive and complex than for refraction • Depth data not as precise as refraction • Signal enhancement needed to identify reflected waves
Seismic Refraction	• Relatively precise depth can be determined • Provide subsurface data between boreholes • Ability to map water table and top of bedrock	• Data collection can be labor intensive • Large geophone line lengths needed
Electromagnetic Conductivity	• Lightweight, portable equipment • Continuous or quick scan survey • Rapid data collection	• Interference from cultural noise and surface metal objects • Limited use where geology varies laterally
Magnetic Investigations	• Can survey large area quickly and cost effectively • Little site preparation needed	• Interference from cultural noise, and large metal objects • Unable to differentiate between steel anomalies
Ground Penetrating Radar	• Can survey large area quickly • Continuous real-time data display • Quick data processing	• Interference from cultural noise, uneven terrain, and vegetation • Clay content and shallow water table inhibit radar penetration
Gravimetric Surveys	• Can survey large area quickly • Little site preparation	• Accurate elevations require surveying • Should be used only as preliminary screening tool
Electrical Resistivity	• Quantitative modeling can estimate depth, thickness, and resistivity of subsurface layers	• Interference from cultural noise, surface metal objects, and industry • A minimum of two to three crew members is required • Surveys are labor intensive

Table 3: Applicability of Borehole Geophysical Techniques to Ground-Water Investigations

	Resistance Logs	Spontaneous Potential Logs	Gamma Logs	Gamma-Gamma Logs	Neutron Logs	Temperature Logs	Acoustic Logs
Contaminant Plume Delineation	P	P	P			P	
Faults/Fracture Detection							P
Lithologic Boundary Delineation	P	P	P	P	A		A
Bedrock Topography Delineation	P	P	P	P	A		P
Stratigraphic Mapping	P	P	P	P	P		P
Water Table Mapping	A	A			P	P	A
Soil Type of Unconsolidated Sediments	P	P	P	P	P		
P - Preferred Method			A - Applicable Method (in most cases)				

Table 4: Advantages and Disadvantages of Borehole Geophysical Techniques to Ground-Water Investigations

	Advantages	Disadvantages
Resistance Logs	• Indicates lithologic changes • Indicates amount and type of subsurface fluid (water quality)	• Can only be run in uncased borehole • Difficult to interpret lithology when using drilling fluid with clay additives
Spontaneous Potential Logs	• Can be run in conjunction with resistance log • Indicates lithologic changes and permeable beds	• Can only be run in uncased borehole • Interpretation for water well often more difficult than for oil well
Gamma Logs	• Easy to operate • Can be run in open or cased borehole • Qualitative guide for stratigraphic correlation and permeability	• Affected by changes in borehole diameter and borehole media • Feldspar and volcanic rock fragments make interpretation difficult
Gamma-Gamma Logs	• Can identify lithology and calculate porosity when fluid and grain density are known	• Porosity readings of low density materials can be erroneously high
Neutron Logs	• Can determine total porosity in saturated zone • Can determine moisture content in unsaturated zones • Can be run in open or cased borehole	• Radioactive source requires special handling by trained personnel • Logging can be somewhat complex
Acoustic Logs	• Useful for determining relative porosity • Indicates fracture patterns in aquifer • Can indicate static water level and perched water tables	• Clays may distort readings
Temperature Logs	• Can indirectly measure permeability • Provides information regarding ground-water movement and water table elevation	• Delay repeat logs until borehole fluid reaches thermal equilibrium

4.0 GROUND-WATER SAMPLE COLLECTION AND PREPARATION

4.1 INTRODUCTION

During a response action, proper field sample collection and preparation is as important as proper sampling equipment selection. Sample collection refers to the physical removal of an aliquot of ground water from its source (i.e., aquifer) for the purpose of either screening or laboratory analysis. Ground-water sample collection procedures should be selected so that the resultant sample is representative of the aquifer or particular water zone being sampled. Field sample preparation refers to all aspects of sample handling from collection to the time the sample is received by the laboratory. This chapter provides information on sample collection and preparation for ground water.

The representativeness of a ground-water sample is greatly influenced by the sampling device used and the manner in which the sample is collected. Proper training and use of SOPs will limit variables and enhance sample representativeness. Selection of ground-water sampling devices such as bailers and pumps should be site-specific and dependent on well diameter, yield, lift capacity, and the analytes being sampled. Excessive aeration should be minimized to preserve volatile constituents. Where possible, the bailer or pump used should be compatible with the analyte(s) of concern.

4.2 STATIC WATER LEVEL

Prior to sampling, the static water level elevation in each well should be measured. All measurements should be completed prior to the sampling event so that static water levels will not be affected. The water level measurements are necessary to establish well purging volumes. These measurements can also be used to construct water table or potentiometric surface maps and hence determine local ground-water flow gradient. Measure the depth to standing water and the total depth of the well to calculate volume of stagnant water in the well for purging. See ERT SOP #2151 for detail on collecting static water level measurements.

4.3 WELL PURGING

There is little or no vertical mixing of water in a nonpumping well, therefore stratification occurs. The well water in the screened section mixes with the ground water due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. The stagnant water may contain foreign material inadvertently or deliberately introduced from the surface, resulting in unrepresentative data. Adequate well purging prior to sample withdrawal will safeguard against collecting nonrepresentative stagnant water samples.

Well purging techniques are specific to the following well types.

- Residential, Commercial, and Public Supply Wells - Sample residential, commercial, and public supply wells as near to the wellhead as possible and at a point before treatment, such as filtering and water softening units, whenever possible. Open the tap to a moderate flow and purge for approximately 15 minutes. If this is not possible, a 5-minute purge is considered a minimum. As an alternative to a minimum volume, purging can be conducted until the field parameters pH, temperature, and specific conductivity have stabilized (see Section 4.3.1).

- Monitoring Wells - To obtain a representative sample from a monitoring well, it is necessary to evacuate the standing water in the well casing prior to sampling. The minimum recommended amount that should be purged from a monitoring well is one casing volume, but three to five casing volumes of standing water should be evacuated where possible in order to obtain a ground-water sample representative of the aquifer. In a high yield aquifer where there is no standing water above the screened section of the well casing, purging three volumes is not as critical as in lower yield aquifers. (The faster recharge rate limits the amount of time that the water has to interact with the atmosphere and casing materials.) If the well is purged dry, it should be considered sufficiently purged for sampling (refer to Section 4.3.2 for additional information).

The amount of purging a well receives prior to sample collection depends on the intent of the sampling as well as the hydrogeologic conditions. When the sampling objective is to assess overall water resource quality, long pumping periods may be required to obtain a sample that is representative of a large volume of the aquifer. The pumped volume is determined prior to sampling, or the well is pumped until the stabilization of parameters such as temperature, specific conductivity, and pH has occurred.

Monitoring to define a contaminant plume requires a representative sample of a small volume of the aquifer. These circumstances require that the well be pumped enough to remove the stagnant water but not enough to induce flow from other areas. Generally, three well volumes are considered effective. Otherwise, the appropriate volume to be removed prior to sampling can be calculated, based on aquifer parameters and well dimensions.

Well purging devices include bailers, submersible pumps (rotary-type), non-gas contact bladder pumps, suction pumps, and hand pumps. See ERT SOP #2007 for specific guidelines on purging wells prior to sampling and for more detail on each purging device.

4.3.1 Stabilization Purging Technique

The stabilization technique is an alternative to volume purging. This method requires that several field parameters be continuously monitored during purging. When these parameters stabilize, begin sampling. The parameters used for this method are pH, temperature, and specific conductivity. Stabilization of these parameters indicates that the standing water in the monitoring well has been removed and that a representative sample of the aquifer water may now be collected. This method of purging is useful in situations where it is not feasible to evacuate three casing volumes from the well prior to sampling (e.g., large casing diameter, extremely deep, and active supply wells). See ERT SOP #2007 for specific volume and stabilization purging techniques.

4.3.2 Wells that Purge Dry

A well that is purged dry should be evacuated and allowed to recover prior to sample withdrawal. If the recovery rate is fairly rapid and time allows, evacuation of more than one volume of water is desirable. If the recovery rate is slow, the first recharge can be considered suitable for sample collection.

4.4 GROUND-WATER SAMPLE COLLECTION

In order to maintain sample representativeness, dedicated samplers should be used for each well whenever possible. When not possible, the sampler should be decontaminated after each sample collection and sufficient QA/QC blank samples should be collected to assess potential cross-contamination.

After well purging is complete, collect and containerize samples in the order of most volatile to least volatile, such as:

- Volatile organic analytes (VOAs)
- Purgeable organic carbon (POC)
- Purgeable organic halogens (POX)
- Total organic halogens (TOX)
- Total organic carbon (TOC)
- Extractable organic compounds
- Total metals
- Dissolved metals
- Phenols
- Cyanide
- Sulfate and chloride
- Turbidity
- Nitrate and ammonia
- Radionucliides

See ERT SOP #2007 for specific detail on filling sample containers, with special considerations for VOA sampling.

If the contaminants in the water column are stratified (e.g., DNAPLs, LNAPLs), be certain to use an appropriate sampling device. Modify, where possible, standard sampling procedures to collect the sample from the suspected depth for the contaminant layer. It may be necessary to lower the bailer used for sample collection to a particular depth in the well, or to use a point-source bailer or other discrete-depth sampling device.

After a monitoring well is initially constructed, it should be developed and purged to remove invaded water. The well should sit idle for at least two weeks to allow the water level to fully stabilize and the suspected stratified layers to settle out. Measurement of the thickness of a floating (LNAPL) layer may be accomplished in several ways. An indicator gel, chalk or paste may be applied to an incremented steel tape.

The indicator changes color in the presence of water or the immiscible layer, depending on the specific use of the indicator compound. For example, water-level indicator gel is used to determine the depth to the water surface. A weighted float is then used to determine the depth to the top of the LNAPL layer. The difference between these two readings is the thickness of the floating layer.

An electronic monitoring device called an interface probe is also available for the LNAPL layer measurement. This device, like an electric water-level sounder, is lowered into the well along an electronic wire/line. When the probe contacts the surface (the LNAPL layer) a sound is generated. As the sampler continues to lower the probe, a different electronic sound is emitted when the water surface, or water/oil interface, is reached. The line of the device is incremented, like a water-level sounder, so the layer thickness can be determined. Standard electric water-level sounding devices, however, will not work properly for these measurements. The interface probe is a specialized instrument which is commonly available and used at fuel oil/ground-water contamination sites.

A sample of a floating layer may be obtained using a bottom-fill bailer. Care should be taken to lower the bailer just through the floating layer, but not significantly down into the underlying ground water. (A clear bailer is preferable for this activity.)

For sampling sinking layers, a discrete-depth-capable sampling device, such as a packer pump or syringe sampler, is best suited. When these specialized devices are not available, depending on the sampling parameters, standard devices may be used. For example, samples at the bottom of the screen or at some intermediate location may also be obtained with a standard bailer and a second well casing. In order to avoid mixing the waters, a separate casing is temporarily lowered inside the permanent well casing. The temporary casing is equipped with an easily removed cap on the bottom so that no fluid enters the casing until it has reached the desired sampling depth. The cap is then freed from the bottom of the inner casing, allowing water to enter to be sampled by a bailer. At significant depths below the nonaqueous layer, several bailers full of water may need to be withdrawn and discarded before the sample is obtained from a fresh formation sample.

When a temporary casing and all other specialized equipment is unavailable, a standard bailer alone may be used. Collect a water sample from the well and transfer it to the sample container. Allow the sample to settle in the sample container into the separate stratified layers. The analytical laboratory may then decant, as appropriate, to obtain a sample of the desired layer. More commonly, the parameters of concern in the stratified layers are simply included in the laboratory analysis of the sample as a whole without the need to separate into unique layers. In this last example, care must be taken to allow the bailer to reach the desired depth in the water column to insure collecting any dense layers at the bottom of the well. (See Section 2.4 for additional discussion on sampling concerns and the physiochemical nature of contaminants.)

4.5 GROUND-WATER SAMPLE PREPARATION

This section addresses appropriate ground-water sample preparation and handling techniques. Proper sample preparation and handling maintain sample integrity. Improper handling can render samples nonrepresentative and unsuitable for analysis.

The analyses for which a sample is being collected determines the type of bottles, preservatives, holding times, and filtering requirements. Samples should be collected directly into appropriate containers that have been cleaned to EPA or other required standards. Check to see that a Teflon® liner is present in the sample bottle cap, if required.

Samples should be labeled, logged, and handled correctly, including appropriate chain-of-custody documentation. Place samples in coolers to be maintained at 4EC. Ship samples to arrive at the designated analytical laboratory well before their holding times are expired. It is preferable that samples be shipped or delivered daily to the analytical laboratory in order to maximize the time available for the laboratory to do the analysis.

Certain conditions may require special handling techniques. For example, treatment of a sample for VOAs with sodium thiosulfate preservative is required if there is residual chlorine in the water (e.g., a public water supply) that could cause free radical chlorination and change the identity of the original contaminants. (The preservative should not be used if there is no chlorine in the water.) All such special requirements must be determined prior to conducting fieldwork.

Sample preparation for ground water may include, but is not limited to:

- Filtering
- Homogenizing/Aliquotting
- Splitting
- Final Preparation

4.5.1 Filtering

Samples may require filtering, such as for total metals analysis. Samples collected for organic analyses should not be filtered. Two types of filters may be used, which must be decontaminated prior to use. A barrel filter works with a bicycle pump, which builds up positive pressure in the chamber containing the sample and then forces it through the filter into a container placed underneath. A vacuum filter has two chambers; the upper chamber contains the sample, and a filter divides the chambers. Using a hand pump or a Gilian®-type pressure pump, a vacuum is created in the upper chamber and the sample is filtered into the lower chamber. Preservation of the sample, if necessary, should be done after filtering.

See ERT SOP #2007, Section 2.7.5, for more detail on filtering ground-water samples.

4.5.2 Homogenizing/Aliquotting

Homogenizing, or aliquotting, is the mixing or blending of a grab sample to distribute contaminants uniformly. Ideally, proper homogenizing ensures that all portions of the sample are equal or identical in composition and are representative of the total sample collected. Incomplete homogenizing can introduce sampling error. Homogenizing disturbs the ground-water sample, so it is not appropriate for VOC sampling.

Homogenizing is done during only one sampling event per well location, and only after the VOC sample portions have first been filled. It may be utilized for wells with extremely low yield and potentially insufficient sample volume to fill all sample containers provided by the laboratory. In some low yielding wells, the percentage of suspended material in a bailer-full of sample will increase as sampling proceeds. Homogenizing ensures that at least a minimum volume is aliquotted per analytical parameter, and the percentage of suspended material is equitably divided among all containers (excluding VOCs).

4.5.3 Splitting

Split samples are created when the samples have to be separated into two or more equivalent parts and analyzed separately. Split samples are most often collected in enforcement actions to compare sample results obtained by EPA with those obtained by the potentially responsible party. Split samples also provide measures of sample variability and analytical error. Fill two sample collection jars simultaneously, alternating the sample stream or bailer full of sample between them.

4.5.4 Final Preparation

Final preparation includes preserving, packaging, and shipping samples.

Sample preservation is used to retard chemical breakdown of the sample. Preservation of ground-water samples includes controlling pH with chemical preservatives, refrigerating samples, and protecting samples from light.

Select sample containers on the basis of compatibility with the material being sampled, resistance to breakage, and capacity. Appropriate sample volumes and containers will vary according to the parameters being analyzed. Actual sample volumes, appropriate containers, and holding times are specified in the U.S. EPA *Compendium of ERT Ground-Water Sampling Procedures*, OSWER Directive 9360.4-06. Package all samples in compliance with current International Air Transport Association (IATA) or U.S. Department of Transportation (DOT) requirements, as applicable. Packaging should be performed by someone trained in current DOT shipping procedures.

See ERT SOP #2007, Section 2.3 for more detail on ground-water sample preparation.

4.6 EXAMPLE SITE

4.6.1 Sample Collection

During Phase 1 and Phase 2, surface soil samples were collected from shallow locations. The samples were collected as grab samples. The sample locations were cleared of surface debris, then samples were retrieved with disposable plastic scoops and placed directly into sample containers. During Phase 2, subsurface soil samples were collected at the soil boring/well installation locations, using stainless steel split spoon samplers. The split spoon samples were collected using a hand-held power auger to advance the hole. A 2-foot stainless steel split spoon sampler with hammer attachment was then pushed into the

hole. The soil sample was retrieved from the split spoon sampler using a disposable plastic scoop to transfer the soil into a stainless steel bowl. Several scoopfuls were collected along the length of the split spoon sampler and composited in the bowl. The composite sample was then transferred directly into the sample container using the disposable plastic scoop.

Phase 1 and Phase 2 residential well ground-water samples were collected directly from the kitchen taps of homes using private wells near to the site. The configuration of the residential system was noted in the logbook prior to sampling. If present, water softeners were taken off line. Any screen or filter was first removed from the tap, which was allowed to run for a minimum of five minutes prior to sampling. The samples were collected directly into the sample containers.

Fifteen monitoring wells were installed at the site at locations described in Section 2.5.6. The wells were drilled with a dual-tube, air percussion rig. Each boring was completed to a 9.5-inch diameter. After completion of the boring, 4-inch Schedule 40 PVC casing and 0.010 slot screen were installed in lengths appropriate to each well. Shallow wells were drilled to approximately 40 feet below grade surface (BGS) and bedrock contact wells were drilled to approximately 55 to 60 feet BGS. Continuous split spoon sampling was performed at each well location from 4 feet BGS to well completion depth. The boreholes were grouted from the bottom to the top of the lower confining layer, then 10 feet of screen were set above the grouted portion. PVC casing was set above the screen to above the ground surface. Casing was extended to accommodate a 2-foot stick-up above grade, and then capped. A 6-inch diameter metal outer casing with locking cover was installed over the well casing stick-up and secured 2 feet BGS in concrete. A concrete spill pad was then constructed around each well outer casing to prevent re-infiltration at the well point. Upon completion, all monitoring wells were developed by purging using a decontaminated rotary pump and flexible PVC disposable hose.

A Team geologist supervising the monitoring well installation logged each borehole soil lithology from the retrieved split spoon samplers collected during drilling of the boreholes. The geologist scanned each sampler with a PID immediately upon opening (into halves) for health and safety monitoring. All logging was accomplished utilizing the Unified Soils Classification System standard method. Figure 3 provides an example of a soil boring and monitoring well completion log.

Soil samples were then collected in wide-mouth clear glass jars by transferring a portion of each lithologic unit in the split spoon with a disposable plastic scoop and compositing the sample in the jar. At the completion of each borehole, each sample was screened in the field using the XRF unit. Select samples (one per borehole location) were forwarded to the laboratory for confirmation analysis. Split spoon samplers were decontaminated after each use.

Upon completion and development, the 15 on-site monitoring wells were sampled for ground-water analysis. The well caps were brushed and cleaned off prior to opening. Immediately upon removing the well cap, a PID was operated over the opening to determine VOC levels, if any, in the breathing zone. The VOC monitoring was performed to establish if a higher level of respiratory protection was required. Depth to water level measurements were then taken of each well to the nearest 0.01 ft. The total depth of the well was obtained with a depth sounder. The volume of water in the well was then calculated using the formula below. For a four-inch well, well volume would equal 0.632 gallon/ft.:

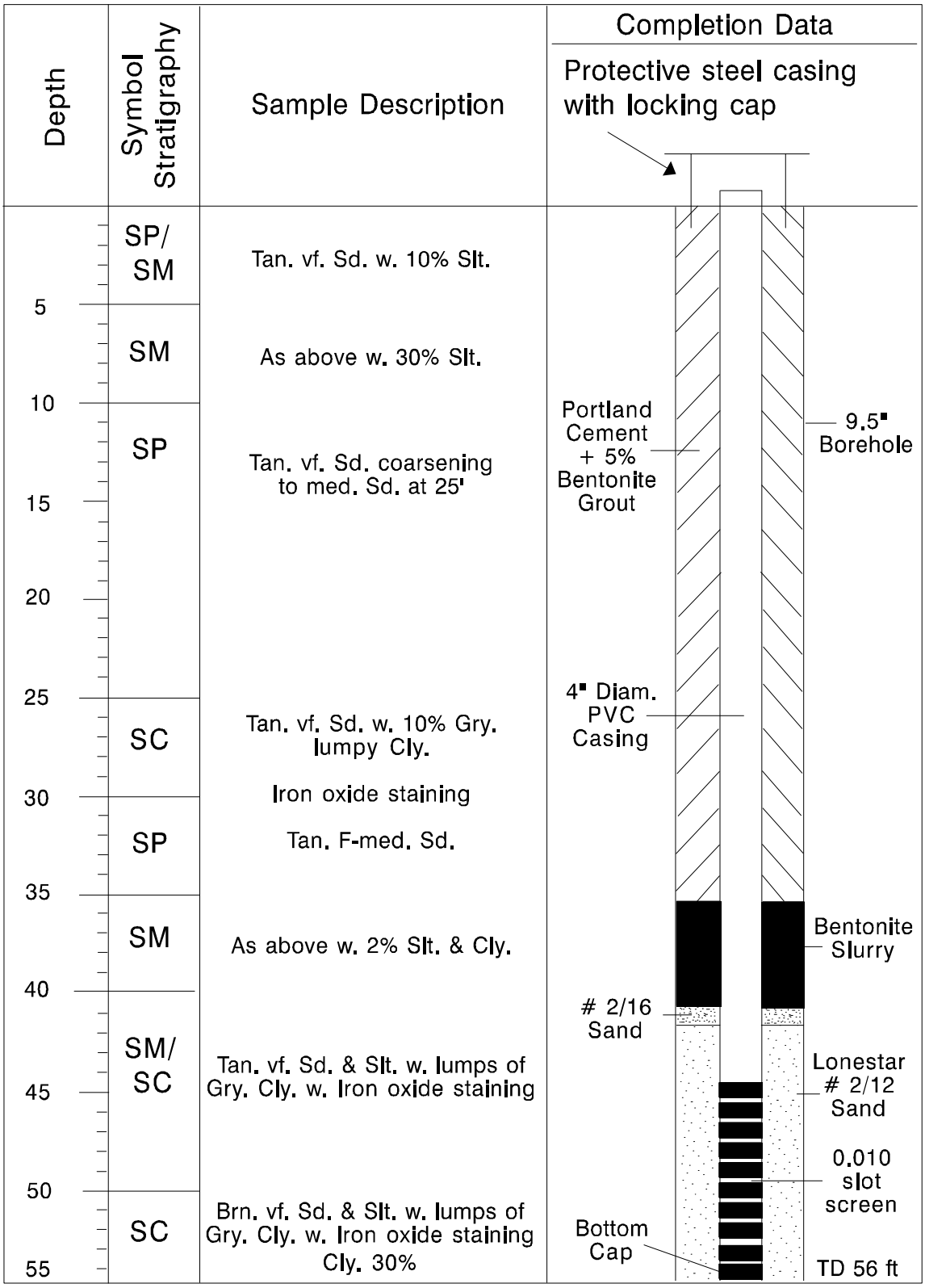
$$\text{Well volume} = \pi \times (\text{radius of well})^2 \times \text{height of water column} \times 7.48 \text{ gallon/ft}^3$$

(conversion factor for ft^3 to gallons)

Each monitoring well was purged prior to obtaining a representative sample. Wells with sufficient yield were purged three well volumes. Low-yielding wells were purged once to dryness. (Most wells on site are low-yielding.) Purging was completed using a 1.5 gpm decontaminated submersible (rotary-type) pump with flexible PVC outflow hose and safety cable. The pump was slowly lowered to a point approximately 3 feet above the bottom of the well. With the known flow rate, length of pumping required was calculated. Purge water was pumped into 55-gallon steel drums. (The drums were staged and later disposed of properly based on the results of analysis of their contents.) Low-yielding overburden wells were purged with a decontaminated stainless steel bottom-fill bailer and polypropylene rope until dry. All wells were allowed to recover overnightbefore sample collection, or until sufficient water was present to complete a sample set.

Each monitoring well was sampled after purging and recovery. Ground-water samples were collected using dedicated disposable Teflon® bailers. Each bailer was attached to a clean polypropylene rope and intro-

Figure 3: Soil Boring/Monitoring Well Completion Log



duced into the well. The bailer was lowered slowly to the approximate mid-point of the well. Once the sample was collected, care was taken not to agitate the water while pouring directly into the appropriate sample containers. An additional ground-water aliquot was placed into a large wide-mouth glass jar in order to obtain conductivity, temperature, and pH measurements. These measurements were recorded in the field logbook.

After well sampling, a hydraulic (pumping) test was performed to determine aquifer characteristics for mathematical modeling of potential contaminant plume migration. The hydraulic test was conducted using one well as a pumping well with three observation wells. The pumping well was purged at a rate of 22 gpm for 30 hours. All wells (observation and pumping) were monitored during pumping and for 4 hours after pumping ceased. Drawdown data from the wells were used to calculate the characteristics of the aquifer.

To generate accurate gradient and well location maps, the 15 newly installed monitoring wells were surveyed for vertical location using feet above mean sea level (MSL) units. Vertical elevations were taken at a mark on the top of the inner casing of each monitoring well, to establish a permanent location for all future water level measurements and elevations. A permanent benchmark was located near to the site by the survey team to determine all the well elevations. Elevations were then measured against the benchmark and mapped in MSL units.

All non-disposable equipment, including drill rig and equipment, stainless steel bailers, pumps, water level indicators, and depth sounders, were decontaminated between each location and prior to the first sampling event each day.

4.6.2 Sample Preparation

All sample containers were supplied by the contracted analytical laboratory. Chemical preservation was also provided by the laboratory through pre-preserved bottleware. Sample containers for ground-water samples consisted of:

- 1-liter polyethylene bottles for total chromium, pre-preserved with reagent-grade nitric acid lowering the pH to less than 2 after addition of the sample
- 1-liter polyethylene bottles for hexavalent chromium
- 1-liter polyethylene bottles for cyanide, pre-preserved with sodium hydroxide

Sample containers for soils consisted of 8-ounce glass jars with Teflon® caps for all parameters.

All samples were preserved to 4E C by placing them in coolers packed with "blue ice" immediately after collection and during shipment. (The laboratory was responsible for cooling and refrigeration of samples upon arrival.)

The samples were packaged in compliance with IATA requirements for environmental samples. Chain-of-custody paperwork was prepared for the samples. Laboratory paperwork was completed as appropriate and the samples were shipped to the predesignated laboratories for analysis. Holding times for total chromium and cyanide are less than six months, but hexavalent chromium has a holding time of less than 24 hours. This was coordinated in advance with the analytical laboratory and required daily ground delivery of samples to the laboratory.

Because many of the ground-water samples from the on-site wells were extremely turbid, the non-volatile portions of samples were filtered in the laboratory prior to analysis. Filtering was accomplished using a barrel filtering device with a minimum pore size of 0.45 microns. Samples for chromium analysis were split and filtered so that dissolved and particulate chromium could be differentiated. Dissolved chromium is of concern because of its ability to be transported in ground water.

5.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

5.1 INTRODUCTION

The goal of representative sampling is to obtain analytical results that accurately depict site conditions during a defined time interval. The goal of quality assurance/quality control (QA/QC) is to implement correct methodologies which limit the introduction of error into the sampling and analytical procedures, and ultimately into the analytical data.

QA/QC samples evaluate three types of information: 1) the magnitude of site variation; 2) whether samples were cross-contaminated during sampling and sample handling procedures; and 3) whether a discrepancy in sample results is a result of laboratory handling and analysis procedures.

5.2 DATA CATEGORIES

EPA has established data quality objectives (DQOs) which ensure that the precision, accuracy, representativeness, and quality of environmental data are appropriate for their intended application. Superfund DQO guidance defines two broad categories of analytical data: *screening* and *definitive*.

Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation than definitive data. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, rather than elaborate extraction/digestion and cleanup. At least 10 percent of the screening data are confirmed using the analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be data of known quality. To be acceptable, screening data must include the following: chain-of-custody, initial and continuing calibration, analyte identification, and analyte quantification. Streamlined QC requirements are the defining characteristic of screening data.

Definitive data are generated using rigorous analytical methods (e.g., approved EPA reference methods). These data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. QC measures for definitive data contain all of the elements associated with screening data, but also may include trip, method, and rinsate blanks; matrix spikes; performance evaluation samples; and replicate analyses for error determination.

For further information on these QA/QC objectives, please refer to U.S. EPA *Data Quality Objectives Process for Superfund*, pp. 42-44.

5.3 SOURCES OF ERROR

There are many potential sources of data error in ground-water sampling. The following is a list of some of the more common potential sources of error:

- Sampling design
- Sampling methodology
- Analytical procedures
- Seasonal variations

See U.S. EPA *Data Quality Objectives Process for Superfund*, pp. 29-36, for more information on error.

5.3.1 Sampling Design

The sampling design should utilize approved SOPs and previously approved sampling designs to ensure uniformity and comparability between samples. The actual sample collection process should be determined prior to sampling. Sampling equipment and techniques must be standardized for like sampling situations.

The sampling design should fulfill sampling and data quality objectives. The quality assurance objectives selected should be built into the sampling design, including all necessary QA/QC samples.

Sampling design errors for ground water include: well selection, well location, well construction and development, background sample location, and equipment (material and type).

5.3.2 Sampling Methodology

Sampling methodology and sample handling procedures have possible sources of error, including: cross-contamination from inappropriate use of sample collection equipment; unclean sample containers; improper sampling equipment decontamination; and improper shipment procedures. Procedures for collecting, handling, and shipping samples should be standardized to allow easier identification of any source(s) of error, and to minimize the potential for error. Use approved SOPs to ensure that all given sampling techniques are performed in the same manner, regardless of the sampling team, date, or location of sampling activity. Use field blanks, replicate samples, trip blanks, and rinsate blanks (discussed in Section 5.4) to identify errors due to improper sampling methodology and sample handling procedures. An example of a sampling methodology error for ground water is inappropriate purging.

5.3.3 Analytical Procedures

Analytical procedures may introduce errors from laboratory cross-contamination, inefficient extraction, and inappropriate methodology. Matrix spike, laboratory duplicate, performance evaluation, and laboratory control samples help to distinguish analytical error from sampling error.

5.3.4 Seasonal Variations

Seasonal variations are not controllable but must be taken into consideration as a source of error during ground-water assessments. Changes in flow direction or volume can redistribute contaminants throughout a site, making assessment difficult. Plan sampling events in order to minimize the effects of seasonal variations, if possible.

5.4 QA/QC SAMPLES

QA/QC samples are collected at the site or prepared for or by the laboratory. Analysis of the QA/QC samples provides information on the variability and usability of sampling data, indicates possible field sampling or laboratory error, and provides a basis for future validation and usability of the analytical data. The most common field QA/QC samples are field replicate, background, and rinsate, field, and trip blank samples. The most common laboratory QA/QC samples are performance evaluation (PE), matrix spike (MS), matrix spike duplicate (MSD), and laboratory duplicate samples. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet site-specific quality assurance objectives.

Ground water is typically characterized by low or trace concentrations of contaminants, making precision and accuracy more important than for samples with higher concentrations (e.g., waste). Frequent field blanks are thus appropriate in ground-water sampling.

The following sections briefly describe the most common types of QA/QC samples appropriate for ground-water sampling.

5.4.1 Field Replicate Samples

Field replicates, also referred to as field duplicates and split samples, are field samples obtained from one sampling point, homogenized (where appropriate), divided into separate containers, and treated as separate samples throughout the remaining sample handling and analytical processes. Use replicate samples to assess error associated with sample methodology and analytical procedures. Field replicates can also be used when determining total error for critical samples with contamination concentrations near the action level. In such a case, a minimum of eight replicate samples is recommended for valid statistical analysis. Field replicates may be sent to two or more laboratories or to the same laboratory as unique samples. For total error determination, samples should be analyzed by the same laboratory. Generally, one field replicate per 20 samples per day is recommended.

5.4.2 Background Samples

Defining background conditions may be difficult because of natural variability and the physical characteristics of the site, but it is important in order to quantify true changes in contaminant concentrations due to a source or site. Defining background conditions is critical for avoiding false positives and for enforcement purposes in naming responsible parties. Background sampling is often required in ground-water sampling to verify plume direction, ambient conditions, and attribution of sources. A properly collected background sample serves as the baseline for the measure of contamination throughout the site. Ground-water background sample locations should be chosen carefully, usually upgradient from the suspected source of contamination where there is little or no chance of migration of contaminants of

concern. Collect at least one background sample for comparison, although additional samples are often warranted by site-specific factors such as natural variability of local geology and multiple sources. Background samples may be collected to evaluate potential error associated with sampling design, sampling methodology, and analytical procedures. Refer to U.S. EPA "Establishing Background Levels" fact sheet, OSWER Directive 9285.7-19FS, for detailed discussion on the proper selection and considerations of a background sample location.

5.4.3 Rinsate Blank Samples

A rinsate blank, also referred to as an equipment blank, is used to assess cross-contamination from improper equipment decontamination procedures. Rinsate blanks are samples obtained by running analyte-free water over decontaminated sampling equipment. Any residual contamination should appear in the rinsate sample data. Analyze the rinsate blank for the same analytical parameters as the field samples collected that day. Handle and ship the rinsate like a routine field sample. Where dedicated sampling equipment is not utilized, collect one rinsate blank per type of sampling device per day.

5.4.4 Field Blank Samples

Field blanks are samples prepared in the field using certified clean water (HPLC-grade water (carbon-free) for organic analyses and deionized or distilled water for inorganic analyses) which are then submitted to the laboratory for analysis. A field blank is used to evaluate contamination or error associated with sampling methodology, preservation, handling/shipping, and laboratory procedures. Handle, ship, and analyze a field blank like a routine field sample. Submit one field blank per day.

5.4.5 Trip Blank Samples

Trip blanks are samples prepared prior to going into the field. They consist of certified clean water (HPLC-grade) and are not opened until they reach the laboratory. Utilize trip blanks for volatile organic analyses only. Handle, transport, and analyze trip blanks in the same manner as the other volatile organic samples collected that day. Trip blanks are used to evaluate error associated with shipping and handling and analytical procedures. A trip blank should be included with each shipment.

5.4.6 Performance Evaluation/ Laboratory Control Samples

A performance evaluation (PE) sample evaluates the overall error contributed by the analytical laboratory and detects any bias in the analytical method being used. PE samples contain known quantities of target analytes manufactured under strict quality control. They are usually prepared by a third party under an EPA certification program. The samples are usually submitted "blind" to analytical laboratories (the sampling team knows the contents of the samples, but the laboratory does not). Laboratory analytical error may be evaluated by the percent recoveries and correct identification of the components in the PE sample. *Note: Even though they are not available for all analytes, analyses of PE samples are recommended in order to obtain definitive data.*

A blind PE sample may be included in a set of split samples provided to the potentially responsible party (PRP). The PE sample will indicate PRP laboratory accuracy, which may be critical during enforcement litigation.

A laboratory control sample (LCS) also contains known quantities of target analytes in certified clean water. In this case, the laboratory knows the contents of the sample (the LCS is usually prepared by the laboratory). PE and LCS samples are not affected by matrix interference, and thus can provide a clear measure of laboratory error.

5.4.7 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike and matrix spike duplicate samples (MS/MSDs) are field samples that are spiked in the laboratory with a known concentration of a target analyte(s) in order to determine percent recoveries in sample extraction. The percent recovery from MS/MSDs indicates the degree to which matrix interferences will affect the identification of a substance. MS/MSDs can also be used to monitor laboratory performance. When four or more pairs of MS/MSDs are analyzed, the data obtained may be used to evaluate error due to laboratory bias and precision. Analyze one MS/MSD pair to assess bias for every 20 samples, and use the average percent recovery for the pair. To assess precision, analyze at least eight matrix spike replicates from the same sample, and determine the standard deviation and the coefficient of variation. MS/MSDs are recommended for screening data and are required as one of several methods for determining analytical error for definitive

data. Since the MS/MSDs are spiked field samples, provide sufficient volume for three separate analyses (triple volume). When selecting a well for spiked samples, choose a well capable of providing steady volume.

5.4.8 Laboratory Duplicate Samples

A laboratory duplicate is a sample that undergoes preparation and analysis twice. The laboratory takes two aliquots of one sample and analyses them as separate samples. Comparison of data from the two analyses provides a measure of analytical reproducibility within a sample set. Discrepancies in duplicate analyses may indicate poor homogenization in the field or other sample preparation error, either in the field or in the laboratory.

5.5 EVALUATION OF ANALYTICAL ERROR

The acceptable level of error in sampling data is determined by the intended use of the data and the sampling objectives, including the degree of threat to public health, welfare, or the environment; response action levels; litigation concerns; and budgetary constraints.

Error may be determined with replicate samples. To evaluate the total error of samples with contaminant concentrations near the response action level, prepare and analyze a minimum of eight replicates of the same sample. Analytical data from replicate samples also serve as a quick check on errors associated with sample heterogeneity, sampling methodology, and analytical procedures. Different analytical results from two or more replicate samples could indicate improper sample preparation, or improper sample handling, shipment, or analysis.

Although a quantified confidence level may be desirable, it may not always be possible. A 95% confidence level (5 percent acceptable error) should be adequate for most Superfund activities. Note that the use of confidence levels is based on the assumption that a sample is homogeneous.

5.6 CORRELATION BETWEEN FIELD SCREENING RESULTS AND DEFINITIVE LABORATORY RESULTS

One cost-effective approach for delineating the extent of site contamination is to correlate inexpensive field screening data and other field measurements with definitive laboratory results. The relationship between the two methods can then be described by a regression analysis. The resulting equation can be used to predict laboratory results based on field screening measurements. In this manner, cost-effective field screening results may be used in conjunction with off-site laboratory analysis.

Statistical regression involves developing an equation that relates two or more variables at an acceptable level of correlation. In this case, the two variables are field screening results and definitive laboratory results. The regression equation can be used to predict a laboratory value based on the results of the screening device. The model can also be used to place confidence limits around predictions. Additional discussion of correlation and regression can be found in most introductory statistics textbooks. A simple linear regression equation can be developed on many calculators or computer databases. Consult a statistician to check the accuracy of more complex models.

Evaluation of the accuracy of a model relies in part on statistical correlation, which involves computing an index called the correlation coefficient (r) that indicates the degree and nature of the relationship between two or more sets of values. The correlation coefficient ranges from -1.0 (a perfect inverse or negative relationship), through 0 (no relationship), to +1.0 (a perfect, or positive, relationship). The square of the correlation coefficient, called the coefficient of determination, or simply R^2, is an estimate of the proportion of variance in the dependent variable. The value of an acceptable coefficient of variation depends on the sampling objectives and intended data uses. As a rule of thumb, statistical relationships should have an R^2 value of at least 0.6 to determine a reliable model. However, for health assessment purposes, the acceptable R^2 value may be more stringent (e.g., 0.8). Analytical calibration regressions have an R^2 value of 0.98 or greater.

Field screening data can be used to predict laboratory results if there is an acceptable correlation between them. The predicted values can be located on a base map and contoured. These maps can be examined to evaluate the estimated extent of contamination and the adequacy of the sampling program.

5.7 EXAMPLE SITE

5.7.1 Data Categories

Screening data which generate non-definitive, unconfirmed results were used to select analytical parameters and samples to be sent for laboratory confirmation analysis. Samples were sent to the analytical laboratory under protocols which provided definitive data. The rigorous laboratory analyses provided definitive identification and quantitation of contaminants.

5.7.2 Sources of Error

All direct reading instruments were maintained and calibrated in accordance with their instruction manuals. Many of these instruments are class-specific (e.g., volatile organic vapors) with relative response rates that are dependent on the calibration gas selected. Instrument response to ambient vapor concentrations may differ by an order of magnitude from response to calibration standards. If compounds of interest are known, site-specific standards may be prepared.

The number and location of initial field samples were based on observation and professional judgment (as outlined in Section 2.5.5). Field standard operating procedures, documented in the site sampling plan, established consistent screening and sampling procedures among all sampling personnel, reducing the chances for variability and error during sampling. Site briefings were conducted prior to all sampling and screening events to review the use of proper screening and sampling techniques.

Other steps taken to limit error included proper sample preparation, adherence to sample holding times, and the use of proper IATA shipment procedures. All off-site laboratory sample analyses were performed using approved EPA standard methods and protocols.

5.7.3 Field QA/QC Samples

Field QA/QC samples were collected during soil and ground-water sampling at the ABC Plating site. Two field replicate samples were collected for subsurface soils; two wells (one overburden and one bedrock) were selected for replicate collection and analysis of ground water. Rinsate blanks were collected from split spoon samplers, a bailer, and the submersible rotary pump after decontamination by pouring deionized water through the respective piece of equipment and then into a sample container. The field replicates and blanks were preserved and prepared as "regular" field samples. A trip blank for VOC analysis and a performance evaluation (PE) sample for metals were sent to the laboratory. (The PE sample is not affected by matrix interferences.) The trip blank was provided by the laboratory (pre-filled and preserved) and sent with the sample containers prior to sample collection. One trip blank per day was submitted to the laboratory. Additional volume was collected and provided to the laboratory for matrix spike/matrix spike duplicate analyses for one per 20 sample locations for each medium.

5.7.4 Laboratory QA/QC

Instructions on matrices, target compounds, and QA/QC criteria of particular interest were provided to the laboratory to help ensure that analytical results met the required quality assurance objectives. The laboratory analyzed for metals using the methods of inductively coupled plasma (ICP) spectrometry and atomic absorption (AA). Two SW-846 methods were employed for hexavalent chromium analysis: Method 7196, a colorimetric method, and Method 2185, a chelation method. These two methods were utilized in an attempt to better quantify hexavalent results. The presence of cyanide was confirmed in the laboratory using total and amenable cyanide analyses (colorimetric manual Method 9010).

6.0 DATA PRESENTATION AND ANALYSIS

6.1 INTRODUCTION

Data presentation and analysis techniques are performed with analytical, field screening, or geophysical results. The techniques discussed below can be used to compare analytical values, to evaluate numerical distribution of data, and to reveal the location of "hot spots," contaminant plumes, and the extent of contamination at a site. The appropriate methods to present and analyze sample data depend on the sampling objectives, the number of samples collected, the sampling approaches used, and other considerations.

6.2 DATA POSTING

Data posting involves placement of sample values on a site base map or cross-section. Data posting is useful for displaying the distribution of sample values, visually depicting the location of contamination with associated assessment data. Data posting requires each sample to have a specific location (e.g., *x*, *y*, and sometimes *z* coordinates). Ideally, the sample coordinates are surveyed values facilitating placement on a scaled map. Data posting is useful for depicting concentration values of ground-water and plume migration.

6.3 CROSS-SECTION/FENCE DIAGRAMS

Cross-section diagrams (two-dimensional) and fence diagrams (three-dimensional) depict subsurface features such as stratigraphic boundaries, aquifers, plumes, impermeable layers, etc. Two-dimensional cross-sections may be used to illustrate vertical profiles of ground-water concentrations on a site. Both cross-sections and fence diagrams can provide useful visual interpretations of contaminant concentrations and migration.

6.4 CONTOUR MAPPING

Contour maps are useful for depicting ground-water contaminant concentration values throughout a site. Contour mapping requires an accurate, to-scale basemap of the site. After data posting sample values on the basemap, insert contour lines (or isopleths) at a specified contour interval, interpolating values between sample points. Contour lines can be drawn manually or can be generated by computer using contouring software. Although the software makes the contouring process easier, computer programs have a limitation: as they interpolate between data points, they attempt to "smooth" the values by fitting contour intervals to the full range of data values. This can result in a contour map that does not accurately represent general site contaminant trends. If there is a big difference in concentration between a "hot spot" and the surrounding area, the computer contouring program, using a contour interval that attempts to smooth the "hot spots," may eliminate most of the subtle site features and general trends.

6.5 WELL LOCATION MAP

A well location map should be prepared using surveyed data for all features at the site. This map serves as a basemap onto which other data may be plotted (e.g., data posting, contaminant plume contours, water elevation contours). The map is drawn to scale and incorporates all wells located, installed, and sampled, including residential and monitoring wells. The surveyed coordinates for each monitoring well location could also be posted onto the map (in feet above mean sea level (msl)) to illustrate topography and surface gradient.

6.6 STATISTICAL GRAPHICS

The distribution or spread of the data set is important in determining which statistical techniques to use. Common statistical analyses, such as the t-test, rely on normally distributed data. The histogram is a statistical bar graph which displays the distribution of a data set. A normally distributed data set takes the shape of a bell curve, with the mean and median close together about halfway between the maximum and minimum values. A probability plot depicts cumulative percent against the concentration of the contaminant of concern. A normally distributed data set, when plotted as a probability plot, appears as a straight line. A histogram or probability plot can be used to see trends and anomalies in the data from a ground-water site prior to conducting more rigorous forms of statistical analysis.

6.7 RECOMMENDED DATA INTERPRETATION METHODS

The data interpretation methods chosen depend on project-specific considerations, such as the number of sampling locations and their associated range in values. Data which are dissimilar in composition should not be compared using statistical interpretation methods. Data posting, screening, and sampling data sheets, and cross-section/fence diagrams may be appropriate. A site feature showing extremely low data values (e.g., non-detects), with significantly higher values (e.g., 5,000 ppm) from neighboring "hot spots" and little or no concentration gradient in between, does not lend itself to contouring software.

6.8 EXAMPLE SITE

A water table contour map was generated with the water level data for the shallow overburden monitoring wells. This indicated a westward flow direction, which generally coincides with the surface topography. The deep bedrock wells lie nearly on a straight line, and therefore a confident determination of flow direction was not possible. A westward component of flow direction is evident in the data, however. The bedrock contact wells provided inconsistent water level data, most likely due to the presence of discontinuous perched water zones at the well locations.

All ground-water samples were analyzed for total chromium and cyanide. Cyanide was not found in any of the samples above the 50 µg/l detection limit.

Using a detection limit of 50 µg/l for chromium, three filtered samples were found to be contaminated at two locations (3OB, and 6OB/6AW). Five of the unfiltered ground-water samples (Wells 2SA, 3OB, 4SA, 6OB, and 6AW) exceeded the detection limit. These data were posted on a site/well location map to illustrate well proximities, as well as a map indicating the contours of contamination.

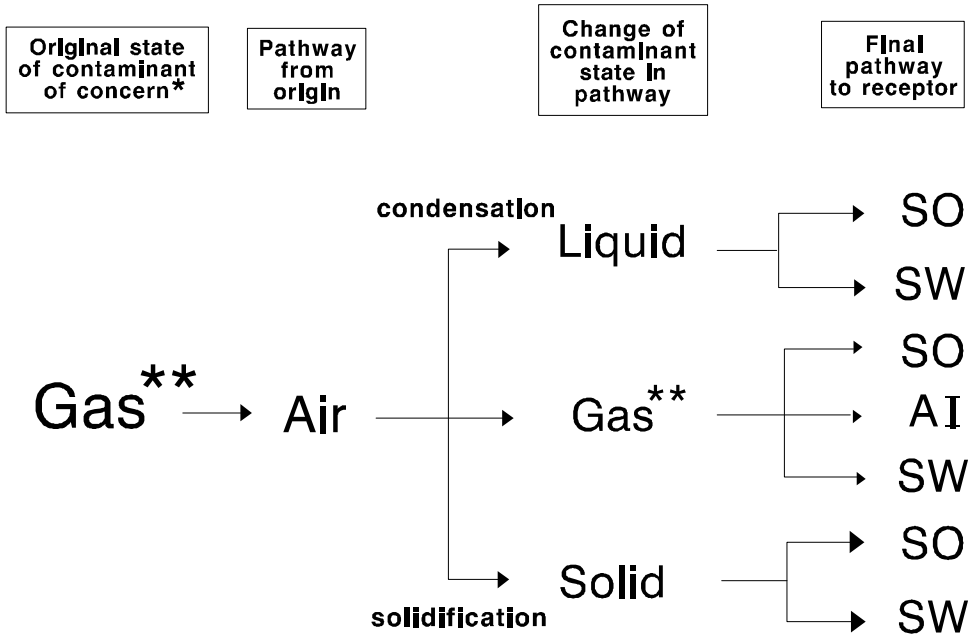
The rate of chromium contaminant migration in ground water and the potential long-term impact to nearby residential wells were estimated using a mathematical model which included worst case assumptions and evaluated attenuation of contaminants through soil and ground water. The OSC concluded that the potential for residential well contamination was minimal. Removal of soil, the source of contamination, was recommended. This decision met the Phase 2 objective of establishing early action options and consideration of long-term remediation requirements for ground water.

All containers of wastes were removed from the site. Soil treatment/disposal was completed using the existing grid design. Cells were sampled and designated as clean or excavated. Excavated material was stockpiled while treatment/disposal options were evaluated. Excavated cells were filled with stone and clean soil. Composite sampling in each cell verified cleanup, using an action level of 100 mg/kg chromium in the soil composite. (The clean-up level was established based on the earlier mathematical model and soil attenuation calculations.) The soil response served as an early action to meet the Phase 3 objective originally established for the site.

APPENDIX A -- Example of Flow Diagram For Conceptual Site Model

Figure A-1

Migration Routes of a Gas Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
G,D	G,D	N/A
G,D	G,D	G,D
I,D	I,D	N/A
I,D	I,D	N/A
G,D	I,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D

* May be a transformation product
** Includes vapors

Receptor Key

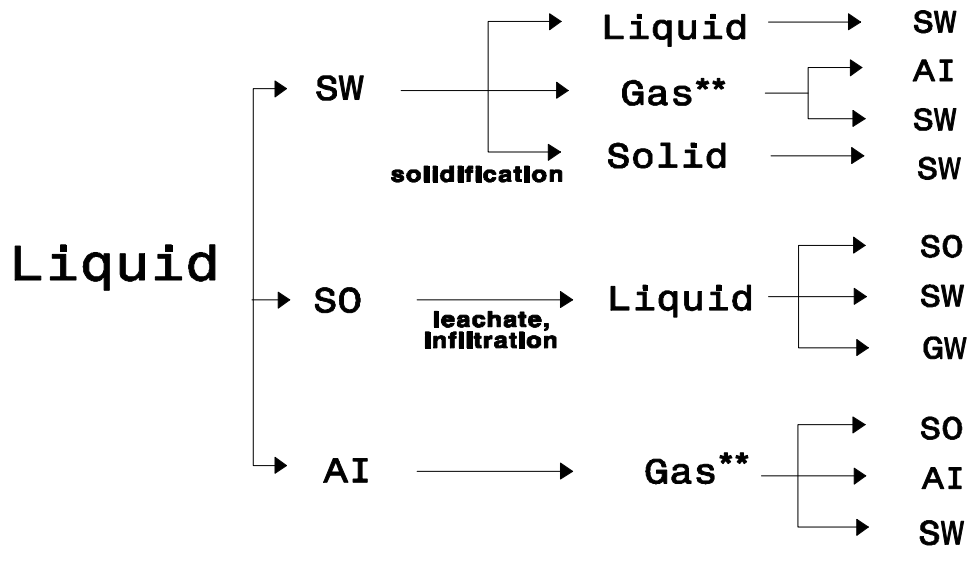
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key

AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

Figure A-2

Migration Routes of a Liquid Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
G,D	G,D	G,D
I,D	I,D	N/A
G,D	G,D	G,A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D
G,D	N/A	N/A
G,D	G,D	N/A
I,D	I,D	N/A
G,D	G,D	G,D

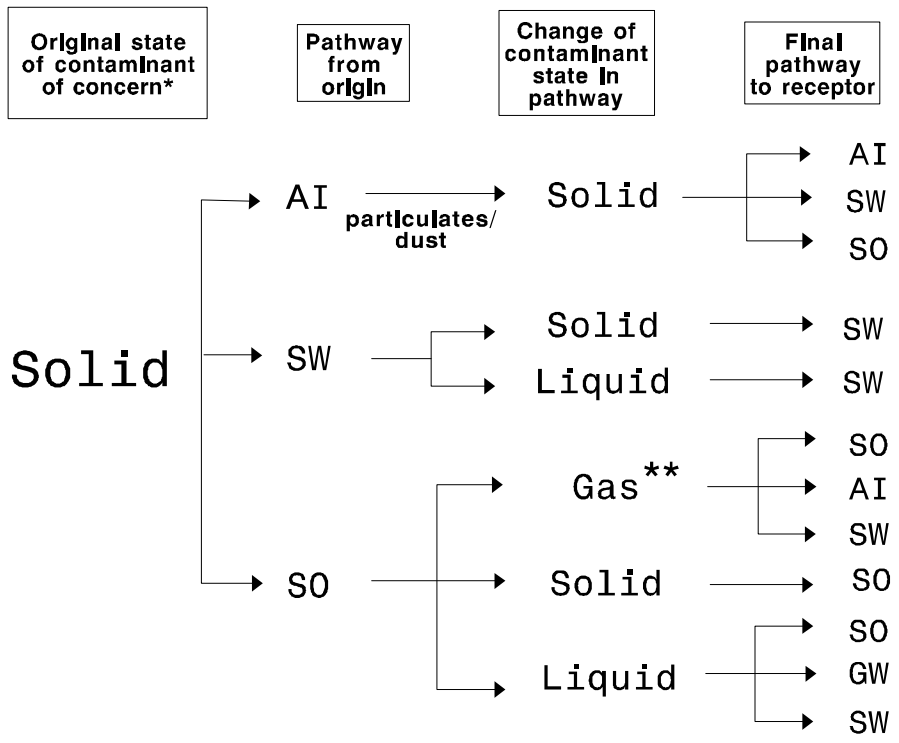
* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

Figure A-3

Migration Routes of a Solid Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D
G,D	G,D	G,D
G,D	G,D	N/A
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	N/A
G,D	N/A	N/A
G,D	G,D	G,D

* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
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Pathway Key
AI = Air
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Attachment D

MiniRAE 2000 Instrument Manual

MiniRAE 2000

Portable VOC Monitor
PGM-7600



OPERATION AND MAINTENANCE MANUAL

(Document No.: 011-4001-000)
Revision E, May 2005

RAE SYSTEMS

ISO 9001 CERTIFIED

ATTENTION!

For European Applications

A. CE 0575 ⟨Ex⟩ II 1G/2G
DEMKO 03 ATEX 0204759X
Eex ia IIC T4

B. Recharge batteries only in non-hazardous locations.

C. Do not connect external cable to serial interface jack in hazardous locations.

D. Use RAE Systems Adapter P/N 500-0072 for connection to communication port and charging jack only in a non-hazardous area.

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⚠ WARNING ⚠

- Do NOT proceed before reading -

This manual must be carefully read by all individuals who have or will have the responsibility for using, maintaining, or servicing this product.

The product will perform as designed only if it is used, maintained, and serviced in accordance with the manufacturer's instructions.

CAUTION!!

To reduce the risk of electric shock, turn off power before removing the monitor cover. Disconnect the battery before removing sensor module for service. Never operate the monitor while the cover is removed. Remove monitor cover and sensor module only in an area known to be non-hazardous.

The model PGM-7600 equipment is classified as to intrinsic safety for use in class I, division 1, groups A, B, C, D, or non-hazardous locations only.

Special Notes

-1-

When the MiniRAE 2000 Monitor is taken out from the transport case and turned on for the first time, there may be some residual organic or inorganic vapor trapped inside the detector chamber. The initial PID sensor reading may indicate a few ppm. Enter an area known to be free of any organic vapor and turn on the monitor. After running for several minutes, the residual vapor in the detector chamber will be cleared and the reading should return to zero.

-2-

The battery of the MiniRAE 2000 monitor will discharge slowly even if it is turned off. If the monitor has not been charged for 5-7 days, the battery voltage will be low. Therefore, it is a good practice to always charge the monitor before using it. It is also recommended to fully charge the monitor FOR AT LEAST 10 HOURS before first use. See Section 7 for more information on battery charging and replacement.

WARNINGS

STATIC HAZARD: Clean only with damp cloth.

For safety reasons this equipment must be operated and serviced by qualified personnel only. Read and understand instruction manual completely before operating or servicing.

Use only RAE Systems battery packs, part nos. 012-3050, 012-3051 or 012-3052. This instrument has not been tested in an explosive gas/air atmosphere having an oxygen concentration greater than 21%. Substitution of components may impair intrinsic safety. Recharge batteries only in non-hazardous locations.

The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service.

For maximum safety, the accuracy of the MiniRAE 2000 should be checked by exposing it to a known concentration calibration gas before each day's use.

AVERTISSEMENTS

DANGER RISQUE D'ORIGINE ELECTROSTATIQUE: Nettoyer uniquement avec un chiffon humide.

Pour des raisons de sécurité, cet équipment doit être utilisé, entretenu et réparé uniquement par un personnel qualifié. Étudier le manuel d'instructions en entier avant d'utiliser, d'entretenir ou de réparer l'équipement.

Utiliser seulement l'ensemble de batterie RAE Systems, la reference 012-3050, 012-3051 au 012-3052. Cet instrument n'a pas été essayé dans une atmosphère de gaz/air explosive ayant une concentration d'oxygène plus élevée que 21%. La substitution de composants peut compromettre la sécurité intrinsique. Ne charger les batteries que dans emplacements désignés non-dangereuse.

La calibration de toute instruments de RAE Systems doivent être testé en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant de mettre en service l'instrument pour la première fois.

Pour une securite maximale, la sensibilité du MiniRAE 2000 doit être verifier en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant chaque utilisation journalière.

1. GENERAL INFORMATION

MiniRAE 2000 Portable VOC Monitor (Model PGM 7600) is a compact monitor designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It monitors Volatile Organic Compounds (VOC) using a Photo-Ionization Detector (PID) with a 9.8 eV, 10.6 eV, or 11.7 eV gas discharge lamp. Features are:

- **Lightweight and Compact**
  -Compact, light weight (19 oz.) and rugged design
  -Built-in sample draw pump

- **Dependable and Accurate**
  - Up to 10 hours of continuous monitoring with rechargeable battery pack
  - Designed to continuously monitor VOC vapor at ppm levels

- **User Friendly**
  -Preset alarm thresholds for STEL, TWA, low and high level peak values. Audio buzzer and flashing LED display are activated when the limits are exceeded.

- **Datalogging Capabilities**
  -15,000 point datalogging storage capacity for data download to PC

MiniRAE 2000 consists of a PID with associated microcomputer and electronic circuit. The unit is housed in a rugged ABS + PC case with a backlit 1 line by 8 character dot matrix LCD and 3 keys to provide easy user interface.

1.1 General Specifications

Table 1.1

Portable VOC Monitor Specification	
Size:	8.2"L x 3.0"W x 2.0"H
Weight:	19.5 oz with battery pack
Detector:	Photo-ionization sensor with 9.8, 10.6, or 11.7 eV UV lamp
Battery:	A 4.8V /1250 mAH Rechargeable Nickel Metal Hydride battery pack (snap in, field replaceable)
Battery Charging:	10 hours charge through built-in charger
Operating Hours:	Up to10 hours continuous operation
Display:	1 line by 8 characters 5x7 dot matrix LCD (0.4" character height) with LED back light automatically in dim light
Range, Resolution & Response time (t_{90}):	
Isobutylene (calibration gas)	
	0-99 ppm 0.1 ppm 2 sec
	100-1,999 ppm 1.0 ppm 2 sec
	2000-10,000 ppm 1.0 ppm 2 sec
Measurement Accuracy (Isobutylene):	
	0 – 2000 ppm: ± 2 ppm or 10% of reading.
	> 2000 ppm: ± 20% of reading
PID Detector:	Easy access to lamp and sensor for cleaning and replacement
Correction Factors:	Built-in 102 VOC gases
Calibration:	Two-point field calibration of zero and standard reference gas
Calibration Memory:	
	Store up to 8 separate calibration, alarm limits and span value
Inlet Probe:	Flexible 5" tubing
Keypad:	1 operation key and 2 programming keys

Direct Readout:	Instantaneous, average, STEL and peak value, battery voltage and elapsed time
Intrinsic Safety:	UL & cUL Class 1, Division I, Group A,B,C,D, Temperature Code T3C (US & Canada); CE 0575 ⟨Ex⟩ II 1G DEMKO 02 ATEX 0204759 Eex ia IIC T4 (Europe)
EM Interference:	No effect when exposed to 0.43 W/cm^2 RF interference (5 watt transmitter at 12 inches)
Alarm Setting:	Separate alarm limit settings for Low, High, STEL and TWA alarm
Operating Mode:	Survey or Hygiene mode
Alarm: 90 dB buzzer and flashing red LEDs to indicate exceeded preset limits, low battery voltage, or sensor failure.	
External Alarm:	Optional plug-in pen-size vibration alarm or remote alarm
Alarm Mode:	Latching or automatic reset
Real-time Clock:	Automatic date and time stamps on data logged information
Datalogging:	15,000 points with time stamp, serial number, user ID, site ID, etc.
Communication:	Upload data to PC and download instrument setup from PC through RS-232 port
Sampling Pump:	Internally integrated. Flow rate: 450-550 cc/min.
Temperature:	0° to 45°C (32° to 113°F)
Humidity:	0 % to 95 % relative humidity (non-condensing)
Housing:	ABS + PC, conductive coating, splash and dust proof, will withstand 1 meter drop test with rubber boot
Attachment:	Wrist strap, rubber boot and belt clip

2. OPERATION OF MINIRAE 2000

The MiniRAE 2000 Portable VOC Monitor is a compact Monitor designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment the MiniRAE 2000 is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. However, the user should test the instrument and verify the calibration before the first use. After the monitor is fully charged and calibrated, it is ready for immediate operation.

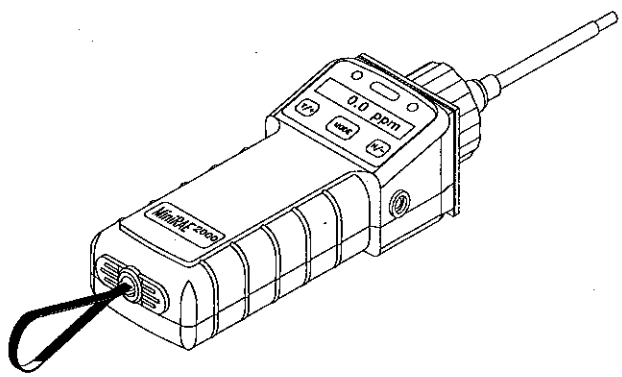


Figure 2-1 MiniRAE 2000

2.1 Physical Description

The main components of the MiniRAE 2000 Portable VOC monitor include:

- Three keys for user to interact with the monitor: 1 operation key and 2 programming keys for normal operation or programming of the monitor
- LCD display with back light for direct readout and calculated measurements
- Buzzer and red LED's for alarm signaling whenever the exposures exceed preset limits
- Wrist strap
- Charge contact for plugging directly to the charging station
- Gas entry and exit ports
- Serial communication port for PC interface
- External alarm and analog output port
- Protective rubber cover

2.2 Keys and Display

Figure 2.2 shows the LCD display and the keypad on the front panel of the monitor. The function of the 3 keys during normal operation are summarized below:

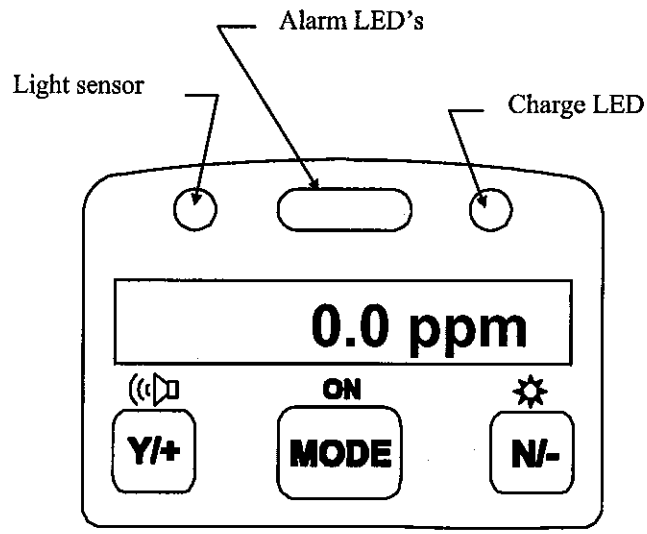


Figure 2-2 LCD Display and Keypad

Key	Function in Normal Operation
[MODE]	-Turn on/off the power* and step through menu items
[N/-]	-Toggle on/off the back light, negative acknowledge, decrease value
[Y/+]	-Start measurement, positive acknowledge, increase value value

* Pressing and holding **[MODE]** key for 5 seconds turns off the power to the monitor. Monitor will beep once per second and display countdown timer during power-down sequence. Press **[MODE]** key momentarily to step through menu items. To save time, press any key during message scrolling to skip to the end of the message.

2.3 Power On/Off

To turn on the MiniRAE 2000 portable VOC monitor, press **[MODE]** key for one second and release. The audio buzzer will beep once and the air pump will turn on. The display will show "ON!.." and then "Ver n.nn" to indicate the unit's current firmware version number. Next displayed are the serial number, the model number, Operating mode, current date and time, unit internal temperature, gas selected, high low, STEL, TWA/AVG alarm limits, battery voltage, and shut off voltage. Also displayed are internal mode settings such as User mode, Alarm mode, datalog time remaining and log periods in the respective order.

To turn off the MiniRAE 2000 portable VOC monitor, press and hold the **[MODE]** key for 5 seconds. The monitor will beep once per second during the power-down sequence with a count down timer showing the number of remaining seconds. The message "Off!.." flashes on the LCD display and the display will go blank indicating that the monitor is turned off.

Data protection during power off

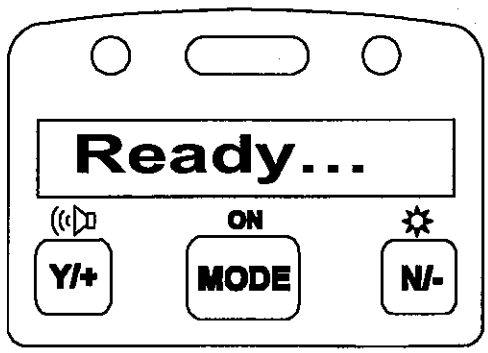
When the monitor is turned off, all the current real time data including last measured value are erased. However, the datalog data is preserved in non-volatile memory. Even if the battery is disconnected, the datalog data will not be lost. While the power is off, the real time clock will continue to operate until the battery is completely drained (usually in 4-5 days without any charging). If the battery is completely drained or is disconnected from the monitor for more than 30 minutes, the real time clock will be lost. In this case, the user needs to enter the real time clock information again, as described in Section 4, or send the PC clock during configuration through the PC communication.

2.4 Operation

The **MiniRAE 2000** VOC monitor has two operation modes: **Survey** and **Hygiene** mode. The **Survey mode** allows the user to manually start and stop the monitoring/measuring operation and display certain exposure values. In the **Hygiene mode,** the monitor runs continuously after the monitor is turned on. Refer to Section 4.7.1 for switching between the two modes.

2.4.1 Survey Mode

After the monitor is turned on, it runs through the start up menu. Then the message "**Ready...**" is displayed (see figure below).



At this point, the user has two options:

1. Step through the Main Menu.
2. Take a measurement.

Press the **[MODE]** button to step through the Main Menu. Press the **[Y/+]** button to proceed to take a measurement.

The Main Menu

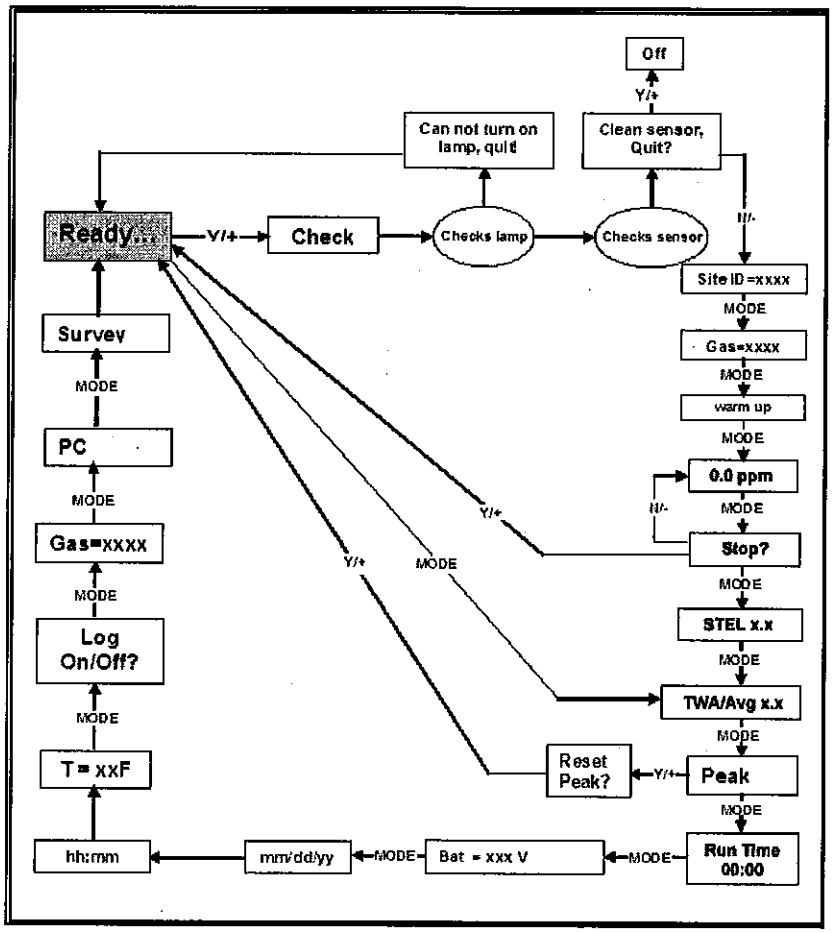
Press the **[MODE]** button to enter the Main Menu. Press the **[Y/+], [MODE]** or **[N/-]** as indicated in the flow chart below to step through the Main Menu. The PID sensor and pump are turned off during this time.

The Main Menu functions are:

- **Ready...**
- **Check**
- **Site ID = xxxx**
- **Gas = xxxx**
- **Warm up**
- **X.x ppm**
- **Stop?**
- **TWA/Avg x.x ppm**
- **STEL x.x ppm**
- **Peak x.x ppm**
- **Run time hh:mm**
- **Bat = X.XV**
- **Mm/dd/yy**
- **hh:mm**
- **T = xxxF [date, time and temperature (°C or °F)]**
- **Log On/Off?**
- **PC Comm?**
- **Survey**

These functions are arranged in a "round robin" order. To select a specific function, press the button as shown below until the desired function appears.

Main Menu



The Main Menu Functions

- **Ready...:** Indicates that the monitor is ready to take a measurement or to step through the Main Menu. Press the **[Y/+]** button to advance to taking a measurement (read "Taking a Measurement" on Page 2-12 for details).

  Note: The **Ready...** screen is skipped if the menu is cycled through while a measurement is running.

- **Check...:** This message displays while the system is checking the lamp and the sensor. If the lamp test succeeds, the system will progress to checking the sensor. If the lamp test does not succeed, the display will read **Can not turn on lamp, quit!**

  - **Can not turn on lamp, quit!:** This message displays when the lamp does not turn on. The system will automatically return to **Ready...** allowing the user to test the lamp again. If the lamp fails a second time, turn the monitor off and refer to Section 7.2 "**PID Sensor & Lamp Cleaning / Replacement**".

  - **Clean Sensor, Quit?:** This message displays when the sensor requires cleaning. Press the **[Y/+]** button to turn the monitor off and clean the sensor. Press the **[N/-]** button and the system will progress to the **Site ID = xx**

- **Site ID = xxxx:** This display shows the Site ID and indicates that the monitor is about to start taking measurements (read "Taking a Measurement" for details)

  Note: This display appears only after a measurement has been started. It does not appear when the user is cyling through the Main Menu and the monitor is idling.

- **Gas = xxxx:** This display identifies the gas to be measured and indicates that the monitor is about to take a measurement (read "Taking a Measurement" for details)

Note: This display appears only after a measurement has been started. It does not appear when the user is cyling through the Main Menu and the monitor is idling.

- **x.x ppm:** (read "Taking a Measurement" for details)

  Note: This display appears only after a measurement has been started. It does not appear when the user is cyling through the Main Menu and the monitor is idling.

- **TWA/Avg:** Displays (in ppm) the Time Weighted Average (TWA) or the Average since the start of the measurement. The average is recalculated every minute.

- **STEL:** Displays the Short Term Exposure Limit.

- **PEAK:** Displays (in ppm) the highest instantaneous reading since the start of the measurement. If **[Y/+]** is pressed while the peak reading is displayed, the unit will ask **Reset Peak?**. If **[Y/+]** is pressed again, the peak value will be cleared and the display will return to the **Ready...** message or instantaneous reading. The peak reading is automatically reset when a new measurement is started by pressing **[Y/+]** from the **Ready...** screen.

- **Run time hh:mm:** The duration of the current measurement period.

- **Bat = X.XV:** The current battery voltage.

  Note: A fully charged battery pack should show 4.8 volts or higher. When the battery voltage falls below 4.4 volts, a flashing **"Bat"** will appear as a warning message. At that point, you have 20-30 minutes of run time remaining. When the battery voltage falls below 4.2 volts the monitor turns off automatically.

- **Mm/dd/yy:** The current date.

- **hh:mm:** The current time (24-hour format)

- **T = xxxF:** The internal unit temperature in degrees Fahrenheit. (see Section 4.7.13 to change temperature units)
- **Log on/Off?:** Allows the user to start datalogging of the current measurement. A superscript "L" flashes in the ppm measurement display when datalogging is on. This screen is not shown when datalogging is disabled or when the monitor is not operating in manual start/stop mode.
- **PC Comm?:** This function enables the user to upload data from the MiniRAE 2000 to a Personal Computer (PC) or send/receive configuration information between a PC and the MiniRAE 2000. Press **[MODE]** to return to **Ready...** .

  To communicate with a PC, connect the monitor to the serial port of a PC and start the MiniRAE 2000 application software. Press the **[Y/+]** button and the LCD displays **"pause monitor, ok?"** Press the **[Y/+]** button one more time, and the display shows **"Comm..."** The monitor is now ready to receive commands from the PC.
- **Survey:** This function displays the Current Operating Mode (**Survey** or **Hygiene**).

Taking a Measurement

There are two ways to start a measurement.

1. Operating in Hygiene mode.
2. Manually start and stop measurement in Survey mode.

To start a measurement in Hygiene mode, please refer to Section 4.7.1, "**Change Operation Mode**". To start a measurement in Survey Mode, the MiniRAE 2000 monitor must first be in the "Ready..." mode. This is the mode to which the monitor normally powers up.

Measurement phases

1. Ready
2. Start measurement
3. Measurement display and datalogging
4. Stop measurement

Ready

The display reads **Ready...** indicating the unit is ready to start a measurement.

Start Measurement

Press the **[Y/+]** button to start the check cycle (see above), and then the measurement cycle.

After completing the **Check** cycle, the display will show the **Site ID** and then the **Gas** selected for measurement. The pump will start and the reading will be displayed. The **Peak** and **Average** values will be automatically reset to zero.

Measurement Display and Datalog

Instantaneous readings of the gas concentration in parts per million (ppm) are updated every second. A flashing superscript **L** is displayed when datalogging is on. Datalog information is saved only after one full datalog period is completed.

Stop Measurement

Press the **[MODE]** button and the display shows Stop? Press **[N/-]** to continue measurement and **[Y/+]** to stop the measurement and datalog event. The pump stops automatically when measurement is stopped. Peak and average values for the current measurement can be read in idle mode until a new measurement is started.

Automatic Increment of Site ID

Every time a measurement is taken, the site ID will be incremented by one automatically in Survey mode.

Variable Alarm Signal

In Survey Mode, if the measurement exceeds the low alarm limit, the buzzer and flashing alarm are activated and will beep/flash once per second. The alarms will increase in frequency as the gas concentration approaches the high alarm limit reaching 8 times per second when the high alarm has been exceeded.

Press **[Y/+]** key to clear if latching alarm.

2.4.2 Hygiene Mode

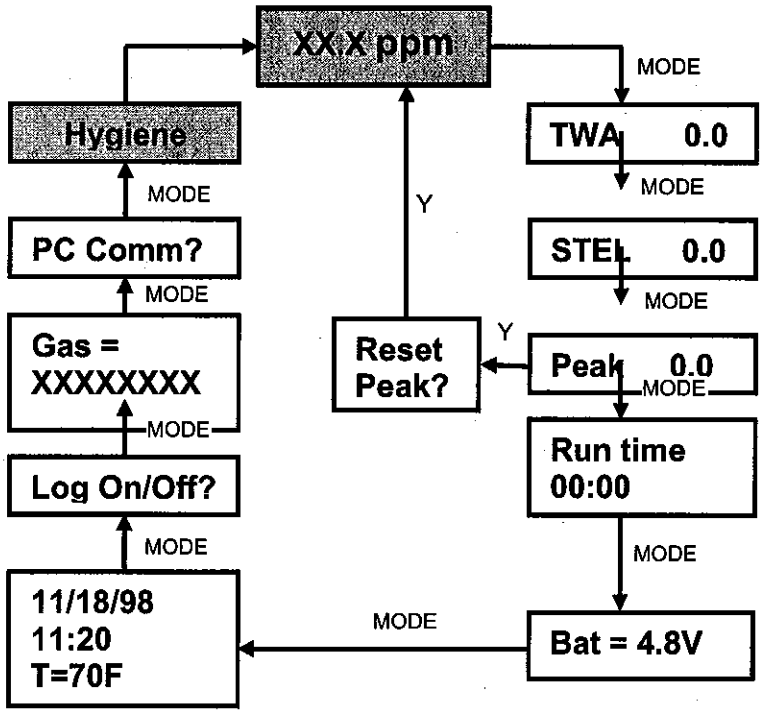
In Hygiene Mode, the unit will continuously taking measurements, once the power is turned on. After the initial start-up sequence displaying the current monitor settings, the LCD displays the instantaneous readings.

The Hygiene operation menu displays include:

- **Real time readings in ppm**
- **Current TWA/Avg, STEL and Peak values (see Section 4.6.6)**
- **Run time**
- **Current battery voltage**
- **Date, time and temperature**
- **Log on/off?**
- **Gas name**
- **PC communication?**
- **Hygiene**

Detailed description of most of these displays are the same as Section 2.4.1.

HYGIENE MODE MAIN MENU



To choose a specific display, press the **[MODE]** key one or more times until the desired display appears, or the **[Y/+]** key where indicated with a Y.

Note: To get back to instantaneous reading from any of the above display, press **[MODE]** key repeatedly until the "XX.X ppm" display appears.

2.5 Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low, High, TWA and STEL). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn the user of the alarm condition.

In addition, the MiniRAE 2000 will alarm if one of the following conditions occurs: battery voltage falls below a pre-set voltage level (4.4 V), failure of UV lamp, pump stall, or when the datalog memory is full. When the low battery alarm occurs, there will be approximately 20-30 minutes of operating time remaining. When the battery voltage falls below 4.2 V, the monitor will turn off automatically.

Alarm Signal Summary:

Condition	Alarm Signal
Gas exceeds "High Alarm" limit	3 beeps/flashes per second
Gas exceeds "Low Alarm" limit	2 beeps/flashes per second
Gas exceeds "TWA" limit	1 Beeps/flashes per seconds
Gas exceeds "STEL" limit	1 Beeps/flashes per seconds
Pump failure	3 beeps/flashes per second plus "Pump" message on LCD
PID lamp failure	3 beeps/flashes per second plus "Lamp" message on LCD
Low battery	1 flash per second, 1 beep per minute plus "Bat" message on LCD
Memory full	1 flash per second plus "Mem" message on LCD

Alarm Signal Testing:

Under normal non-alarm conditions, it is possible to test the MiniRAE 2000 LED and buzzer in Special Diagnostic Mode (see Section 8 for details).

2.6 Preset Alarm Limits and Calibration

The MiniRAE 2000 portable VOC monitor is factory calibrated with standard calibration gas, and is programmed with default alarm limits. There are 102 gas settings stored in the library. Some examples of calibration and alarm limits are shown below. Refer to Section 4 on programming procedures for selecting a different gas, perform a calibration or set new alarm limits.

Factory Calibration and Preset Alarm Limits

Cal Gas	Cal Span	unit	Low	High	TWA	STEL
Isobutylene	100	ppm	50	100	100	250
Hexane, n-	100	ppm	500	750	500	750
Xylene, m-	100	ppm	100	150	100	150
Benzene	5	ppm	2	5	5	2
Styrene	50	ppm	20	40	20	40
Toluene	100	ppm	50	100	50	100
Vinyl Chloride	10	ppm	5	10	5	10
Custom	100	ppm	50	100	50	100

2.7 Integrated Sampling Pump

The MiniRAE 2000 portable VOC monitor includes an integrated sampling pump. This is a diaphragm type pump that provides a 500-600 cc per minute flow rate. Connecting a Teflon or metal tubing with 1/8 inch inside diameter to the gas inlet port of the MiniRAE 2000, this pump can pull in air samples from 200 feet away horizontally, or 90 feet vertically, at about 3 feet per second flow speed.

The pump turns on when a measurement is started, and turns off when the sample is manually stopped in Survey mode or when the unit is turned off from Hygiene Mode.

If liquid or other objects are pulled into the inlet port filter, the monitor will detect the obstruction and shut down the pump immediately. The alarm will be activated and a flashing error message "Pump" will be also displayed on the LCD display.

The user should acknowledge the pump shut off condition by clearing the obstruction and pressing the **[Y/+]** key to re-start the pump.

The pump stall threshold is set in the special Diagnostic Mode (Section 8).

2.8 Back Light

The LCD display is equipped with an LED back light to assist in reading the display under poor lighting conditions. Pressing and holding the **[N/-]** key for one second in normal operation can turn on the backlight. The backlight can be turned off by pressing **[N/-]** a second time. If the **[N/-]** key is not pressed, the back light will be turned off automatically after a pre-programmed time-out period to save power.

In addition, the ambient light is sensed and the back light will be turned on automatically if the ambient light is below a threshold level. The back light is turned off automatically when the ambient light exceeds the threshold level.

See Section 8 for instructions on how to set the light threshold level.

Note: The LED backlight consumes about 20-30% of the total average current, when the instrument is idle or not taking a measurement.

2.9 Datalogging

During datalogging, the MiniRAE 2000 Portable VOC monitor flashes a superscript "L", on the display to indicate that datalogging is enabled. The monitor stores the time stamp, sample number, and measured gas concentration at the end of every sample period (when data logging is enabled). In addition, the following information are stored: user ID, site ID, serial number, last calibration date, and alarm limits. All data are retained (even after the unit is turned off) in non-volatile memory so that it can be down loaded at a later time to a PC.

Datalogging event

When Datalogging is enabled, measurement readings are being saved. These data are stored in "groups" or "events. A new event is created and stored each time the monitor is turned on, or a configuration parameter is changed, or datalogging is interrupted (e.g. Communication with PC during Hygiene mode). Information, such as start time, user ID, site ID, gas name, serial number, last calibration date, and alarm limits will be recorded.

Datalogging sample

After an event is recorded, the unit records a shorter form of the data. This data contains: the sample number, time (hour/minute) and gas concentration.

3. OPERATION OF ACCESSORIES

The accessories for the MiniRAE 2000 include:

- An AC Adapter (Battery Charger)
- Alkaline battery holder
- Water Trap Filter

Optional Accessories:

- Dilution Fitting
- Calibration adapter
- Calibration regulator and Flow controller
- Organic Vapor Zeroing kit

3.1 Standard Kit and Accessories

1) AC Adapter (Battery Charger)

WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in area known to be non-hazardous.

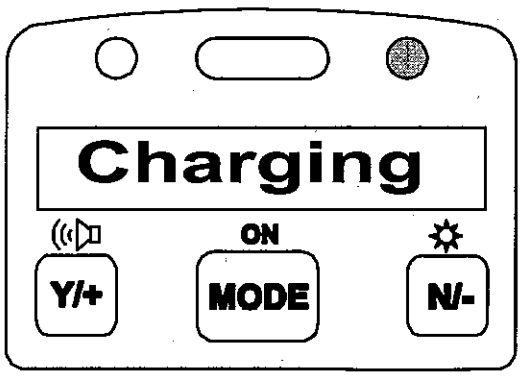
Ne charger les batteries que dans emplacements designés non-dangereuses.

A battery charging circuit is built into the MiniRAE 2000 monitor. It only needs a regular AC to 12 V DC adapter (wall mount transformer) to charge the monitor.

To charge the battery inside the MiniRAE 2000 monitor:

1. Power off the Monitor.
2. Connect the AC adapter (or the optional automotive charging adapter) to the DC jack on the MiniRAE 2000 monitor. If the unit was off, it will automatically turn on.
3. The first message displayed will be "Deep discharge?" The unit will ask this question for three times. If the user wants to discharge the battery pack, affirm this query with the **[Y/+]** key, otherwise the unit will move on to the charge mode directly.

4. While charging, the display message will alternate between "Charging" and "Bat=x.xV" (x.x is the present battery voltage). The LED should be red in color when charging.



5. When the battery is fully charged, the LED will change from red to green and the message "Fully charged" will appear on the display. After the battery is fully charged, the unit will enter the "trickle charge" mode. In which, the red LED will turn on for several seconds every minute, to maintain the full charge.

A completely discharged MiniRAE 2000 monitor will be charged to full capacity within 10 hours. The battery will be drained slowly even if the monitor is turned off. If the monitor has not been charged for 7-10 days, the battery voltage will be low.

The factory-supplied battery is designed to last for 10 hours of normal operation (no alarm, no back light condition), for a new battery under the best condition. As the battery becomes older or is subject to adverse conditions (such as cold ambient temperature), the battery capacity will be reduced significantly.

2) Alkaline Battery Holder

An alkaline battery holder is supplied with each MiniRAE 2000. It accepts four AA size alkaline batteries and can be used in place of the Ni-MH or Ni-Cd battery pack to provide approximately 12-14 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Ni-Cd or Ni-MH battery pack.

To install the adapter, remove the cover of the battery compartment. Remove the Ni-Cd or Ni-MH battery pack from the battery compartment and replace with the alkaline battery adapter. Replace the battery compartment cover.

The internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the monitor.

***Note*:** The AA Alkaline battery adapter supplied by RAE Systems Inc. is intrinsically safe!

3) Water Trap Filter

The water trap filter is made of PTFE (Teflon®) membrane with a 0.45 micron pore size to prevent water from being sucked into the sensor manifold, which would cause extensive damage to the monitor. It will also remove any dust and other particles from entering the monitor and prolong the operating life of the sensor. To install the water trap, simply insert it to the front of the inlet tube of the MiniRAE 2000 monitor.

3.2 Optional Accessories

1) Dilution Fitting

The user may wish to install a dilution fitting on the inlet to dilute the gas samples. One application for a dilution fitting is to measure organic gas when the concentration exceeds the upper limit of the sensor range.

Make sure to set the dilution ratio in the programming mode (see Section 4.7.9) so that the correct gas reading will be displayed when the dilution fitting is used.

WARNING: To use a dilution fitting, the user must have the monitor located in a clean atmosphere outside the confined space and use a remote access probe or Tygon tubing to measure the gas concentration inside the confined space.

2) Calibration Adapter

The calibration adapter for the MiniRAE 2000 is a simple 6-inch Tygon tubing with a metal adapter on one end. During calibration, simply insert the metal adapter into the regular gas inlet probe of the MiniRAE 2000 and the tubing to the gas regulator on the gas bottle.

3) Calibration Regulator and Flow Controller

The Calibration Regulator and Flow controller is used in the calibration process. It regulates the gas flow rate from the Span gas cylinder into the gas inlet of the MiniRAE 2000 monitor during calibration process. The maximum flow rate allowed by the flow controller is about 0.5L/min (500 cc per min.). Alternatively, a Demand-flow Regulator or a Tedlar gas bag may be used to match the pump flow precisely.

4) Organic Vapor Zeroing kit (Charcoal filter)

The Organic Vapor Zeroing Kit is used for filtering organic air contaminants that may affect the zero calibration reading. To use the Organic Vapor Zeroing Kit, simply connect the filter to the inlet port of the MiniRAE 2000.

4. PROGRAMMING OF MINIRAE 2000

The MiniRAE 2000 Monitor is built with a microcomputer to provide programming flexibility. Authorized users can re-calibrate the monitor, change the alarm limits, change site ID, user ID, lamp type, and real time clock, etc.

Programming is menu-driven to provide intuitive end-user operation. The display shows the menu options and the key pad used for menu selection and data entry.

4.1 Programming Mode

The programming mode allows the users to change the setups in the monitor, calibrate the monitor, modify the sensor configuration and enter user information, etc. The programming mode has four menu items. Each menu item includes several sub-menus to perform additional programming functions. Appendix A shows a more detailed menu tree structure.

Programming Menu

Calibrate/Select Gas?

Change Alarm Limits?

Change Datalog?

Change Monitor Setup?

Once inside the programming mode, the LCD will display the first menu. Each subsequent menu item can be viewed by pressing the **[N/-]** repeatedly until the desired menu is displayed. To enter the sub-menu of a particular menu, press **[Y/+]** key, the sub-menu will be displayed.

Return to Operation mode: To exit the programming mode and return to operation, press the **[MODE]** key once at any of the programming menu displays.

4.2 Keys for Programming Mode

The three keys perform a different set of functions during the programming mode as summarized below.

Key	Function in Programming Mode
[MODE]:	Exit menu when pressed momentarily or exit data entry mode when pressed and held for 1 second
[Y/+]:	Increase alphanumerical value for data entry or confirm (yes) for a question
[N/-]:	Decrease alphanumerical value for data entry or deny (no) for a question

4.3 Entering into Programming Mode

1. Turn on the MiniRAE 2000 monitor and wait for the **"Ready.."** message or the instantaneous reading display **"0.0 ppm"** message displayed.
2. Press and hold down both **[N/-]** and **[MODE]** keys for three seconds to enter programming mode. This delay is to prevent the user from entering programming mode by accident.
3. The first menu item "Calibrate/select Gas?" will be displayed.
4. Release both **[MODE]** and **[N/-]** keys simultaneously to start the programming mode
5. Press **[N/-]** key to scroll to the next menu item of the programming menu. Press **[Y/+]** key to select the displayed menu item.

The following Sections 4.4 - 4.7 describe the details of each menu options.

4.4 Calibrate and Select Gas

WARNINGS

The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service for the first time.

For maximum safety, the accuracy of the MiniRAE 2000 should be checked by exposing it to known concentration calibration gas before each day's use.

In the first menu of the programming mode, the user can perform functions such as calibration of the MiniRAE 2000 Monitor, select default cal memories, and modify cal memories (see Table 4.4).

Table 4.4

Calibrate/Select Gas Sub-Menu
Fresh Air Cal?
Span Cal?
Select Cal Memory?
Change Span Value?
Modify Cal Memory?
Change Correction Factor?

Calibrating the MiniRAE 2000 monitor is a two-point process using "fresh air " and the standard reference gas (also known as span gas). First a "Fresh air" calibration, which contains no detectable VOC (0.0 ppm), is used to set the zero point for the sensor. Then a standard reference gas that contains a known concentration of a given gas is used to set the second point of reference.

Note: The span value must be set prior to calibrating for fresh air or span.

The user can store calibrations for up to 8 different measurement gases. The default gas selections are as follows:

Cal Memory #0.....Isobutylene

Cal Memory #1.....Hexane

Cal Memory #2.....Xylene

Cal Memory #3.....Benzene

Cal Memory #4.....Styrene

Cal Memory #5.....Toluene

Cal Memory #6.....Vinyl Chloride

Cal Memory #7.....Custom?

Memory #0 functions differently than the other 7 memories. For Memory #0, isobutylene is always the calibration gas. When the gas is changed in Memory #0 to one of 100 other preprogrammed chemicals or to a user-defined custom gas, a correction factor is applied to all the readings. During calibration, the unit requests isobutylene gas and displays the isobutylene concentration immediately following calibration, but when the unit is returned to the normal reading mode, it displays the selected gas and applies the correction factor.

The other 7 cal memories require the same calibration gas as the measurement gas. These memories may also be modified to a preprogrammed chemical or to a user-defined custom gas. In the gas library, only the gases that can be detected by the installed UV lamp will actually be displayed. Note that although the correction factor for the new gas will be displayed and can be modified, this factor is not applied when Memories #1-7 are

used. Therefore the factor will not affect the readings in these memories.

Once each of the memories has been calibrated, the user can switch between the calibrated gases by changing the cal memory without the need to recalibrate. Or the user can switch the measurement gas in Memory #0 and the appropriate correction factor will automatically be applied without the need to recalibrate. If the gas is changed in Memories #1-7, it is necessary to recalibrate.

To change a default gas from the list above to a library or custom gas, first go to Select Cal Memory (Section 4.4.3) and then proceed to Modify Cal Memory (Section 4.4.5) to enter the desired gas. If the desired compound does not appear in the preprogrammed library, the user can use the Custom_VOC entry in the library, or the name and correction factor of any of the existing compounds can be changed as described in Section 4.4.5. A list of some 300 correction factors is given in Technical Note 106, available at the website www.raesystems.com.

4.4.1 Fresh Air Calibration

This procedure determines the zero point of the sensor calibration curve. To perform a fresh air calibration, use the calibration adapter to connect the MiniRAE 2000 to a "fresh" air source such as from a cylinder or Tedlar bag (option accessory). The "fresh" air is clean dry air without any organic impurities. If such an air cylinder is not available, any clean ambient air without detectable contaminant or a charcoal filter can be used.

1. The first sub-menu shows: "Fresh air Cal?"
2. Make sure that the MiniRAE 2000 is connected to one of the "fresh" air sources described above.
3. Press the **[Y/+]** key, the display shows "zero in progress" followed by "wait.." and a countdown timer.

After about 15 seconds pause, the display will show the message "update data...zeroed... reading = X.X ppm..." Press any key or wait about 20 seconds, the monitor will return back to "Fresh air Calibration?" submenu.

4.4.2 Span Calibration

This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure. Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar Bag, or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the MiniRAE 2000 Monitor, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

Before executing a span calibration, make sure the span value has been set correctly (see next sub-menu).

1. Make sure the monitor is connected to one of the span gas sources described above.
2. Press the **[Y/+]** key at the "Span Cal?" to start the calibration. The display shows the gas name and the span value of the corresponding gas.
3. The display shows "Apply gas now!" Turn on the valve of the span gas supply.

4. Display shows "wait.... 30" with a count down timer showing the number of remaining seconds while the monitor performs the calibration.
5. To abort the calibration, press any key during the count down. The display shows "Aborted!" and return to "Span Cal?" sub-menu.
6. When the count down timer reaches 0, the display shows the calibrated value.

   Note: The reading should be very close to the span gas value.
7. During calibration, the monitor waits for an increased signal before starting the countdown timer. If a minimal response is not obtained after 35 seconds, the monitor displays "No Gas!" Check the span gas valve is on and for lamp or sensor failure before trying again.
8. The calibration can be started manually by pressing any key while the "Apply gas now!" is displayed.
9. After a span calibration is completed, the display will show the message "Update Data Span Cal Done! Turn Off Gas."
10. Turn off the flow of gas. Disconnect the calibration adapter or Tedlar bag from the MiniRAE 2000 Monitor.
11. Press any key and it returns back to "Span Gas Cal?"

4.4.3 Select Cal Memory

This function allows the user to select one of eight different memories for gas calibration and measurement. For Memories #1-7, the calibration and measurement gas is the same and no correction factor is applied. For Memory #0, the calibration gas is always isobutylene and the measurement gas may be different, in which case the correction factor for that gas is automatically applied. The default gas selections are listed in Section 4.4

1. "Select Cal Memory?" is the third sub-menu item in the Calibration sub-menu. Pressing the **[Y/+]** key, the display will show "Gas =" gas name followed by "Mem # x?"
2. Press **[N/-]** to scroll through all the memory numbers and the gas selections respectively. Press **[Y/+]** to accept the displayed Cal Memory number.
3. After the **[Y/+]** key is pressed, the display shows "Save?" Press **[Y/+]** key to save and proceed. Press **[N/-]** to discard the entry and advance to the next sub-menu.
4. If the gas in a newly selected Cal Memory number is not calibrated, the display shows "CF= x.xx". A correction factor with the value "x.xx" will be applied.
5. If the gas of a newly selected cal memory number has been calibrated previously, the display shows "Last calibrated xx/xx/xx".

4.4.4 Change Span Value

This function allows the user to change the span values of the calibration gases.

1. "Change Span Value?" is the fourth sub-menu item in the Calibration sub-menu
2. Press **[Y/+]**, display shows the gas name and the span value. A cursor will blink at the first digit of the Span value. To modify the span gas value, go to Step 3. Otherwise, press and hold the **[MODE]** key for 1 second to accept the previously stored span gas value and move to the next sub-menu.
3. Starting from the left-most digit of the span gas value, use the **[Y/+]** or **[N/-]** key to change the digit value and press **[MODE]** key momentarily to advance to next digit. Repeat this process until all digits are entered. Press and hold the **[MODE]** for 1 second to exit.
4. The display shows "Save?" To accept the new value, press the **[Y/+]** key. Press the **[N/-]** key or the **[MODE]** key to discard the change and move to the next sub-menu.

4.4.5 Modify Cal Memory

If the current cal memory number selected is not memory 0, users will be prompted whether to modify the settings of the selected cal memory. Press **[Y/+]** to modify the cal memory and **[N/-]** to go to the next sub-menu.

Once **[Y/+]** is pressed the LCD display will show the current memory number, current Gas selected and prompt user for acceptance of current gas selected.

1. Press **[N/-]** to modify the gas selection if desired. Or press **[Y/+]** key to skip the change of gas selection, and proceed to the next sub-menu.
2. After pressing **[N/-]**, display shows "Copy gas from library?" Press **[Y/+]** to accept or **[N/-]** for the next sub-menu, "Enter Custom gas?"
3. In the "Copy gas from library" submenu, use **[Y/+]** and **[N/-]** keys to scroll through the selections in the library. Press **[MODE]** key momentarily to select the gas. The display shows "Save?" Press **[Y/+]** to save or **[N/-]** to discard the changes and proceed to next sub-menu.
4. In the Custom gas sub-menu, the user can enter the gas name. Press the **[Y/+]** or **[N/-]** key to cycle through all 26 letters and 10 numerals. Press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all digits (up to 8 digits) of the custom gas name is entered.

Press and hold the **[MODE]** key for 1 second to exit the name entry mode. The display will show "Save?" Press **[Y/+]** to save the entry, or **[N/-]** to discard the changes.

4.4.6 Change Correction Factor

This function allows the user to change the Correction Factor of the standard calibration gas (only for Cal Memory #0).

1. "Change Correction Factor?" is the sixth sub-menu in the Calibration sub-menu.
2. Press **[Y/+]** key. Display shows the gas name, then the correction factor.

   A cursor blinks at the left-most digit of the correction factor. If user wants to modify the correction factor, go to Step 3. Otherwise, press and hold the **[MODE]** key for 1 second to accept the previously stored correction factor value and return to the first sub-menu of the calibrate/select gas menu.
3. Starting from the left-most digit of the correction factor, use **[Y/+]** or **[N/-]** key to change the digit value and press **[MODE]** key momentarily to advance to the next digit, the cursor will move to the next digit to the right. Repeat this process until all digits are entered. Press and hold the **[MODE]** for 1 second to exit.
4. The display shows "Save?" To confirm the new value, press **[Y/+]** to accept the change. Press **[N/-]** or **[MODE]** to discard the change and return to the first sub-menu, Calibrate and Select Gas.

4.5 Change Alarm Limits

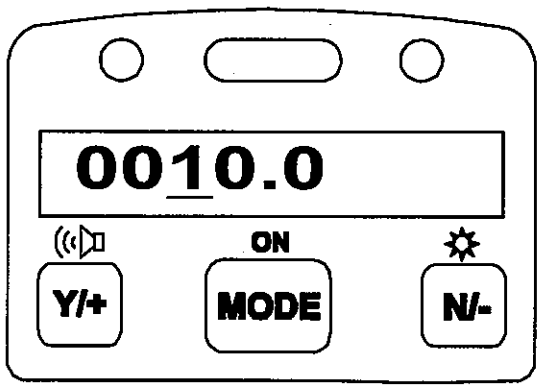
In this menu, the user can change the high and low alarm limits, the STEL limit and the TWA limit (see Table 4.5 below). Press the **[Y/+]** key and the display shows the current gas selected followed by the first sub-menu item below.

Table 4.5

Alarm Limit Sub-Menu
Change High Alarm limit?
Change Low Alarm limit?
Change STEL limit?
Change TWA limit?

1. Scroll through the Alarm Limit sub-menu using the **[N/-]** key until the display shows the desired limit to be changed, e.g.,"High limit?", "STEL limit?", etc.

2. Press the **[Y/+]** key to select the desired limit and the display shows a flashing cursor on the left-most digit of the previously stored alarm limit.



3. To modify this limit value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to its right. Repeat this process until the new limit value is entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
4. If there is any change to the existing value, the display shows "Save?" Press **[Y/+]** to accept the new value and move to the next sub-menu. Press **[N/-]** to discard the changes and move to the next sub-menu.

4.5.1 Change Low Alarm Limit

The second sub-menu item in the Alarm Limit sub-menu allows the user to change the Low Alarm limit. The LCD displays "Low limit?" To change Low Alarm limit, press **[Y/+]** key, or Press **[N/-]** key advance to next sub-menu in Table 4.5.

1. Press **[Y/+]** and the display will show a flashing cursor on the left-most digit of the previously stored Low alarm limit.
2. To modify this limit value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to its right. Repeat this process until the new limit values is entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
3. If there is any change to the existing value, the display shows "Save?" Press **[Y/+]** to accept the new value and move to the next sub-menu. Press **[N/-]** to discard the changes and move to the next sub-menu.

4.5.2 Change STEL Limit

This sub-menu item allows the user to change the STEL limit. The display shows "STEL limit?"

1. Press the **[Y/+]** key and the display will show a flashing cursor on the left-most digit of the previously stored STEL limit.
2. To modify this limit value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new limit values is entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
3. If there is any change to the existing value, the display shows "Save?" Press **[Y/+]** to accept the new value and move to the next sub-menu. Press **[N/-]** to discard the changes and move to the next sub-menu.

4.5.3 Change TWA Limit

This sub-menu item allows the user to change the TWA limit. The LCD displays "TWA limit?"

1. Press **[Y/+]** and the display will show a flashing cursor on the left-most digit of the previously stored TWA limit.
2. To modify this limit value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new limit values is entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
3. If there is any change to the existing value, the display shows "Save?" Press **[Y/+]** to accept the new value and move to the next sub-menu. Press **[N/-]** to discard the changes and move to the next sub-menu.

4.6 Change Datalog

The MiniRAE 2000 monitor calculates and stores the concentration and ID of each sample taken. In the datalog sub-menu, a user can perform the tasks and functions shown below.

Datalog Sub-Menu

Reset Peak/Minimum?

Clear Data?

Change Data Period?

Change Average Type?

4.6.1 Reset Peak

This function will reset the peak and minimum stored in the data memory. Note: this function will not clear the STEL or TWA data.

1. "Reset Peak?" is the first sub-menu item in the Datalog sub-menu (Table 4.6).
2. Press the **[Y/+]** key to reset the Peak/Minimum Values. The display shows "Are You Sure?"
3. Pressing the **[Y/+]** key again will reset the values. The display shows "Peak/Minimum Cleared" and moves to the next submenu.
4. Press the **[N/-]** or **[MODE]** key to exit without resetting the values and move to the next sub-menu.

4.6.2 Clear Data

This function will erase all data stored in the non-volatile datalog memory. Note: This function does not change STEL, TWA, Peak, Minimum and run time values, which are stored in the regular data memory.

1. "Clear Data?" is the third sub-menu item in the Datalog sub-menu.
2. Press the **[Y/+]** key to clear the datalog memory. The display shows "Are you sure?"
3. Press the **[Y/+]** key again to confirm erasure of all the datalog memory.
4. Press the **[N/-]** or **[MODE]** key to exit without clearing the datalog memory and move to the next datalog sub-menu.

4.6.3 Change Data Period

The datalog period can be programmed from 1 to 3,600 seconds (1 hour).

1. "Change Data Period?" is the fifth sub-menu item in the Datalog sub-menu.
2. Press the **[Y/+]** key and the display shows "Datalog Period = XXXX" with the left-most digit flashing, where "XXXX" is the previously stored data log period.
4. To modify this period, starting from the left-most digit, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all 4 digits of the new period are entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
5. If there is any change to the existing value, the display will show "Save?" Press **[Y/+]** to accept the new value or **[N/-]** to discard the changes and move to the next sub-menu.

4.6.4 Change Average Type

The user can select either an 8-hour Time Weighted Average (TWA) or a running Average. The running average is simply the average of all instantaneous (1-second) readings since the measurement was started. This average may increase or decrease with time depending on the readings. The TWA is a cumulative value used to estimate the fraction of the 8-hour limit to which the user has been exposed since the start of the measurement. This value can only increase or remain constant, never decrease. Refer to Technical Note 119 for more information on how TWA is calculated.

1. "Change Average Type?" is the sixth sub-menu in the Datalog sub-menu.
2. Press the **[Y/+]** key to enter the function.
3. The display will show "Running Average?" or " Time Weighted Average?" depending on the current average type.
4. Press **[N/-]** key to toggle between the average types. Press **[Y/+]** key to select the displayed average type.
5. If there is any change to the existing setting, the display shows "Save?" Press **[Y/+]** to save the change. Press **[N/-]** or **[MODE]** to discard the change and return to the first sub-menu.

4.7 Change Monitor Setup

Several monitor specific variables can be changed in this menu. The following is a list of configuration data that can be modified by the user.

Monitor Setup Sub-Menu	Diagnostic Mode
Change Operation Mode?	"
Change Site ID?	Change Unit ID?
Change User ID?	Change Host ID?
Change Alarm Mode?	"
Change User Mode?	"
Change Date?	"
Change Time?	"
Change Lamp?	"
Change Pump Duty Cycle?	"
Change Unit?	"
Change Dilution Ratio?	"
Change Output?	"
Change DAC Range?	"
Set Temperature Unit?	"

4.7.1 Change Operation Mode

MiniRAE 2000 supports two operation modes: Survey and Hygiene mode.

Survey mode: Manual start/stop of measurements and display of certain exposure values.

Hygiene mode: Automatic measurements, running and datalogging continuously and calculates additional exposure values.

1. "Change Op Mode?" is the first sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the **[Y/+]** key and the display shows the current user mode: "Op Mode = *current mode*?"
3. Press the **[Y/+]** key to accept the currently displayed operation (Op) mode. Press **[N/-]** to toggle to the other operation mode. Press **[MODE]** to exit this sub-menu and move to the next monitor setup sub-menu.
4. When changing Op mode from Hygiene to Survey, the display shows the additional message "Warning! Exit Hygiene?" to prevent accidental exit from Hygiene mode. Press the **[Y/+]** key to acknowledge.
5. If there is any change to the existing setting, the display will show "Save?" Press the **[Y/+]** key to accept or the **[N/-]** key to discard and move to the next sub-menu.

Note: If a new Op Mode is saved, the display shows "Op Mode changed!!" when exiting the programming mode.

4.7.2 Change Site ID

The user can enter an 8-digit alphanumeric site ID in the programming mode. This site ID will be included in the datalog report.

1. "Change Site ID?" is the second sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the **[Y/+]** key and the display shows the current site ID: "Site ID = xxxxxxx" with the left most digit flashing.
3. Press the **[Y/+]** or **[N/-]** key to cycle through all 26 letters and 10 numerals. Press **[MODE]** momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all 8 digits of the new site ID are entered.
4. Press and hold the **[MODE]** key for 1 second to exit the data entry mode.
5. If there is any change to the existing site ID, the display shows "Save?" Press the **[Y/+]** key to accept the new site ID. Press the **[N/-]** key to discard the change and move to the next sub-menu.

4.7.3 Change User ID

The user can enter an 8-digit alphanumeric user ID in the programming mode. This user ID will be included in the datalog report.

1. "Change User ID?" is the third sub-menu item the Monitor Setup menu.
2. Press the **[Y/+]** key and the display shows the current user ID: "User ID = xxxxxxxx" with the left most digit flashing.
3. Press the **[Y/+]** or **[N/-]** key to cycle through all 26 letters and 10 numerals. Press **[MODE]** momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all 8 digits of the new user ID are entered.
4. Press and hold the **[MODE]** key for 1 second to exit the data entry mode.
5. If there is any change to the existing user ID, the display shows "Save?" Press the **[Y/+]** key to accept the new user ID. Or press the **[N/-]** key to discard the changes and move to the next sub-menu.

4.7.4 Change Alarm Mode?

There are two different alarm modes: **Latched** and **Automatic Reset** (Auto Reset) in the MiniRAE 2000 that can be selected from the programming menu.

1. "Change Alarm Mode?" is the fourth sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key; the display shows the current alarm mode.
3. Press the **[Y/+]** key to accept the currently displayed alarm mode. Press **[N/-]** key to toggle to the other alarm mode. Press **[MODE]** to exit this sub-menu and move to the next monitor setup sub-menu.
4. If there is any change to the existing setting, the display will show "Save?" Press **[Y/+]** to save the change. Press **[N/-]** or **[MODE]** to discard the change and move to the next sub-menu.

4.7.5 Change User Mode

There are two different user modes: **Display** and **Program** that can be selected from the programming menu.

1. "Change User Mode?" is the fifth sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the **[Y/+]** key; the display shows the current user mode selected.
3. Press the **[Y/+]** key to accept the currently displayed user mode. Press **[N/-]** key to toggle to the alternate user modes. Press **[MODE]** to exit this sub-menu and move to the next monitor setup sub-menu.
4. If there is any change to the existing selection, the display shows messages "Program change" and "Are you sure?" Press **[Y/+]** to confirm the change or press **[N/-]** or **[MODE]** to discard the changes and move to the next sub-menu.

CAUTION: If the user mode is changed to **Display** mode, the user can no longer enter the programming mode. Therefore, the user can not change the user mode back to **Program** mode in normal mode.

To restore the user mode back to **Program** mode, turn the unit off and back on in Diagnostic Mode. Next enter Program mode by holding the **[MODE] and [N/-]** keys for three seconds. Enter the password at the prompt (the default is 0000). Once program mode is entered, go to the "Change Monitor Setup" / "Change User Mode" and change the mode back to **Program.**

An alternative way to change Display mode back to Program mode is through the PC and the ProRAE-Suite software.

4.7.6 Change Date

The MiniRAE 2000 monitor is equipped with a real time clock (RTC). The user can enter the correct date and time (see 4.7.7) for the real time clock.

1. "Change Date?" is the sixth sub-menu item in the Monitor Setup menu.
2. Press **[Y/+]** and the display shows the current date "mm / dd / yy" with the left most digit of the date flashing.
5. To modify this value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new date and time values are entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
4. If there is any change to the existing value, the display shows "Save?" Press **[Y/+]** to confirm the new value or press **[N/-]** or **[MODE]** to discard the changes and move to the next sub-menu.

4.7.7 Change Time

To change the time in the RTC of the MiniRAE 2000:

1. "Change Time?" is the seventh sub-menu item in the Monitor Setup menu.
2. Press **[Y/+]** and the display shows the current time in the 24-hour format "hh : mm" with the left most digit of the time flashing.
3. To modify this value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new date and time values are entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
4. If there is any change to the existing value, the display shows "Save?" Press **[Y/+]** to confirm the new value or press **[N/-]** or **[MODE]** to discard the changes and move to the next sub-menu.

4.7.8 Change Lamp

There are three UV lamps with different photon energies available for the PID sensor: **9.8 eV, 10.6 eV** and **11.7 eV**. The user can select any one of the lamps from the programming mode.

1. "Change Lamp Type?" is the eighth sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the **[Y/+]** key; the display shows the current PID lamp selection.
3. Press the **[Y/+]** key to accept the currently displayed lamp. Press **[N/-]** key to scroll through the sub-menu for other lamp selections. Press **[MODE]** to exit this sub-menu and return to the next sub-menu in Table 4.7.
4. If there is any change to the existing selection, the display will show "Save?" Press **[Y/+]** to save the new selection or press **[N/-]** or **[MODE]** to discard the change and return to the next sub-menu in Table 4.7.

4.7.9 Change Unit

User can change the display and datalog unit from parts per million (ppm) to milli-gram per cubic meter (mg/m^3).

1. "Change Unit?" is the ninth sub-menu item in the Monitor Setup sub-menu.
2. Press the **[Y/+]** key, the display should show the current unit "Display Unit = ppm?" or "Display Unit = mg?"
3. Press **[Y/+]** key to accept the currently displayed unit. Press **[N/-]** key to toggle to the other unit. Press **[MODE]** key to exit this sub-menu.
4. If there is any change to the existing selection, press **[Y/+]** to confirm the new selection or press **[N/-]** or **[MODE]** to discard the changes and move to the next sub-menu.

Caution:

1. **The correction factor in the gas library is calculated based on "ppm" unit. If "mg" unit is selected, the built-in correction factor library is not valid.**
2. **No automatic conversion between "ppm" and "mg/m^3" reading is performed by the monitor.**
3. **When the unit name is changed from "ppm" to "mg", the unit must be recalibrated with the span gas concentration entered in mg/m^3. The converse rule applies when the unit is changed from "mg" to "ppm".**

4.7.10 Change Dilution Ratio

If a dilution system is used upstream of the MiniRAE 2000 inlet port, the user can enter the dilution ratio (from 1 to 10) to compensate the readings. The unit will then display the actual concentration of the gas before dilution. The dilution ratio should be 1 in normal operation where no dilution gas is applied to the sample gas. Dilution improves accuracy and linearity when the concentrations are above a few thousand ppm.

1. "Change Dilution Ratio?" is the tenth sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key; the display shows the current dilution ratio: "Dilution Ratio = xx" with the left most digit flashing.
3. Press the **[Y/+]** or **[N/-]** key to increase or decrease the value of the digit. Press **[MODE]** momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until both digits of the new dilution ratio are entered.
4. Press and hold the **[MODE]** key for 1 second to exit the data entry mode and move to the next sub-menu.
5. If there is any change to the existing dilution ratio, the display shows "Save?" Press **[Y/+]** to confirm the new value or press **[N/-]** or **[MODE]** to discard the changes and move to the next sub-menu.

4.7.11 Change Output?

There are two different external output options: DAC (Analog output) and Alarm in the MiniRAE 2000 that can be selected from the programming menu. The alarm output can be used to connect to the optional vibration alarm (vibrator) only. The analog output, which is proportional to the gas concentration, can be connected a chart recorder or can be queried by a computer to download data in real time (see Technical Note 141).

1. "Change External Output?" is the eleventh sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key and the display shows the current output option selection: "Output = DAC?"
3. Press the **[Y/+]** key to accept the currently displayed output option. Press **[N/-]** to change to the other external option: "Output = Alarm?" Press **[MODE]** to exit this sub-menu and move to the next monitor setup sub-menu.
4. If there is any change to the existing selection the display will show "Save?" Then, press **[Y/+]** to save the change, press **[N/-]** to go back to Step 2, or press **[MODE]** to exit this sub-menu and move to the next monitor setup sub-menu.

4.7.12 Change DAC Range?

There are four different DAC (Digital-to-Analog Conversion) range values available in the **MiniRAE 2000: 20, 200, 2000** and **10K ppm**. The maximum 2.5V DC analog signal output from the unit will represent the range value chosen. (See for analog signal output connection.)

1. "Change DAC Range?" is the twelfth sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key, the display shows the current DAC Range value: "DAC Range = 2000 ppm?"
3. Press the **[Y/+]** key to accept the currently displayed value. Press **[N/-]** to scroll through the sub-menu for other range values. Press **[MODE]** to exit this sub-menu and return to the first sub-menu in Table 4.7.
4. If there is any change to the existing selection, press the **[Y/+]** key and the display will show "Save?" Press the **[Y/+]** key to save the change or press the **[N/-]** key to discard and return to the first sub-menu in Table 4.7.

4.7.13 Set Temperature Unit?

The temperature display can be switched between Fahrenheit and Celsius units.

1. "Set Temperature Unit?" is the thirteenth sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key, and the display shows the current setting: "Temperature Unit = Fahrenheit?"
3. Press the **[Y/+]** key to accept the currently displayed value. Press **[N/-]** to select the sub-menu "Temperature Unit = Celsius?" Press **[MODE]** to exit this sub-menu and return to the first sub-menu in Table 4.7.
4. If there is any change to the existing selection, press the **[Y/+]** key and the display will show "Save?" Press the **[Y/+]** key to save the change and return to the first sub-menu in Table 4.7 or press the **[N/-]** key to discard and return to Step 3..

4.8 Exit Programming Mode

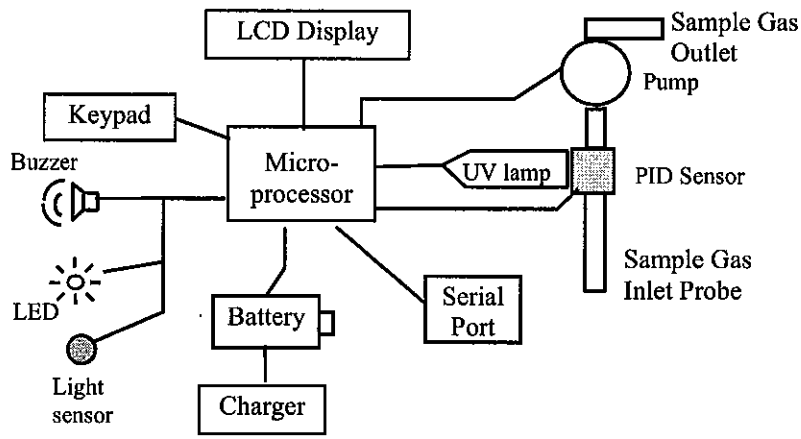
1. To exit programming mode from the first tier menu level, press the **[MODE]** key once.
2. To exit programming mode from 2nd tier sub-menu, press the **[MODE]** key twice.
3. To return to programming mode, press and hold down both the **[MODE]** and **[N/-]** keys for 3 seconds.

5. THEORY OF OPERATION

The MiniRAE 2000 monitor uses a newly developed electrodeless discharge UV lamp as the high-energy photon source for the PID. As organic vapors pass by the lamp, they are photo-ionized and the ejected electrons are detected as a current. The PID sensor with a standard 10.6 eV lamp detects a broad range of organic vapors. A lamp with high photon energy (e.g. 11.7 eV) will measure the more kinds of compounds, whereas low photon energies (e.g. 9.8 eV) are selective for easily ionizable compounds such as aromatics. In principle, any compound with an ionization energy lower than that of the lamp photons can be measured.

The PID sensor for the MiniRAE 2000 monitor is constructed as a small cavity in front of the UV lamp. A diaphragm pump draws the gas sample into the sensor and then pumps it out through the side of the instrument.

Figure 5-1 MiniRAE 2000 Block Diagram



A single chip microcomputer is used to control the operation of the alarm buzzer, LED, pump and light sensor. It measures the sensor readings and calculates the gas concentrations based on calibration to known standard gases. The data are stored in non-volatile memory so that they can be sent to a PC for record keeping. RS-232 transceivers provide a serial interface between the monitor and the serial port of a PC. An LCD display consisting of a single row of eight alpha/numeric characters is used to display the readings. The user interacts with the monitor through three keys on the front panel keypad.

A rechargeable NiMH, NiCd battery, or an alkaline battery pack powers the monitor.

6. MAINTENANCE

The major maintenance items of the MiniRAE 2000 are:

- Battery pack
- Sensor module
- PID lamp
- Sampling pump
- Inlet connectors and filters

Note: Maintenance should be performed by qualified personnel only.

NOTE: The printed circuit board of the monitor is connected to the battery pack even if the power is turned off. Therefore, it is very important to disconnect the battery pack before servicing or replacing any components inside the monitor. Severe damage to the printed circuit board or battery may occur if the battery pack is not disconnected before servicing the unit.

6.1 Battery Charging and Replacement

When the display shows a flashing message "Bat", the battery requires recharging (see Section 3.1 for Battery charging). It is recommended to recharge the MiniRAE 2000 monitor upon returning from fieldwork. A fully charged battery runs a MiniRAE 2000 monitor for 10 hours continuously. The charging time is less than 10 hours for a fully discharged battery. The built-in charging circuit is controlled by the micro-controller to prevent over-charging. The battery may be replaced in the field (in area known to be non-hazardous) if required.

WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non- hazardous. Remove and replace battery only in area known to be non-hazardous.

Replacing Battery Pack

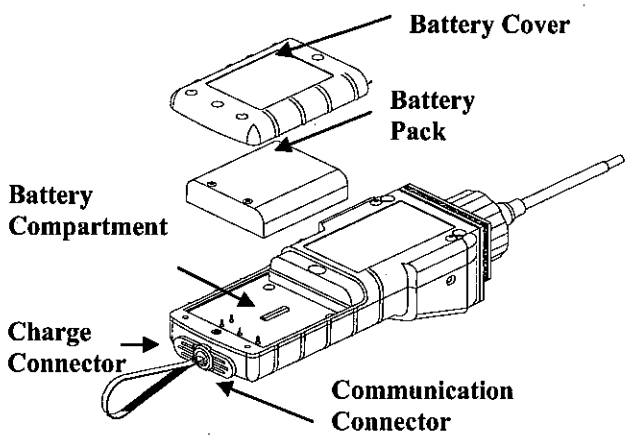


Figure 6-1 Battery Replacement

1.

Turn off the power of the MiniRAE 2000.

2. Unscrew the two battery compartment screws, located on the bottom of the monitor, and remove the cover.
3. Remove the battery pack from the battery compartment.
4. Replace a fully charged spare battery pack inside the battery compartment. Make sure the battery pack is oriented properly inside the compartment
5. Close the battery cover and tighten the two screws.

Replacing Alkaline Battery Adapter

1. Insert four fresh AA size alkaline batteries into the alkaline battery holder. Make sure that the polarity of the batteries is correct.
2. Follow the same procedure as described above to replace the battery holder.

Note: The internal charging circuit is designed to prevent charging to alkaline batteries.

6.2 PID Sensor & Lamp Cleaning/Replacement

The sensor module is made of several components and is attached to the lamp-housing unit as shown in Figure 7-2.

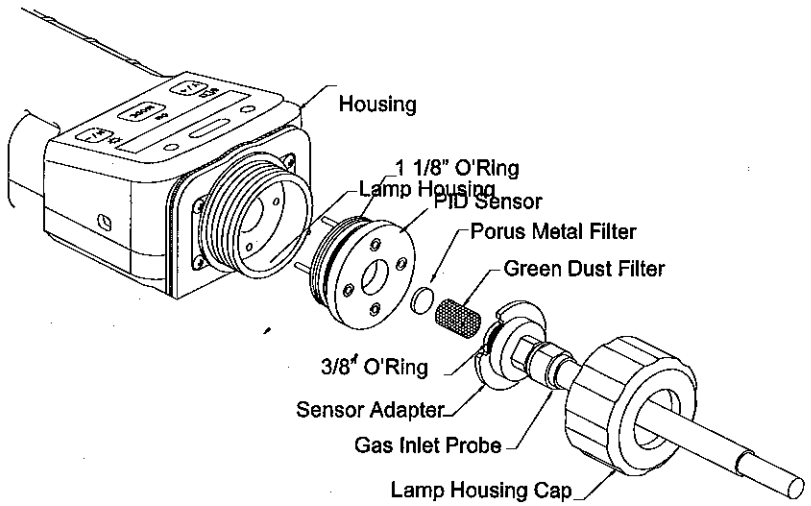


Figure 7-2 Sensor Components

Note: Normally the cleaning procedure is not needed. Clean the PID sensor module, the lamp and the lamp housing only when one of the following happened:

1. The reading is inaccurate even after calibration.
2. The reading is very sensitive to air moisture.
3. A chemical liquid has been sucked into the unit and damaged the unit.

Use of the water trap filter will help prevent contamination and accidentally drawing liquid into the sensor.

To access the sensor components and lamp, gently unscrew the lamp-housing cap, remove the sensor adapter with the gas inlet probe and the metal filter all together. Then hold the PID sensor and pull straight out to avoid bending the electrical pins on the sensor (see Figure 7-2). A slight, gentle rocking motion helps release the sensor.

To clean the PID sensor:

Place the entire PID sensor module into GC grade methanol. It is highly recommended that an ultrasound bath to be used to clean the sensor for at least 15 minutes. Then dry the sensor thoroughly. Never touch the electrodes of the sensor by hand.

Also use a methanol-soaked cotton swab to wipe off the lamp housing where it contacts the sensor when the sensor is installed.

Turn over the sensor so that the pins point up and the sensor cavity is visible. Examine the sensor electrodes for any corrosion, damage, or bending out of alignment. The metal sensor electrode "fingers" should be flat and straight. If necessary, carefully bend the sensor fingers to ensure that they do not touch the Teflon portions and that they are parallel to each other. Make sure that the nuts on the sensor pins are snug but not overtight. If the sensor is corroded or otherwise damaged, it should be replaced.

To clean lamp housing or change the lamp:

To clean lamp housing or change the lamp:

If the lamp does not turn on, the monitor will display an error message to indicate replacement of the lamp may be required.

1. If the lamp is operational, clean the lamp window surface and the lamp housing by wiping it with GC grade methanol using a cotton swab using moderate pressure. After cleaning, hold the lamp up to the light at an angle to detect any remaining film. Repeat the process until the lamp window is clean. Never use water solutions to clean the lamp. Dry the lamp and the lamp housing thoroughly after cleaning.

   CAUTION: Never touch the window surface with the fingers or anything else that may leave a film. Never use acetone or aqueous solutions.

2. If the lamp does not turn on, remove the lamp from the lamp housing. Place the lamp O-ring onto the new lamp. Insert the new lamp, avoiding contact with the flat window surface.
3. Reinstall the PID sensor module.
4. Tighten the Lamp Housing Cap.
5. If the lamp type has been changed, adjust the lamp type setting in the programming mode (Section 4.7.8).

6.3 Sampling Pump

When approaching the end of the specified lifetime of the pump, it will consume higher amount of energy and reduce its sample draw capability significantly. When this occurs, it is necessary to replace or rebuild the pump. When checking the pump flow, make sure that the inlet connector is tight and the inlet tubing is in good condition. Connect a flow meter to the gas inlet probe. The flow rate should be above 450 cc/min when there is no air leakage.

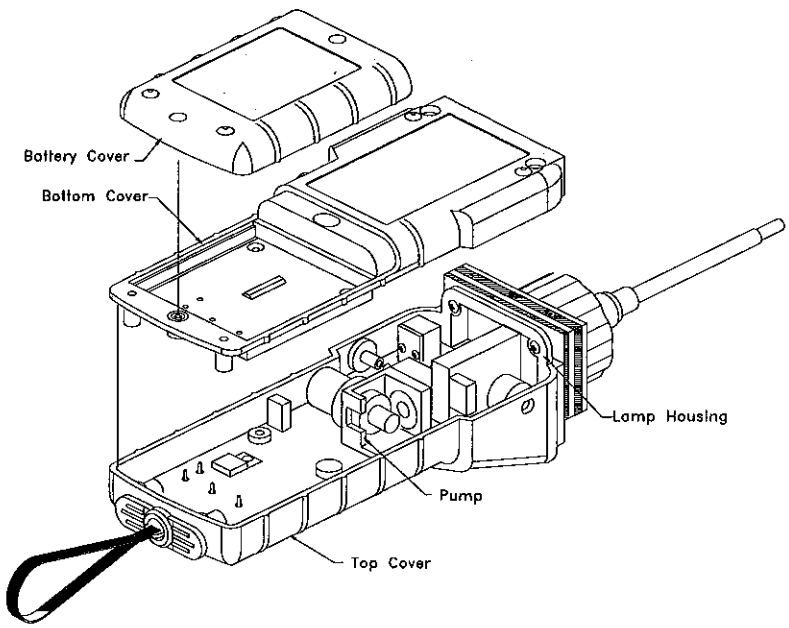


Figure 7-3 Sampling Pump

Pump Replacement

1. Turn off the MiniRAE 2000 power.
2. Open the battery cover, remove the battery pack, and carefully unscrew the six screws to open the bottom cover.
3. Unplug the pump from the PCB. Unscrew the two screws that hold the pump assembly to the PCB. Disconnect the Tygon tubing that connects the pump to the gas inlet port and gas outlet port.
4. Insert a new pump assembly. Connect the Tygon tubing to the gas inlet port. Plug the pump connector back into the PCB and screw down the pump assembly to the board.
5. Replace the bottom cover and tighten the six screws. Re-connect the battery pack. Replace the battery pack and its cover.

6.4 Turning on the UV Lamp

The UV lamp is made of a glass envelope and a UV window (salt crystal) on one end of the envelope. The inside of the lamp is filled with low pressure gases. To turn on the lamp, a high voltage electric field is applied from the outside of the glass envelope. The molecules inside the lamp are ionized and produce a glow discharge that generates the UV light. The MiniRAE 2000 has a built-in sensing mechanism to monitor the status of the UV lamp and display a "Lamp" error message if it is not on.

If the UV lamp has not been used for a long period of time (> 1 month) or is cold, it may become slightly harder to turn on. If such a condition occurs, the "Lamp" message will appear in the monitor display during the power on sequence. This phenomenon is more significant in 0.25" UV lamps used in ToxiRAE and MultiRAE Plus products, because of the relatively small lamp size. To solve this problem, simply turn on and off the monitor a few times and the lamp should turn on. After the UV lamp is turned on for the first time, it should be easier to turn on the UV lamp next time.

It is possible that the UV lamp is actually on when the lamp error message appears. This is because when the lamp becomes old, the internal threshold level to detect lamp failure may have shifted and cause a false alarm. To eliminate such possibility, simply check to see the UV lamp is actually on. This can be done easily by removing the sensor cap and observing the glow light of the UV lamp in a dark place. The user can also feed the monitor with calibration gas and observe if the sensor reading changes. If the reading changes significantly with the gas, the UV lamp is actually on.

A possible failure mechanism for the UV lamp is a leak developed along the seal of the glass envelope. When such condition occurs, the lamp will become very hard or impossible to turn on and will need to be replaced.

7. TROUBLESHOOTING

To aid the user in diagnosing the monitor, a special diagnostic mode can be used displays critical, low level parameters. Section 7.1 describes the operation of the diagnostic mode. Section 7.2 summarizes the frequently encountered problems and suggested solutions. By turning on the MiniRAE 2000 monitor in diagnostic mode and by using the troubleshooting table in Section 7.2, the user can usually correct the problem without having to return the monitor for repair.

WARNING

This function should be used by qualified personnel only! The diagnostic mode allows the user to set several low-level parameters that are very critical to the operation of the monitor. Extra care should be taken when setting these parameters. If the user is not familiar with the function of these parameters and sets them incorrectly, it may cause the monitor to shut down or malfunction.

7.1 Troubleshooting Table

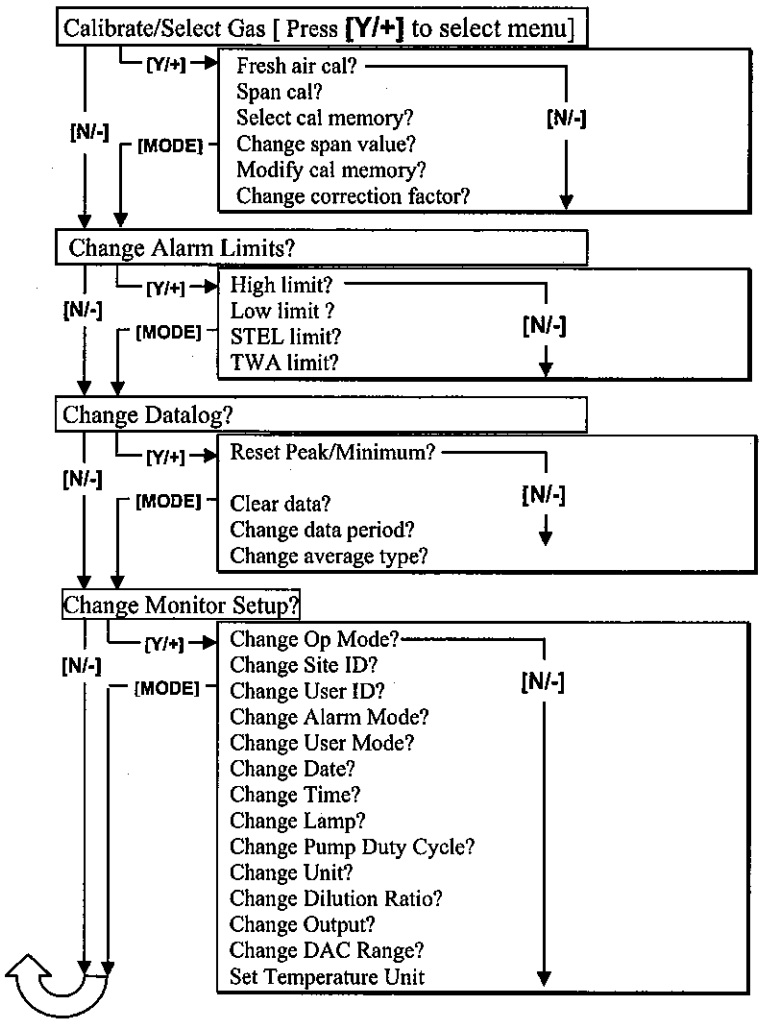
Problem	Possible Reasons & Solutions
Cannot turn on power after charging the battery	**Reasons:** Discharged battery. Defective battery. Microcomputer hang-up. **Solutions:** Charge or replace battery. Disconnect, then connect battery to reset computer.
No LCD back light	**Reasons:** Trigger level too low, the current mode is not user mode, and the mode does not support automatic turn on back light. **Solutions:** Adjust trigger level. Verify the back light can be turned on in user mode. Call authorized service center.
Lost password	**Solutions:** Call Technical Support at +1.408 .752 .0723 or +1. 888 .723 .4800
Reading abnormally High	**Reasons:** Dirty sensor module. Dirty water trap filter. Excessive moisture and water condensation. **Solutions:** Clean sensor module and lamp housing. Replace water trap filter. Blow dry the sensor module.
Buzzer Inoperative	**Reasons:** Bad buzzer. **Solutions:** Call authorized service center.

Inlet flow too low	**Reasons:**	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Replace pump or diaphragm.
"Lamp" message during operation	**Reasons:**	Lamp drive circuit. Weak or defective PID lamp, defective.
	Solutions:	Turn the unit off and back on Replace UV lamp
Full scale measurement in humid environment	**Reasons:**	Dirty or wet sensor.
	Solutions:	Clean and dry sensor and lamp housing. Adjust sensor fingers to ensure not touching Teflon. Use water trap filter.
Reading abnormally low	**Reasons:**	Incorrect calibration. Low sensitivity to the specific gas. Weak or dirty lamp. Air leakage.
	Solutions:	Calibrate the monitor. Replace sensor. Clean or replace lamp. Check air leakage.

APPENDIX A. QUICK REFERENCE GUIDE

Press [N/-] and [MODE], simultaneously, for 3 seconds, to enter Programming Mode. Press [MODE] to return to Survey Mode.

PROGRAMMING MODE



RAE Systems, Inc. Contact Information

Main Office:	3775 N. First St. San Jose, CA 95134-1708 USA
Telephone:	408-952-8200
Fax:	408-952-8480
Instrument Sales:	877-723-2878
Email:	RaeSales@raesystems.com
Website:	www.raesystems.com
Technical Service:	888-723-4800 Tech@raesystems.com

Special Note

If the monitor needs to be serviced, contact either:

1. The RAE Systems distributor from whom the monitor was purchased; they will return the monitor on your behalf.
2. The RAE Systems Technical Service Department. Before returning the monitor for service or repair, obtain a Returned Material Authorization (RMA) number for proper tracking of your equipment. This number needs to be on all documentation and posted on the outside of the box in which the monitor is returned for service or upgrade. Packages without RMA Numbers will be refused at the factory.

Attachment E

ENSR SOP #7315 – Operation/Calibration of a Photoionization Detector (PID)

SOP NUMBER: 7315

Operation/Calibration of a Photoionization Detector (PID)

Date:	3rd Qtr. 2002
Revision Number:	2
Author:	Debra McGrath
Discipline:	Quality Assurance

1.0 INTRODUCTION

1.1 Purpose and Applicability

This document describes the procedures that will be followed by field staff for operation and calibration of a photoionization detector (PID). The PID is primarily used by ENSR personnel for safety and survey monitoring of ambient air, determining the presence of volatiles in soil and water, and detecting leakage of volatiles.

PIDs routinely used by ENSR personnel include the Photovac Microtip, Thermoelectron 580EZ, and MiniRAE 2000. Personnel responsible for using the PID should first read and thoroughly familiarize themselves with the instrument instruction manual.

1.2 Principle of Operation

The PID is a non-specific vapor/gas detector. The unit generally consists of a hand-held probe that houses a PID, consisting of an ultraviolet (UV) lamp, two electrodes, and a small fan which pulls ambient air into the probe inlet tube. The probe is connected to a readout/control box that consists of electronic control circuits, a readout display, and the system battery. Units are available with UV lamps having an energy from 9.5 electron volts (eV) to 11.7 eV.

The PID analyzer measures the concentration of trace gas present in the atmosphere by photoionization. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and become a positive ion. This will occur when the ionization potential of the molecule (in electron volts (eV)) is less than the energy of the photon. The source of photons is an ultraviolet lamp in the probe unit. Lamps are available with energies ranging from 9.5 eV to 11.7 eV. All organic and inorganic vapor/gas compounds having ionization potentials lower than the energy output of the UV lamp are ionized and the resulting potentiometric change is seen as a positive reading on the unit. The reading is proportional to the concentration of organics and/or inorganics in the vapor.

Sample gases enter the probe through the inlet tube and enter the ion chamber where they are exposed to the photons emanating from the UV lamp. Ionization occurs for those molecules having ionization potentials near to or less than that of the lamp. A positive-

biased polarizing electrode causes these positive ions to travel to a collector electrode in the chamber. Thus the ions create an electrical current which is amplified and displayed on the meter. This current is proportional to the concentration of trace gas present in the ion chamber and to the sensitivity of that gas to photoionization.

In service, the analyzer is first calibrated with a gas of known composition equal to, close to, or representative of that to be measured. Gases with ionization potentials near to or less than the energy of the lamp will be ionized. These gases will thus be detected and measured by the analyzer. Gases with ionization potentials greater than the energy of the lamp will not be detected. The ionization potentials of the major components of air, i.e., oxygen, nitrogen, and carbon dioxide, range from about 12.0 eV to 15.6 eV and are not ionized by any of the lamps available. Gases with ionization potentials near to or slightly higher than the lamp are partially ionized, with low sensitivity.

1.3 Specifications

Refer to the manufacturer's instructions for the technical specifications of the instrument being used. The operating concentration range is typically 0.1 to 2,000 ppm isobutylene equivalent.

1.4 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP), hereafter referred to as the project plan.

1.5 Health and Safety Considerations

The health and safety considerations for the site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP). In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

Only PIDs stamped Division I Class I may be used in explosive atmospheres. Refer to the project HASP for instructions pertaining to instrument use in explosive atmospheres.

2.0 RESPONSIBILITIES

2.1 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

2.2 The field operator is responsible for verifying that the PID is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

- Calibration Gas: Compressed gas cylinder of isobutylene in air or similar stable gas mixture of known concentration. The selected gas should have an ionization potential similar to that of the vapors to be monitored, if known. The concentration should be at 50-75% of the range in which the instrument is to be calibrated.
- Regulator for calibration gas cylinder
- Approximately 6 inches of Teflon® tubing
- Tedlar bag (optional)
- Commercially-supplied zero grade air (optional)
- "Magic Marker" or “Sharpie” or other waterproof marker
- Battery charger
- Moisture traps
- Spare lamps
- Manufacturer’s instructions
- Field data sheets or logbook

4.0 METHOD

4.1 Preliminary Steps

4.1.1 Preliminary steps (battery charging, check-out, calibration, maintenance) should be conducted in a controlled or non-hazardous environment.

4.2 Calibration

4.2.1 The PID must be calibrated in order to display concentrations in units equivalent to ppm. First a supply of zero air (ambient air or from a supplied source), containing no ionizable gases or vapors is used to set the zero point. A span gas, containing a known concentration of a photoionizable gas or vapor, is then used to set the sensitivity.

4.2.2 Calibrate the instrument according to the manufacturer's instructions. Record the instrument model and identification number, the initial and adjusted meter readings, the calibration gas composition and concentration, and the date and the time in the field records.

4.2.3 If the calibration cannot be achieved or if the span setting resulting from calibration is 0.0, then the lamp must be cleaned (Section 4.4).

4.3 Operation

4.3.1 Turn on the unit and allow it to warm up (minimum of 5 minutes). Check to see if the intake fan is functioning; if so, the probe will vibrate slightly and a distinct sound will be audible when holding the probe casing next to the ear. Also, verify on the readout display that the UV lamp is lit.

4.3.2 Calibrate the instrument as described in Section 4.2, following the manufacturer's instructions. Record the calibration information in the field records.

4.3.3 The instrument is now operational. Readings should be recorded in the field records.

4.3.4 When the PID is not being used or between monitoring intervals, the unit may be switched off to conserve battery power and UV lamp life; however, a "bump" test should be performed each time the unit is turned on and prior to taking additional measurements. To perform a bump test, connect the outlet tubing from a Tedlar bag containing a small amount of span gas to the inlet tubing on the unit and record the reading. If the reading is not within the tolerance specified in the project plan, the unit must be recalibrated.

4.3.5 At the end of each day, recheck the calibration. The check will follow the same procedures as the initial calibration (Section 4.2) except that no

adjustment will be made to the instrument. Record the information in the field records.

4.3.6 Recharge the battery after each use (Section 4.4).

4.3.7 When transporting, ensure that the instrument is packed in its stored condition in order to prevent damage.

4.4 Routine Maintenance

4.4.1 Routine maintenance associated with the use of the PID includes charging the battery, cleaning the lamp window, replacing the detector UV lamp, replacing the inlet filter, and replacing the sample pump. Refer to the manufacturer's instructions for procedures and frequency.

4.4.2 All routine maintenance should be performed in a non-hazardous environment.

4.5 Troubleshooting Tips

4.5.1 One convenient method for periodically confirming instrument response is to hold the sensor probe next to the tip of a magic marker. A significant reading should readily be observed.

4.5.2 Air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings.

4.5.3 A fogged or dirty lamp, due to operation in a humid or dusty environment, may cause erratic or fluctuating readings. The PID should never be operated without the moisture trap in place.

4.5.4 Moving the instrument from a cool or air-conditioned area to a warmer area may cause moisture to condense on the UV lamp and produce unstable readings.

4.5.5 A zero reading on the meter should not necessarily be interpreted as an absence of air contaminants. The detection capabilities of the PID are limited to those compounds that will be ionized by the particular probe used.

4.5.6 Many volatile compounds have a low odor threshold. A lack of meter response in the presence of odors does not necessarily indicate instrument failure.

4.5.7 When high vapor concentrations enter the ionization chamber in the PID the unit can become saturated or "flooded". Remove the unit to a fresh air environment to allow the vapors to be completely ionized and purged from the unit.

5.0 QUALITY CONTROL

Calibration of the PID will be conducted at the frequency specified in the project plan. In the absence of project-specific guidance, calibration will be performed at the beginning of each day of sampling and will be checked at the end of the sampling day or whenever instrument operation is suspect. The PID will sample a calibration gas of known concentration. The instrument must agree with the calibration gas within ±10%. If the instrument responds outside this tolerance, it must be recalibrated.

Checks of the instrument response (Section 4.5.1) should be conducted periodically and documented in the field records.

6.0 DOCUMENTATION

Safety and survey monitoring with the PID will be documented in a bound field logbook, or on standardized forms, and retained in the project files. The following information is to be recorded:

- Project name and number.
- Instrument manufacturer, model, and identification number.
- Operator's signature.
- Date and time of operation.
- Calibration gas used.
- Calibration check at beginning and end of day (meter readings before adjustment).
- Span setting after calibration adjustment.
- Meter readings (monitoring data obtained).
- Instances of erratic or questionable meter readings and corrective actions taken.
- Instrument checks and response verifications – e.g., battery check, magic marker response (Section 4.5.1) or similar test.

7.0 REFERENCES

United States Environmental Protection Agency. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM). USEPA, Region 4, SESD, Enforcement and Investigations Branch, Athens, GA. November 2001.

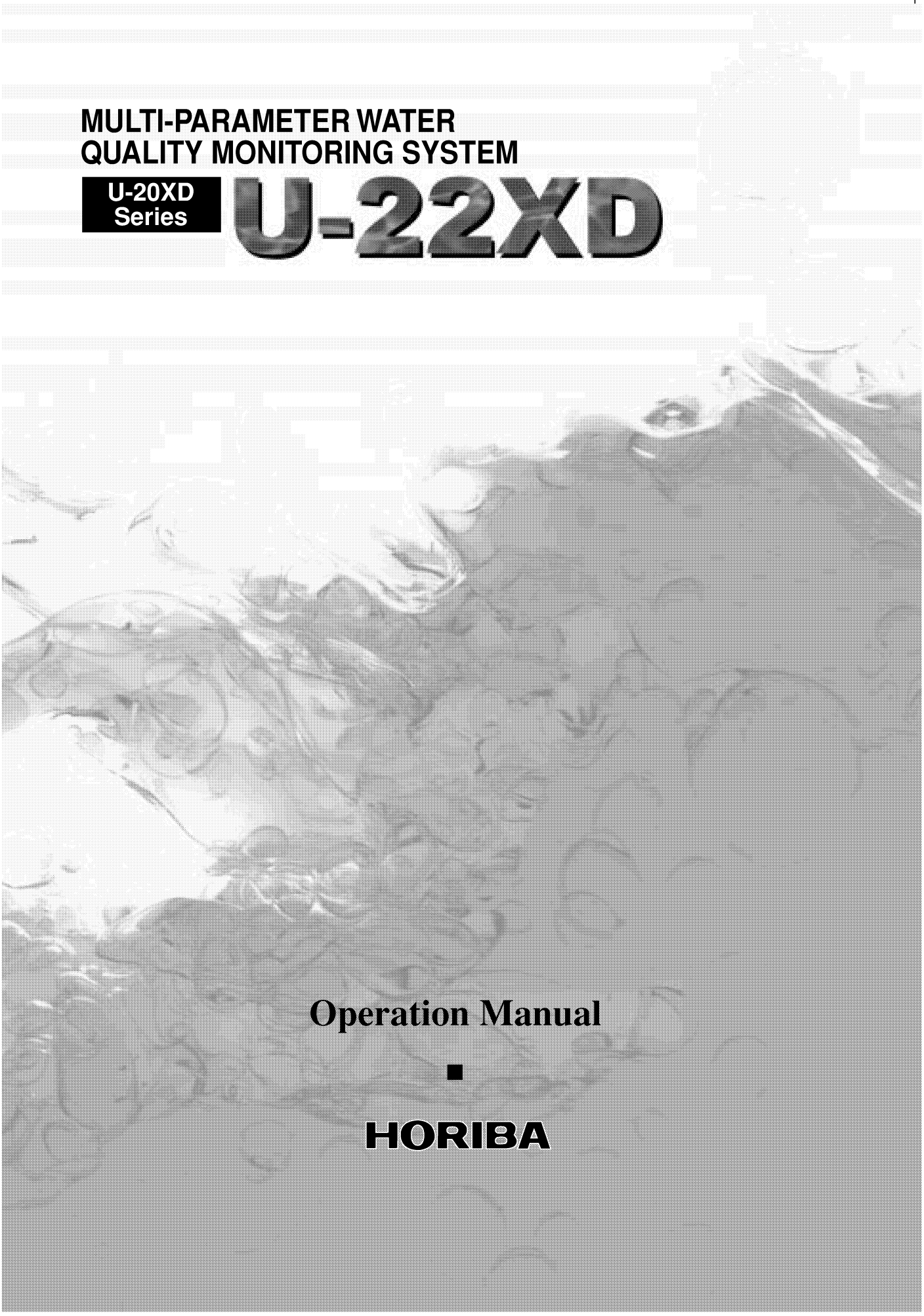
Attachment F

Horiba U-22XD Water Quality Meter Instrument Manual

MULTI-PARAMETER WATER QUALITY MONITORING SYSTEM

U-20XD Series

U-22XD



Operation Manual

■

HORIBA

HORIBA's Warranty and Responsibility

Your U-20XD series multi-parameter water quality monitoring system is covered by HORIBA's warranty for a period of one (1) year, under normal use. Although unlikely, if any trouble attributable to HORIBA should occur during this period, necessary exchange or repairs shall be conducted by HORIBA, free of charge.

The warranty does not cover the following:

- Any trouble or damage attributable to actions or conditions specifically mentioned in the operation manuals to be avoided
- Any trouble or damage attributable to use of the multi-parameter water quality monitoring system in ways or for purposes other than those described in the operation manuals
- If any repairs renovations, disassembly, etc. are performed on this multi-parameter water quality monitoring system by any party other than HORIBA or a party authorized by HORIBA
- Any alteration to the external appearance of this multi-parameter water quality monitoring system attributable to scratches, dirt, etc. occurring through normal use
- Wear and tear to parts, the exchange of accessories, or the use of any parts not specified by HORIBA

INSTALLATION ENVIRONMENT

This product is designed for the following environment:

→ Installation Categories II

→ Pollution degree 2

LIMITATION OF LIABILITY FOR DAMAGES

HORIBA will not accept responsibility for damage or malfunction that may occur as a result of operation or situation not recommended in this manual. HORIBA shall not be liable for Customer's incidental, consequential or special damages, or for lost profits or business interruption losses, in connection with the operation of the Manufactured Parts, CPU hardware, disk drives or Software.

CE MARKING

CE

U-20XD Series conforms with the following directive(s) and standard(s):

Directives:

the EMC Directive 89/336/EEC, in accordance with Article 10(1) of the Directive

the Low Voltage Directive 73/23/EEC

Standards:

[the EMC Directive] EN61326:1997+A1:1998
(EMISSION : Class B, IMMUNITY Category : General)

[the Low Voltage Directive] EN61010-1:1993 +A2:1995

FCC Warning

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Unauthorized reprinting or copying of this operation manual

No unauthorized reprinting or copying of all or part of this operation manual is allowed. The utmost care has been used in the preparation of this operation manual. If, however, you have any questions or notice any errors, please contact the HORIBA customer service printed on the back cover of this operation manual.

© Copyright HORIBA, Ltd. 2001

Preface

Thank you very much for purchasing HORIBA's "MULTI-PARAMETER WATER QUALITY MONITORING SYSTEM" U-20XD Series.

Compact and one-hand-held, our multi-parameter water quality monitoring system makes measurements about a large number of items simultaneously.

The instrument uses a large-sized LCD display and has a variety of functions through easy operation, being useful for use at sites where measurements are to be made.

The water-proof construction of the instrument is compliant with IP-67 of IEC 529, "Water-proof test on electrical and mechanical equipment and tools and protection grade against entry of solids." Please use the instrument by following the information in this Operation Manual to maintain the water-proof construction of the instrument.

IP-67 standards

- Keeping dust and grit out of the instrument
- Up to 5 °C difference between water and an instrument employed and no entry of water into. the inside of the instrument at a depth of 1 m for 30 minutes

This Operation Manual contains information on the basic way of handling the instrument, notes, etc. for the user. Be sure to read through the Operation Manual before use.

Symbols employed

The symbols employed herein have the following meanings:

⚠WARNING : Improper use can result in serious injury or even death.

⚠CAUTION : The improper use of the instrument may cause the following dangers:

- Danger of injury
- Danger of damage to the instrument, its peripherals, and data

: Description of what should never be done, or what is prohibited.

: Description of what should be done, or what should be followed.

Important : Explanation necessary for the proper operation of the instrument

Note : Explanation that is useful and necessary for handling the instrument

☞ : Refer to the item shown.

Symbols employed in screen description

The symbols and used in screen description have the following meanings:

: The letters and numbers in this symbol are blinking on the screen.

: The letters and numbers in this symbol are lighting up on the screen.

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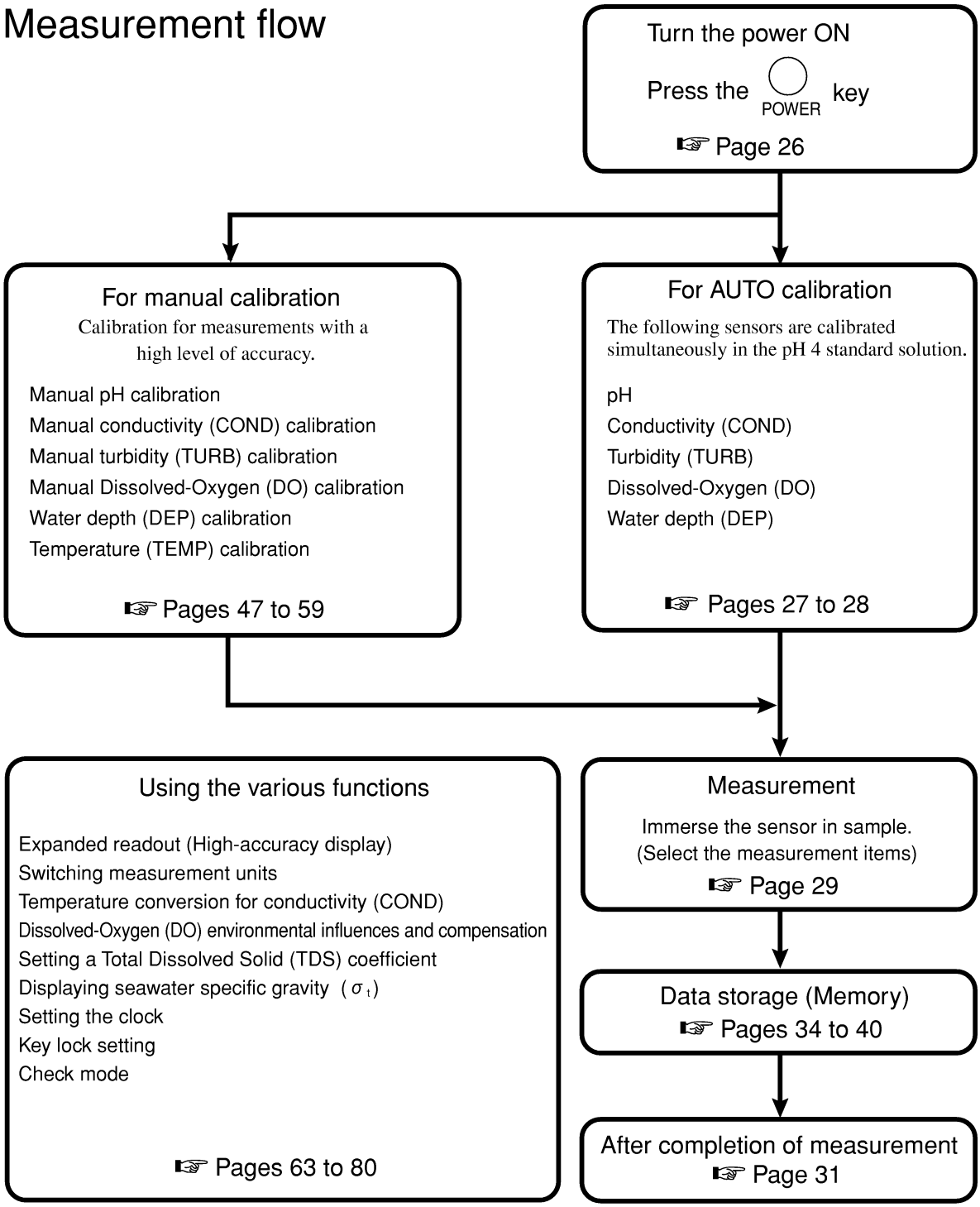
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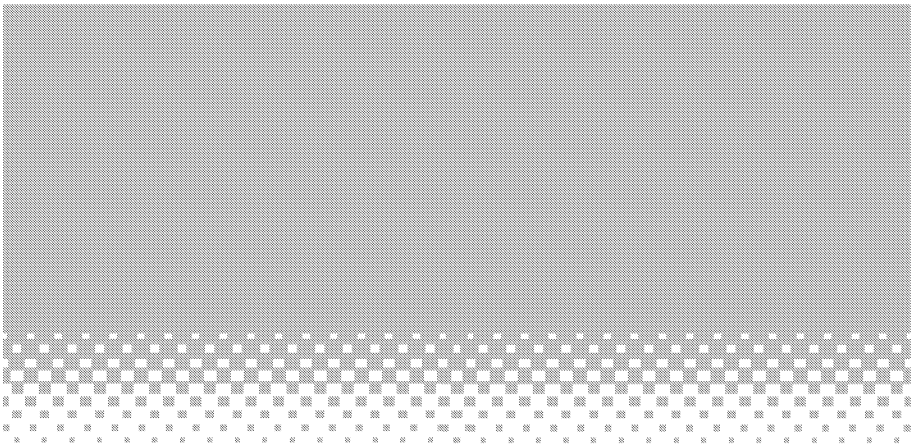
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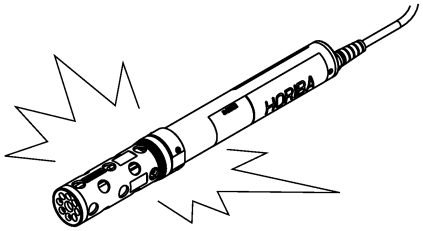
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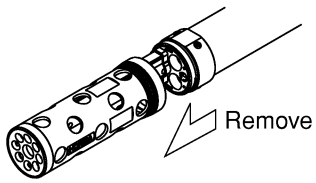
1.1 Notes on handling the instrument

Handling of sensor probe

Do not give a shock to the sensor probe.
The sensor will be damaged.

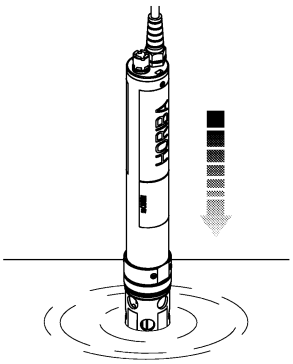


Do not remove the protection cover from the sensor probe to use.
Damage may occur to the sensor.



Slowly lower the sensor probe into the sample.

Dropping it from a height of 1m or more may cause damage to the sensor.
Do not immerse the sensor probe at the depth of exceeding 100 m.
The device can resist the hydraulic pressure at the dept up to 100 m.



- The protection cover may rust due to the environment in which it is used. The damage caused by this usage shall not be warranted by the manufacturer. Solve it with parts which users need to replace periodically.

⚠WARNING

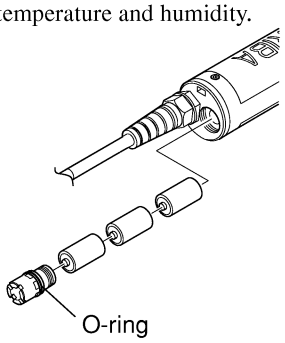
- Fix the sensor probe to the cable or the reel to use.
- In place with a large distance to the water level or with a rapid water flow, fix the sensor probe hook to a point except your body before use for safety purposes.
  Be careful not to let go off the sensor probe by mistake. Otherwise, the sensor probe together with the instrument will fall into the water or a sharp shock will occur to yourself while you are holding the instrument.

Replacing batteries and sensor of the sensor probe

Do not replace the sensor probe batteries and sensor in the atmosphere of high temperature and humidity.

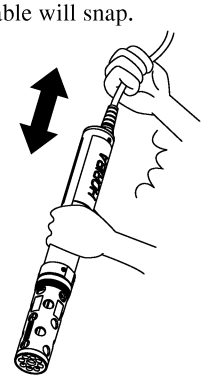
Put connector plugs into the sensor probe connectors with sensors off.

The sensor probe's battery cover is kept waterproof by the use of an O-ring. After checking that there are no foreign bodies adhering to the O-ring, apply silicon grease (included) to the face of the O-ring and close. Be sure to close it all the way to the indicated level. Do not close with the O-ring twisted or warped.

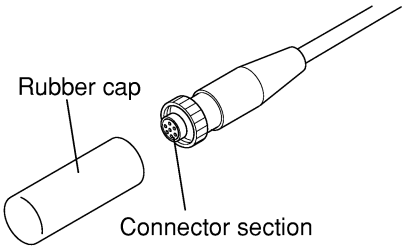


Handling of cable

Do not store the cable with its connector being greatly tensed or bent.
Do not submit the connector to strong shocks or the cable will snap.

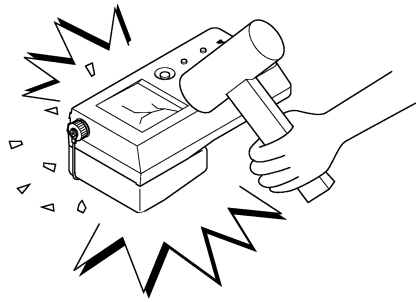


If sample waterdrops remain onto the connector section, metal part of the connector is likely to rust. When storing, wipe the area around the connector well and cover it with the rubber cap.

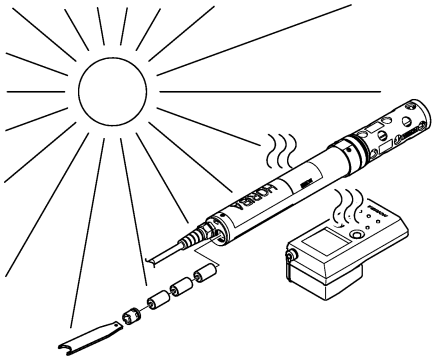


Handling of the instrument

Do not give a shock to or drop the sensor or instrument. The sensor or instrument will be damaged.

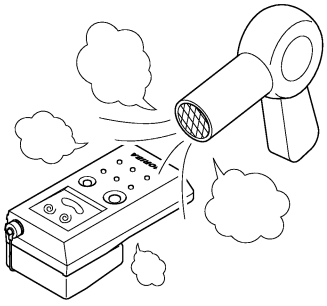


The display part includes LCD. Do not expose the instrument to ultraviolet rays for a long time. Otherwise, the LCD may deteriorate.

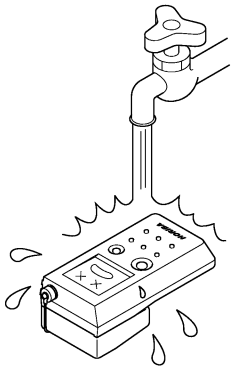


The instrument will be water-proof in construction (IP-67) when the sensor connector is connected to the instrument. However, if the instrument has been dropped into water or become wet, use a soft cloth to dry up the instrument.

Do not use a hair dryer to dry up the instrument.

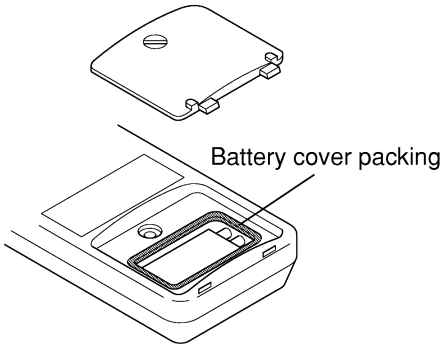


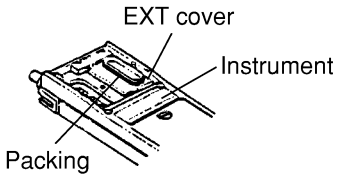
Do not wash directly the instrument using tap water from the faucet.



Note on replacing battery of the instrument and the section to which the EXT unit is attached

Waterproof function of the main unit is maintained by the packing of battery cover and EXT unit cover. Foreign matter on the packing can cause water to enter the instrument. Check for foreign matter on the packing before closing the battery cover and the EXT cover.
If the packing is twisted, do not close the battery cover and the EXT cover.





For a long use

We recommend that the packing be replaced once a year.
For battery cover packing replacement, contact your sales agent.

Note on place for use

- Avoid continuous measurement in water containing alcohol, organic solvent, strong acid, strong alkali or neutral detergent; otherwise the sensor surface will deteriorate.
- Do not use the instrument in the atmosphere with ambient temperatures below 0 °C (incl.) or above 55 °C (incl.).
- Avoid using the instrument in the condition exposed to strong vibrations or corrosive gases.
- Do not use the instrument near a source of strong electromagnetic field such as high-voltage cables and motors.

Batteries

The improper use of batteries may cause leaks and explosion.
Observe the followings:

- Set the batteries in place properly while paying attention to the plus (+) and minus (-) poles.
- Do not use both an old and new batteries or batteries of different types.
- Batteries for use in the instrument are not of the rechargeable type.
- Remove the batteries when not in use for a long time.
  In case of leaks, wipe off the solution in the battery case thoroughly and place new batteries in position.

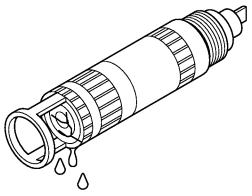
Handling the DO sensor

- In case of breakage of DO sensor diaphragm, replace DO sensor or replace just the diaphragm by using diaphragm replacement unit, without directly touching the internal solution.
- When removing the DO sensor from the sensor probe, make sure to install the short socket (included).

- Do not give a shock to the DO sensor. The sensor will be damaged.

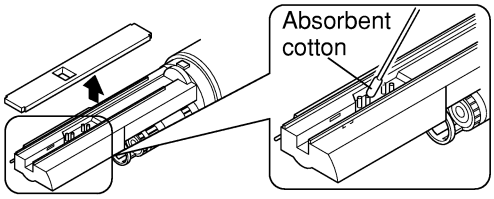
⚠CAUTION

- The DO sensor holds a strong-alkaline solution. Protect the eye and skin from the solution. If there is any solution in the eye or on the skin, immediately use sufficient water to wash off the solution. Consult a doctor as required.



Handling the COND/TURB unit

When cleaning the COND/TURB unit, use an absorbent cotton to avoid damage to the TURB cell.

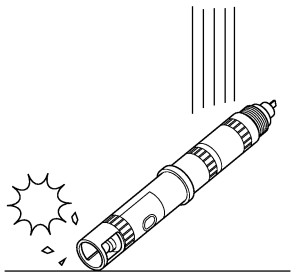


Handling the pH/ORP sensor

The pH/ORP sensor has a glass electrode at the end. Handle the sensor carefully to avoid a break in the glass electrode.

⚠CAUTION

- Be careful not to break the glass on the top of the sensor. Otherwise you may get hurt with a piece of glass.



Disposal

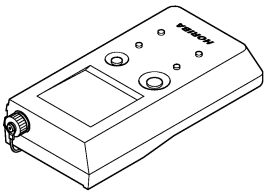
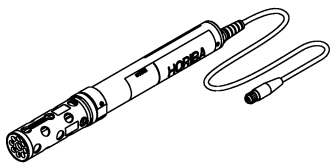
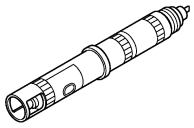
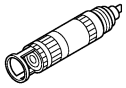
Dispose of this product as special waste, otherwise this may affect the environment.

Handling in transportation

- When transporting this product as freight, use the carrying case to prevent damage.
- Remove the flow cell from the sensor probe in transportation.

1.2 Packing list

The U-20XD series is comprised of the following items.

Model	U-22XD
Meter (U-2000)	
Sensor probe	
Sensor	pH/ORP sensor 
	DO (Dissolved-Oxygen) Sensor 
Accessories	pH 4 standard solution (500 mL)
	pH internal solution (250 mL)
	pH syringe with needle
	Sensor spanner
	Calibration beaker
	Probe cap
	Grip holder
	Carrying case
	Manganese battery 6F22 (006P) (1 piece)
	Alkaline batteries LR03 (AAA) (3 pieces)
	Silicon grease
	Sensor O-ring (S8) (10 pieces)
	Sponge for probe cap (5pieces)
	Operation manual

- The included battery is for the monitor. Its life is not guaranteed.

2. Before Use

2.1 Introduction to the instrument 8
2.1.1 Measurement items 8
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2.1.3 Functions of expansion units 9
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2.1 Introduction to the instrument

2.1.1 Measurement items

Components that can be measured with the U-20XD series models are as follows:

Model	U-22XD
Measurement items	
pH	○
Dissolved Oxygen (DO)	○
Conductivity (COND)	○
Salinity (SAL) [Conductivity conversion]	○
Total dissolved solids (TDS) [Conductivity conversion]	○
Specific gravity of seawater [Conductivity conversion]	○
Temperature (TEMP)	○
Turbidity (TURB)	○
Water depth (DEP)	○
Oxidation-Reduction Potential (ORP)	○

○ ..... Measurable

2.1.2 Introduction to functions of the instrument

Outline of the functions of the instrument is described below.

Feature	Function name	Page
Data obtained during measurement can be saved in the memory.	Manual data storage	Page 34
Data can be automatically saved in the memory at constant time intervals.	Auto data storage	Page 36
Saved data can be called.	DATA OUT	Page 41
The latest date of calibration and its details can be called.	Calibration history	Page 43
Enlarged display is available.	Expand readout	Page 63
Measurement units can be switched.	Switching measurement unit	Page 64

* Other functions possible in the check mode are available. (☞ Page 73)

2.1.3 Functions of expansion units

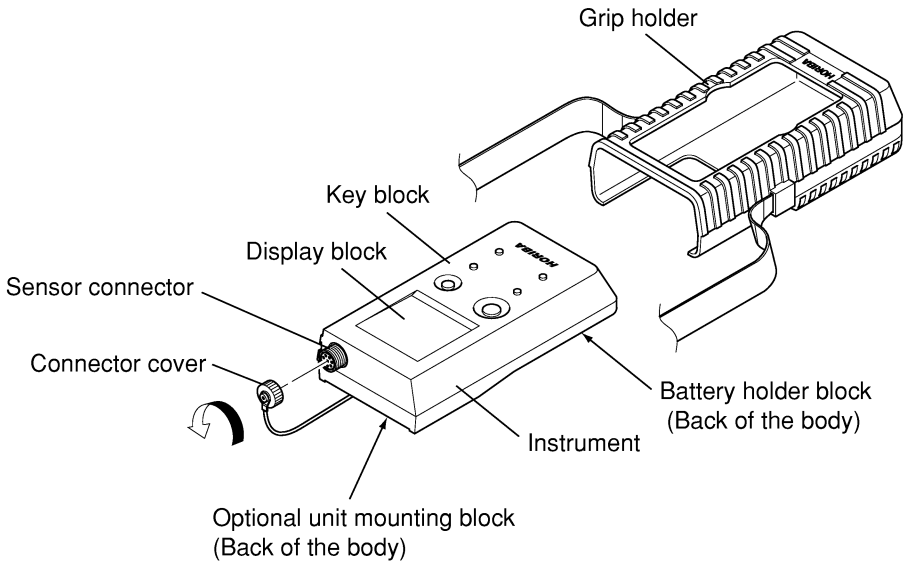
For the U-20XD series, use of expansion units allows communications with personal computers through RS-232C, the storage of G.P.S. data in the memory, and printer output, and commercial power supply.
Expansion units are available in the following two types:

Unit/name	Contents	Functions
U-2001	• Expansion adaptor	<RS-232C communications, G.P.S connection, and printer output>
Expansion adaptor	• Software for PC	The above functions cannot be used at the same time. One of the connectors for these three functions needs to be used.
U-2002	• System unit contain case	<RS-232C communications, G.P.S connection, printer output, battery power supply*>
System unit	• Software for PC	The above functions can be used at the same time.
	• G.P.S. unit	* A battery power supply can be used for measurements outdoors for 30 consecutive days.
	• Printer set	

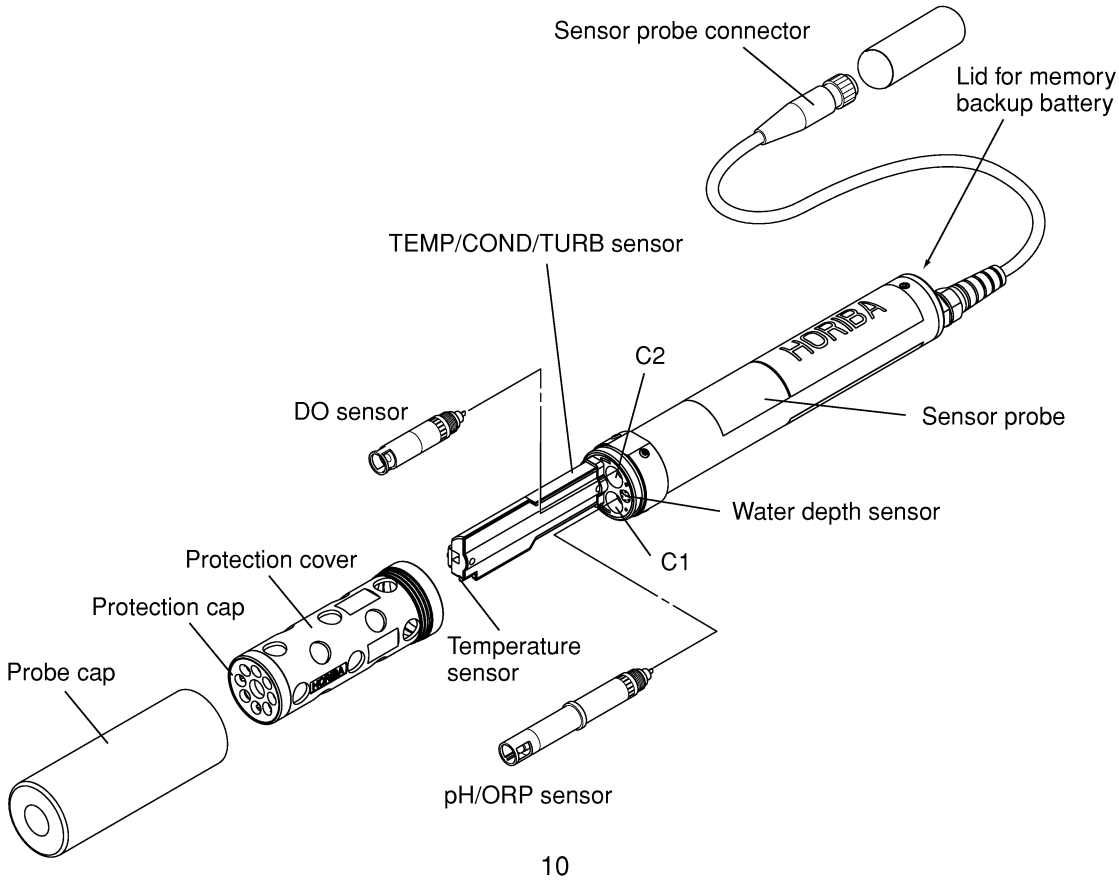
* U-2001 and U-2002 can operate on a commercial power supply through the use of an AC adapter (optional). However, the AC adapter cannot be used for the G.P.S. unit or printer set.

2.2 Names of the parts

2.2.1 Instrument name

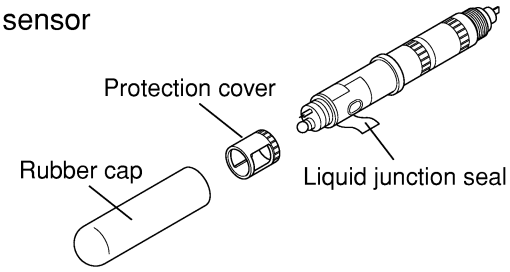


2.2.2 Sensor probe names

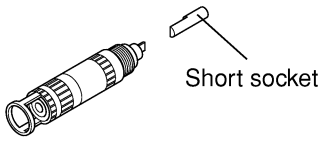


2.2.3 Sensor names

pH/ORP sensor



DO sensor



2.2.4 Use of carrying case

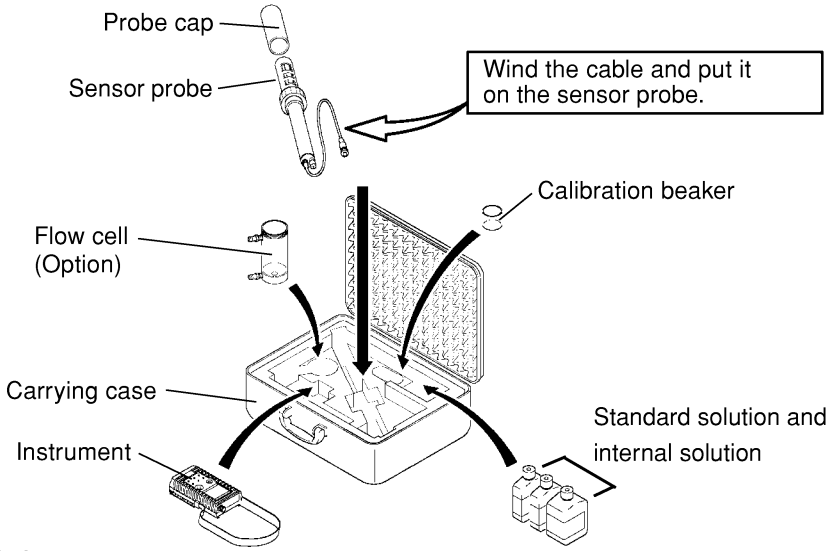
The carring case models W-2010 and W-2030 are applicable to store or transport U-22XD series.

Model	Applied to	Storage temperature	Material
W-2010	Cable length 10 m or less	-5 to 60℃	PP, ABS
W-2030	Cable length 30 m or more		

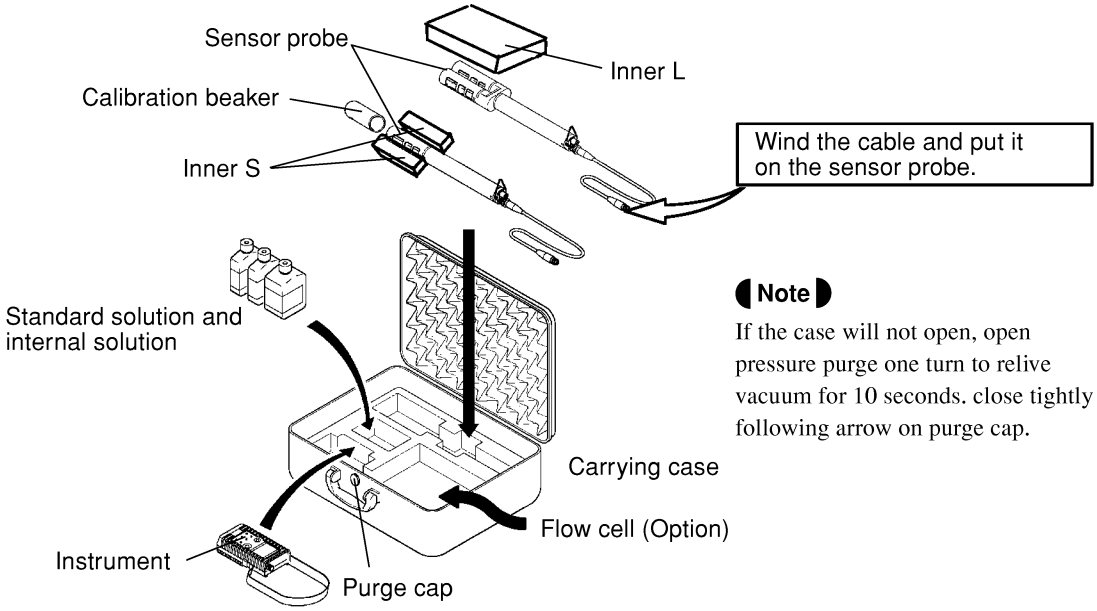
⚠CAUTION

- Do not drop or hit the carring case to protect the units against damage.
- When using the sensor probe with flow cell, separate them for strage.
- Be careful not to catch your finger, when fastening or releasing the laches.

• W-2010



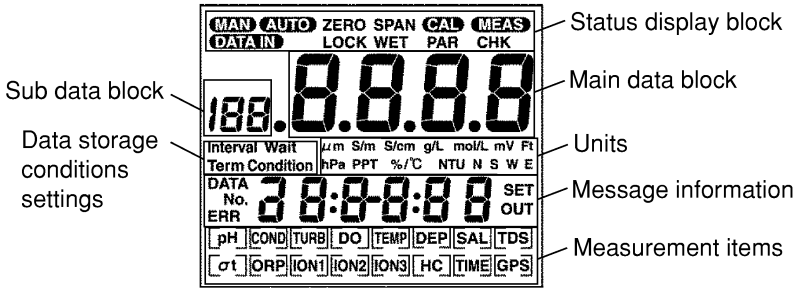
• W-2030



Note

If the case will not open, open pressure purge one turn to relive vacuum for 10 seconds. close tightly following arrow on purge cap.

2.2.5 Display



Status display block

MAN ..... On when the data memory and calibration settings are set to manual.
AUTO ..... On when the data memory and calibration settings are set to automatic.
DATA IN ..... On when the data memory operation and the data memorys operation settings are being performed. Blinking during calibration.
ZERO ..... On in the Zero calibration mode.
SPAN ..... On in the Span calibration mode.
CAL ..... On in the Calibration mode.
MEAS ..... On in the Measurement mode. (Measurements are being made when light up.)
LOCK ..... On when the keys are locked.
CHK ..... On when the instrument is in the check mode.

Sub data block

Display of the pH, Latitude (degree), Longitude (degree), Year and Check No.

Main data block

Display of Measurement data, Latitude (minute, 〔second〕), Longitude (minute, 〔second〕), and month and day.

Data storage conditions setting

Interval ..... On when a time interval is set for storage of data.
Wait ..... On when a time is set for waiting from the automatic data storage instruction until the start and during data processing through individual operations.
Term ..... On when a period is set for automatic data storage.

Units

Displays the units for measurement items.

Message information

Displays the stored data (data mode) and the data No. when the data is stored.
SET ..... Indicates that the instrument is in Set mode.

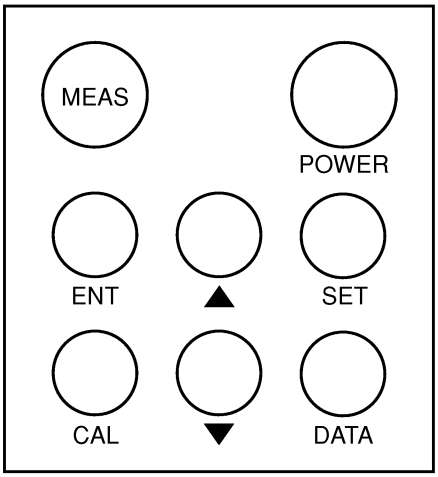
Measurement items

Displays the measurement items for the data in the main data block display. The display is read as follows.
Items without brackets ([ ]) ..... Items with the highlighted text will be stored in the data memory.
(☞ *Measurement item setting*, page 76)
Items with brackets ([ ]) ..... Displays the measurement items with data display.
(☞ *Measurement item setting*, page 76)

Note

- Because of the instrument's automatic power off function, the indication will disappear if the unit is not used for about 30 minutes. For operation of the unit and display of the indication, turn ON the instrument again.

2.2.6 Key names



POWER: Power key

Turns the instrument On and Off. Immediately after the power is switched on, the initial screen is displayed to indicate the status of the instrument.

MEAS: Measurement key

In the Measurement mode (MEAS is on), this key switches the measurement item. In addition, pressing the MEAS key returns you from the Setting, Calibration and Memory Call Up modes to the Measurement mode.

Note

- Regardless of which mode the instrument is in, it is always possible to return to the Measurement mode by pressing the MEAS key.

ENT: Enter key

In the Measurement mode (MEAS is on), pressing the ENT key stores the data in memory.
In the Calibration mode (CAL is on), pressing the ENT key performs calibration.
In the Setting mode, pressing the ENT key switches the setting and registers entered setting values.

CAL: Calibration key

Pressing the CAL key switches the instrument to the Calibration mode. If automatic data storage is in progress, it is aborted.

SET: Set key

Pressing the SET key switches the instrument from the Measurement mode to the Set mode. If the SET key is pressed on the “year, month, day, time” display screen, it switches the instrument to the Check mode.

DATA: DATA key

Pressing the DATA key switches the instrument to the Data mode.

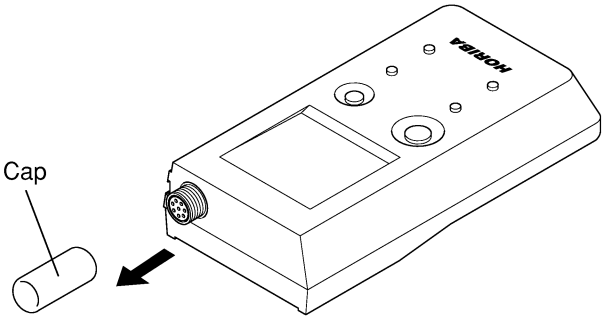
▲▼ : UP/DOWN keys

Use the UP/DOWN (▲▼) keys to set the calibration value in the Manual mode.

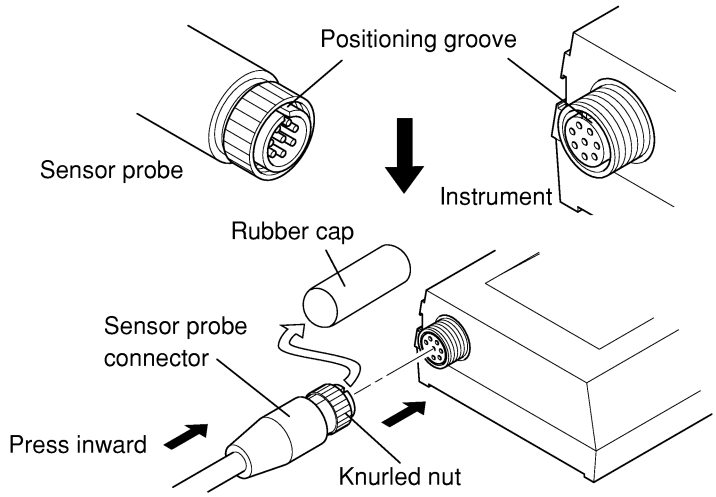
2.3 Setting up the U-20XD series models

2.3.1 Instrument and sensor probe connection

1. Remove the cap from the instrument's connector.



2. Align the positioning grooves of the instrument's connector and sensor probe connectors, and fit the connector of the sensor probe into the this other.



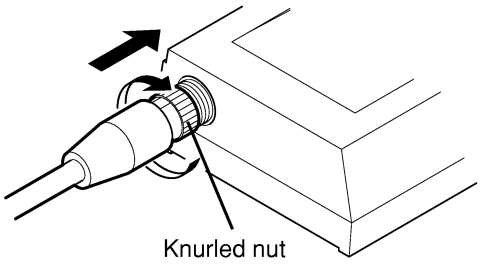
3. Press the sensor probe connector inward and turn. Tighten the connector until it will not turn any more.

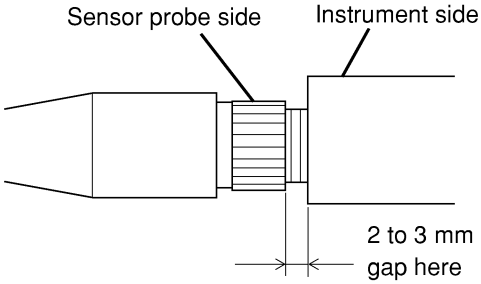
⚠CAUTION

- Turn the knurled nut with holding the knurled part. Otherwise, it will cause breaking of wire.

Important

- The connector cover or sensor probe connector should be connected to the instrument. Otherwise, the instrument will not be waterproof.
- Unless snugly attached, the instrument is not fully waterproof. When the sensor probe connector is tightened as far as it can go, a 2 to 3 mm gap is left between the instrument's connector and sensor probe connector.





Note

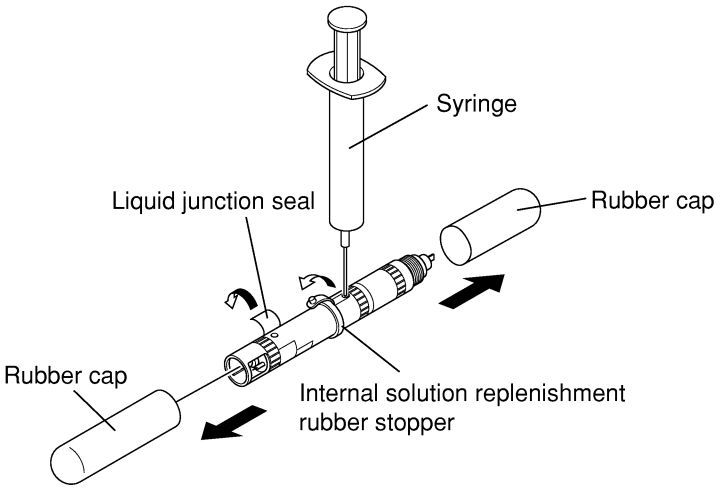
- Tighten the sensor probe connector until it will not turn any more.

2.3.2 Sensor installation

Connect the Dissolved Oxygen (DO) and pH/ORP sensors to the sensor probe.

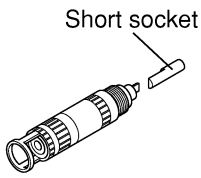
Preparing pH/ORP sensor

1. Remove the liquid junction seal and rubber caps.
2. Open the internal solution replenishment rubber stopper. Then use a syringe to take internal solution (#330).
   Air bubbles in the internal solution may impair the pressure compensation of the sensor. Allow as few air bubbles as possible to enter the inside solution.



Preparing DO sensor

1. Remove the short socket.



Important

- Provide the DO sensor with a short socket or connect the sensor to the sensor probe for storage. Otherwise, the sensor may have a shorter life or stable instructions may not be obtained.
- The short socket is used when storing. Do not throw it away.

Resetting the DO sensor when storing without having installed the short socket.

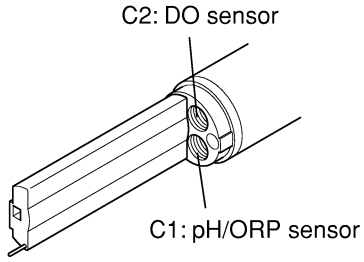
When leaving the DO sensor unattended for a brief period (1 or 2 days) without the short socket, the DO sensor can be reset by connecting it to the short socket or the probe. However, an amount of time corresponding to the period it was left unattended is necessary. If left unattended without being connected to the short socket or the probe for a long period (1 month), it cannot be reset.

Where to attach

1. The hole on the sensor probe in which each sensor is attached is determined by the type of sensor. Check the type of sensor and the assigned hole before attaching anything.

Important

- Installing the sensor in the wrong hole will damage both the sensor and sensor probe.

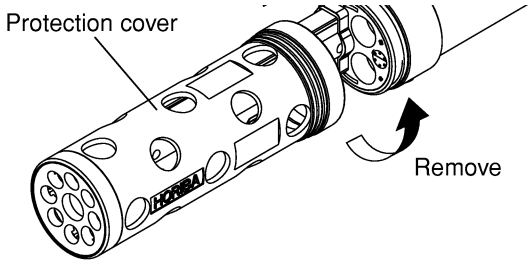


Installation procedure

Important

- With the U-22XD sensor probe install the DO sensor first and then the pH/ORP sensor.
- We recommend that the O-ring of the sensor be replaced with a new one each time the sensor is removed.

1. Remove the probe cap and remove the protection cover from the sensor probe.



When the protection cover's screws are firmly fixed in place and cannot be removed by hand, place a spanner on the protection cover and the surface of the cover guide and remove.

Do not try to remove the protection cover by hitting it or submitting it to shocks.

2. Apply silicon grease to the DO sensor's O-ring.

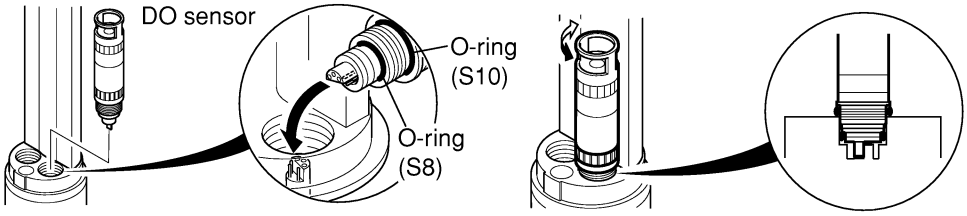
Make sure that no silicon grease gets on the connector.

3. Fit the DO sensor inside the sensor probe hole, being careful to align the shape of the connectors.

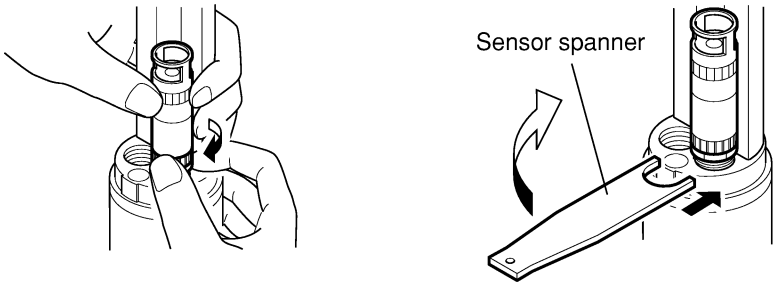
- Make sure that the O-ring is not scratched or twisted. Leakage will cause failures.
- Remove the DO sensor connected to the probe and, when reconnecting them, replace the O-ring (S8) on the smaller end of the DO sensor with a new O-ring.

Important

- Press the sensor slightly inward and try turning to check the fit. The sensor cannot be turned if inserted properly.



4. Turn the screw 2 or 3 turns by hand and then fully tighten with the sensor spanner.

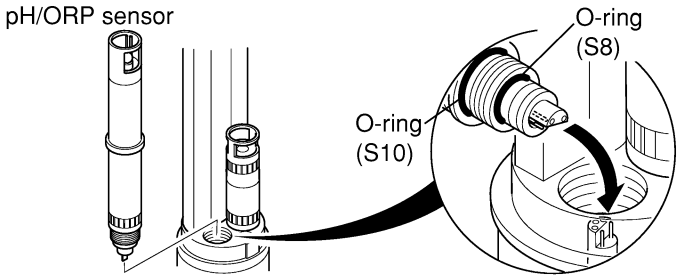


5. Apply silicon grease to the pH/ORP sensor's O-ring.

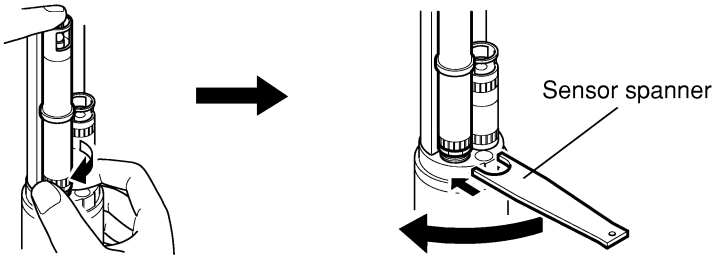
Make sure not to get silicon grease on the connector.

6. Fit the pH/ORP sensor inside the sensor probe hole, being careful to align the shape of connectors.

- Make sure that the O-ring is not scratched or twisted. Leakage will cause failures.
- Remove the pH/ORP sensor connected to the probe and, when reconnecting them, replace the O-ring (S8) on the smaller end of the pH/ORP sensor with a new O-ring.



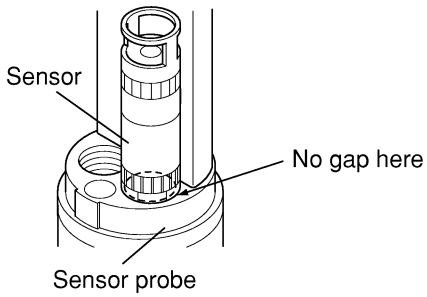
7. Holding the top of the pH/ORP sensor with your finger, turn the screw 2 or 3 turns by hand and then fully tighten with the sensor spanner.



⚠CAUTION

- Unless snugly attached, the sensor is not fully waterproof. The sensor is snugly fit inside the sensor probe when tightened as far as it will go.

Example for DO sensor



8. Attach the removed protection cover to the sensor probe as it was.

Important

- Before attaching each sensor to the sensor probe, do not soak the connector block in water.
- Be careful not to contaminate or wet the sensor probe or sensor connector.

Fasten the guard cover with your hand until it touches the end surface. If improperly fastened, it will slacken and, when storing the instrument, there will be a lack of humidity control. Fastening by hand is enough, do not use a spanner or other tool to fasten or the screws may break.

2.3.3 Installation and replacement of the battery

The U-20XD series is shipped from the factory with the battery packed separately.
When using the instrument for the first time or replacing the battery, perform the following procedure:

Type of battery:

Instrument (U-2000) ..... Alkaline battery 6LR61 (Manganese battery 6F22 [006P])
1 piece. (Battery for instrument operation)
Sensor probe ..... Alkaline batteries LR03 [AAA] (Manganese battery [R03])
3 pieces. (Battery for memory backup)

Notes on handling the battery

The improper use of battteries may cause leaks and explosion.
Observe the followings:

- Set the batteries in place properly while paying attention to the plus (+) and minus (-) poles.
- Do not use both an old and new batteries at a time or batteries of different types.
- Battteries for use in the instrument are not of the rechargeable type.
- Remove the batteries when not in use for a long.
  In case of leaks, wipe off the solution in the battery case thoroughly and place new battteries in position.

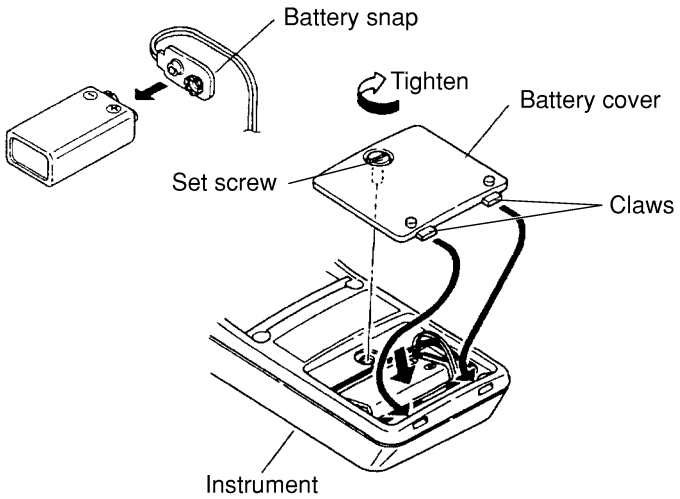
Note

- The battery originally attached to your unit is for monitor and the service life of the battery cannot be guaranteed.

Instrument (U-2000)

1. Loosen the set-screw on the battery cover and remove the cover.
2. Remove any old battery.
3. Fit the battery snaps to a new battery and insert the battery assembly into the instrument.
4. Insert the claws on the battery cover into the grooves in the instrument. Then tighten the set screw.

   The battery snap may be loose for some batteries. In such a case use radio pliers and tighten the metal snap fittings.

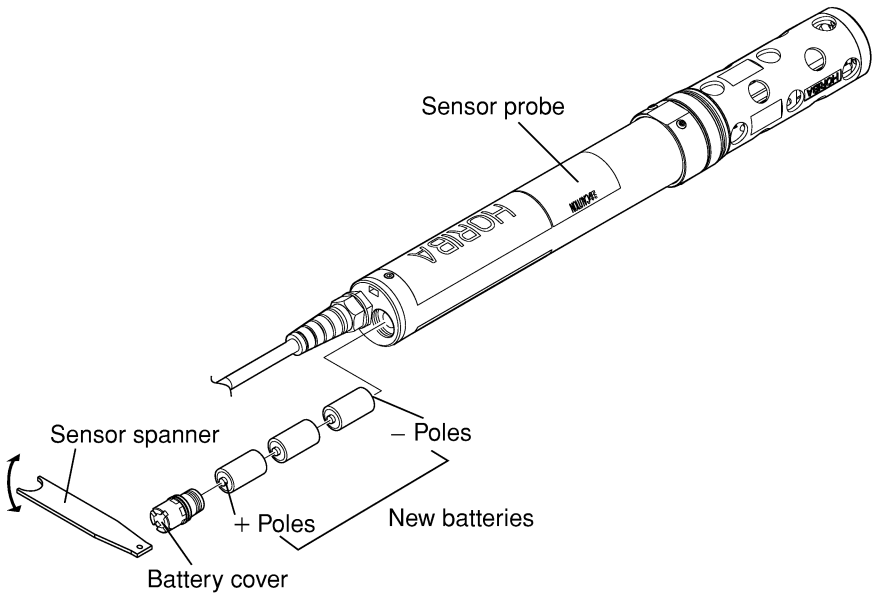


Important

- When removing the battery snap, do not pull it too strongly.

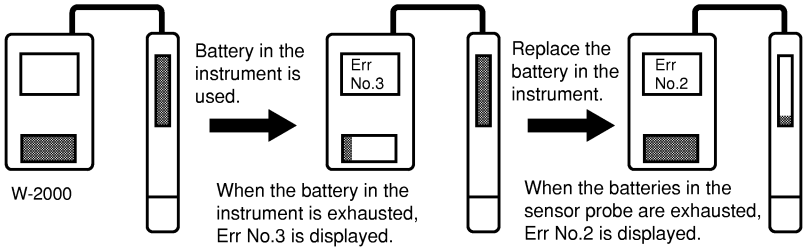
Sensor probe (for memory back up)

1. Remove the battery cover using a sensor spanner or a suitable object.
2. Remove any old batteries.
3. Insert new batteries making sure that the plus (+) and minus (-) poles match the terminals correctly.
4. To keep the sensor probe water-resistant, use a chip spanner as illustrated below and tighten the battery cover until the cover does not turn any more.



⚠ CAUTION

- When replacing the batteries of the sensor probe, be sure to connect the sensor probe to the instrument. Otherwise, the memory will be reset and all the data saved in the memory will disappear.
- When the sensor probe is connected to the instrument, battery in the instrument is consumed.



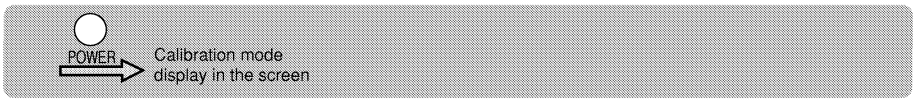
Note

- The battery on the main unit is used up first allowing up to 30 hours use at room temperature. (When using alkaline batteries.)
- Life is reduced by approximately one half when manganese batteries are used.

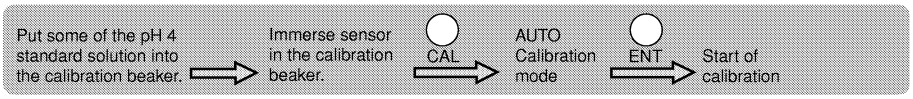
3. Basic operation

The pH, conductivity (COND), turbidity (TURB), dissolved-oxygen (DO) and water depth (DEP) sensors can be calibrated automatically. Upon completion of this chapter, even beginners should be able to make measurements easily.

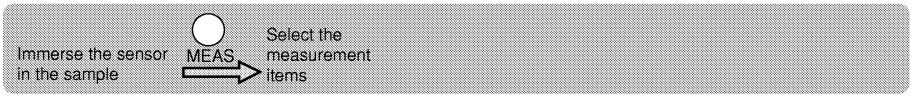
3.1 Key operations and mode switching 24
3.2 Operation procedure 26
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3.2.2 AUTO calibration method 27



3.2.3 Measurement 29



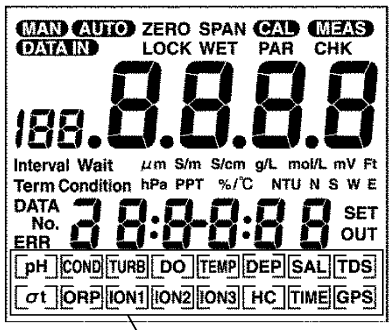
3.2.4 After completion of measurement 31

3.1 Key operations and mode switching

Measuring items and displays which are switched with the MEAS key

The items measurable with individual models are displayed. The items selected with the MEAS key will be indicated with [ ].

Example: In the pH Measurement mode: [pH]



Display block

The symbols displayed and their meanings are as follows:

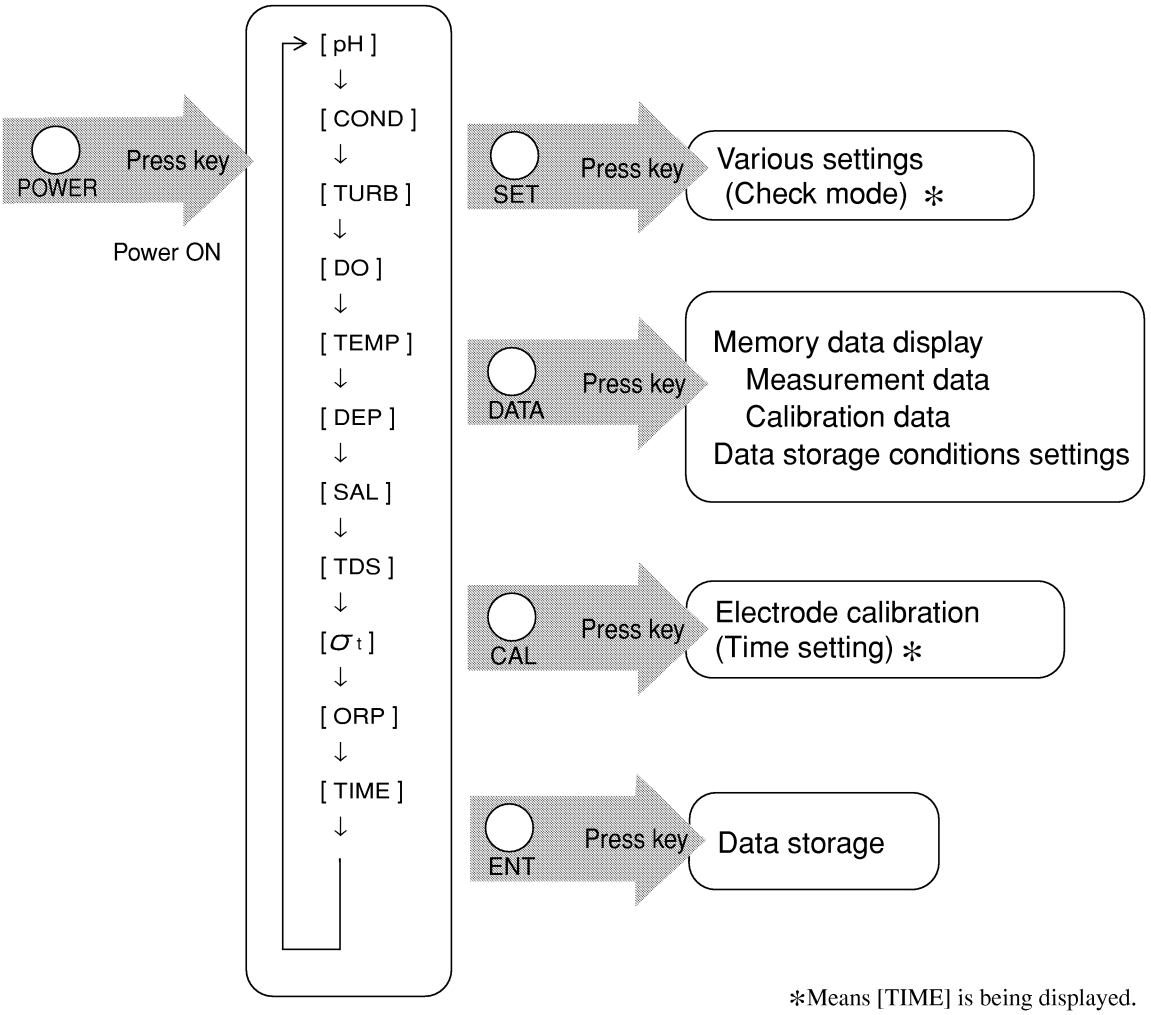
pH ..... pH
COND ..... Conductivity
TURB ..... Turbidity
DO ..... Dissolved-Oxygen
TEMP ..... Temperature
DEP ..... Depth
SAL ..... Salinity
TDS ..... Total dissolved solids
σ_t ..... Specific gravity of seawater
ORP ..... Oxidation-reduction potential
TIME ..... Display of date and time
GPS ..... G.P.S. (Global Positioning System) for imformation of position

Note

- [GPS] lights up when the optional G.P.S. sensor has been connected to the instrument and position information is received from the G.P.S. sensor during the measurement. For more information, refer to the instruction manual for the expansion units.

Measurement mode

MEAS When the MEAS key is pressed, the next measurement item appears.



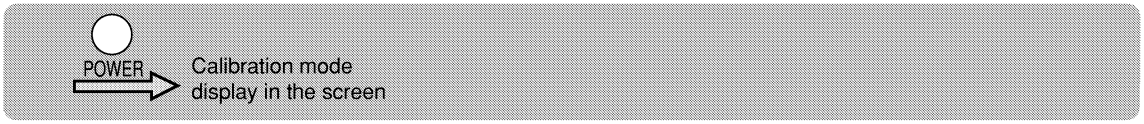
∗Means [TIME] is being displayed.

Note

- "*Measurement item setting*" on page 76 explains how to set the display so items are not displayed.

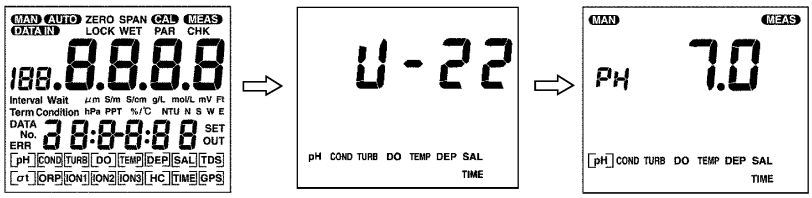
3.2 Operation procedure

3.2.1 Power ON

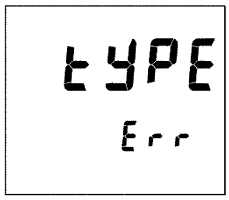


1. Press the **POWER** key.

The display will change in the order of All segment display → Sensor detector display → pH Measurement mode.



With the sensor probe is not connected,



is displayed.

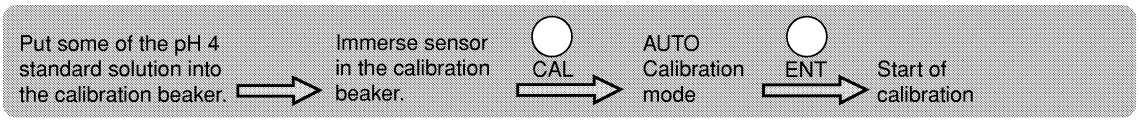
Before turning ON the instrument, connect the sensor probe properly.

3.2.2 AUTO calibration method

To obtain correct measurement, it is necessary to calibrate the sensor using the standard solution before performing measurement.
Previous calibration records shown in calibration log.
(☞ 4.3.2 *Calling up The calibration log*, page 43.)

Note

- In the AUTO calibration mode, the pH, COND, and TURB sensors are calibrated in the pH 4 standard solution, and the DO and DEP sensors in the atmosphere simultaneously.
  Calibrate contents at 25°C are as follows:
  pH: set at 4.01 (zero calibration) and the Span is the adjustment value at the factory when shipping.
  COND: 0.449 S/m (Span calibration), the Zero is the adjustment value at the factory when shipping.
  TURB: 0 NTU (zero calibration), the Span is the adjustment value at the factory when shipping.
  DO: 8.52 mg/L (Span calibration), the Zero is the adjustment value at the factory when shipping.
  DEP: 0 m (Zero calibration), the Span is the adjustment value at the factory when shipping.
- Values may be unstable if there is temperature fluctuation. Calibrate after waiting for about an hour.

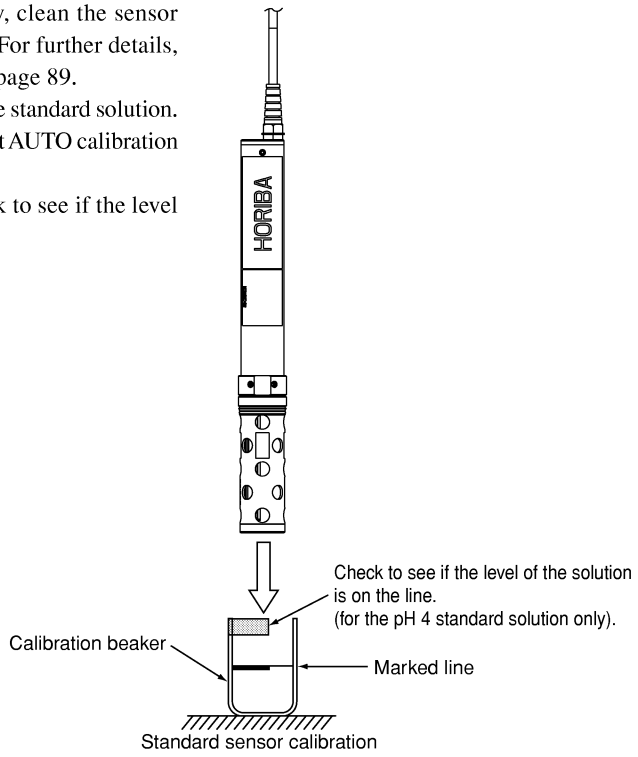


Calibrate using the following procedure.

1. Wash the sensor in distilled water a few times and put some of the pH 4 standard solution into the calibration beaker to the marked line. Then immerse the sensor in it.

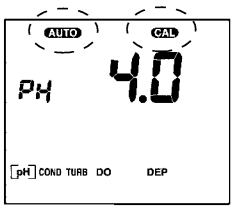
Important

- To carry out calibration for turbidity accurately, clean the sensor surface that will be soaked in standard solution. For further details, see "Troubleshooting for the TURB sensor" on page 89.
- Use the "100-4" manufactured by HORIBA for the standard solution. With other standard solutions, you cannot carry out AUTO calibration correctly.
- Use the label on the calibration beaker and check to see if the level of the calibration solution is on the label line.



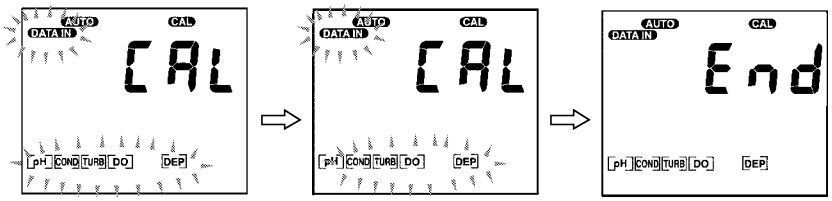
2. Press the **CAL** key in one of the Measurement modes pH, COND, TURB, DO and DEP.

AUTO and **CAL** appear and the instrument enters the AUTO Calibration mode.



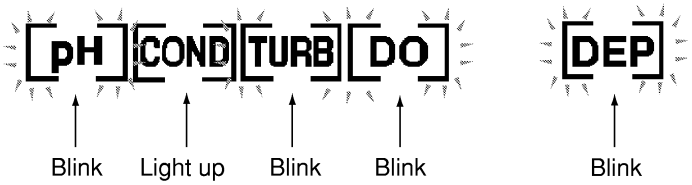
3. Press the **ENT** key to start AUTO Calibration.

Upon completion of all of the pH, COND, TURB, DO, and DEP modes, End will be displayed.
During calibration, **DATA IN** and [ ] for the selected measurement item blink. [ ] light up for the item of which calibration is finished.



With DATA IN is blinking
To stop calibrating the sensor .... press the CAL key.
To establish the calibration ..... press the ENT key.

Example: When COND calibration is finished:
[ ] for [COND] stops blinking and light up steadily.



Note

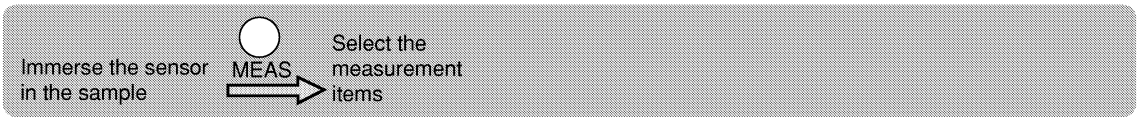
- [ ] continues to blink because calibration is not performed for the item for which an error has happened. If two or more errors happen, an error with a smaller number appears. (See pages 85 to 88 for these errors and ways to solve them.) These calibration errors disappear when the sensor is calibrated properly again, or when the instrument is turned ON again.
- Calibration should be performed for maximum three minutes. When the indications become stable, calibration should be finished.

4. Press the **MEAS** key to return to the Measurement mode.

Important

- Neutralize any basic pH 4 fluids before disposal.

3.2.3 Measurement



1. Immerse the sensor in the sample.

2. Select the measurement item.

Use the MEAS key to switch measurement items in the following order:

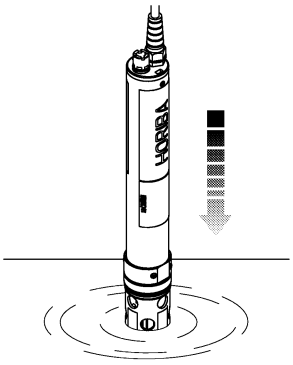
pH → COND → TURB → DO → TEMP → DEP → SAL → TDS → σ_t → ORP → TIME → then back to pH.

Note

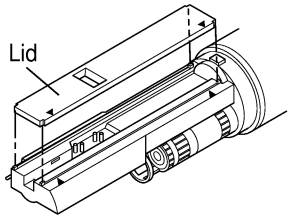
- [GPS] lights up when the optional G.P.S. sensor is connected to the instrument and position information is received from the G.P.S. sensor.
- The above measurement items can be changed by setting ""*Measurement item setting*" described on page 76.

Important

- When immersing the sensor probe in the sample, slowly lower the sensor probe into the sample.
- Dropping it from a height of 1m or more may cause damage to the sensor.



- Don't remove the COND/TURB lid during calibration or measurement.
- Attach the lid to the cell with fitting four corners and facing ▲ marks each other.



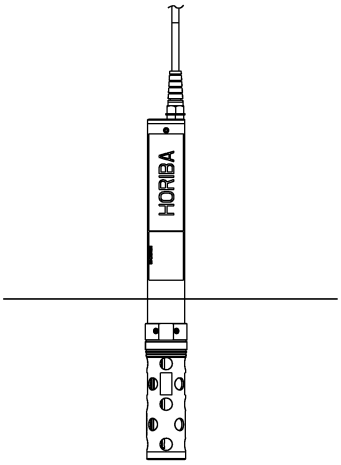
- Perform AUTO calibration after attaching the lid again, when the lid has been removed for the cleaning. A slight difference of the fitting position of the lid causes the difference of the indicated value for turbidity.
- Contacting with a different kind of metal, protection cover of the sensor probe may cause an error in measurement. Be careful not to let protection cover touch with any metal in measurement.

Two useful uses of the U-20XD Series models

Making measurements

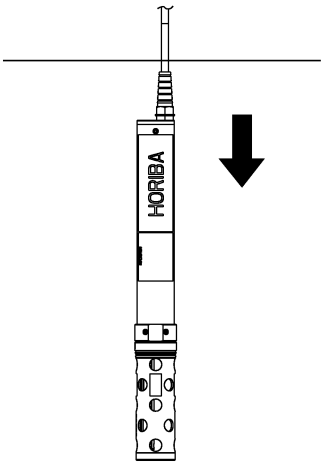
1. Manually storing the measurement data after checking the indication becomes stable

Example: After switching measurement items with the MEAS key, you can then store the measurement data after checking the indication becomes stable.
(☞ 4.1 *Manual storage of data while monitoring the measurement data*, page 34.)



2. Storing data

Example: Data can be stored continuously at constant intervals from the start of the automatic data storage. This function is useful in obtaining data in depth direction and in storing data continuously.
(☞ 4.2 *Automatic data storage*, page 36.)



Notes in obtaining data on depth

- When the instrument is placed at a depth of 100 m or more, the instrument may be broken.

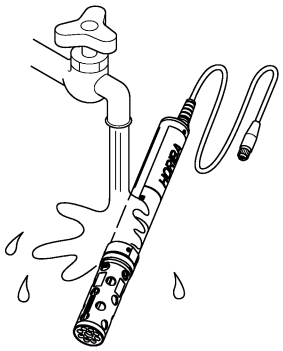
Notes for reliable measurements

- Any sensor contamination may affect measurements. Use the AUTO calibration mode to check for contamination on sensors about once a week for measurements.

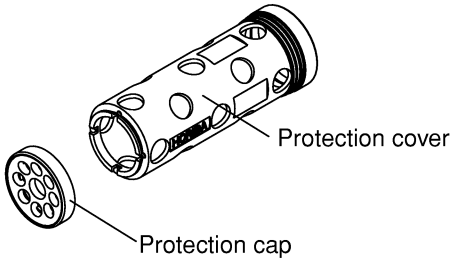
3.2.4 After completion of measurement

1. Turn the power to the instrument off.

2. Use tap water to completely wash off the sample on the sensor and then wipe waterdrops.



Remove the protection cover once and completely wash out with tap water the left over sample on the screws. Reinstall the cover after having wiped off the drops of water. If there is any sample (especially sea water) left over on the screws, rust may form which may prevent the protection cover from being removed. (☞ Installation procedure, page 18.)
Depending on the level of contamination, remove the rubber protection cap from the tip of the protection cover and wash out with tap water. Reinstall it after wiping off the drops of water.



3. Pour about 20 mL (about 2 cm from the bottom)of pure water in the probe cap and install it on the sensor probe. Place the rubber cap on the connector and store the instrument in the carrying case. (☞ 2.2.2 Sensor probe names, page 10.)

When storing with the ph/ORP and DO sensors attached to the probe, make sure to install the probe cap after having poured pure water into it.
Letting the ph/ORP and DO sensors get dry may cause deterioration of the instrument's performance. Should the sponge inside the probe cap be contaminated, replace it with a clean sponge (included).

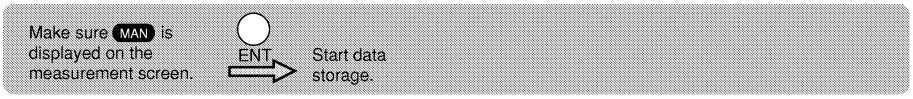
Now you have read the description for performing measurements. For further information on how to use the instrument, refer to the chapters hereafter.

MEMO

4. Using the data memory function

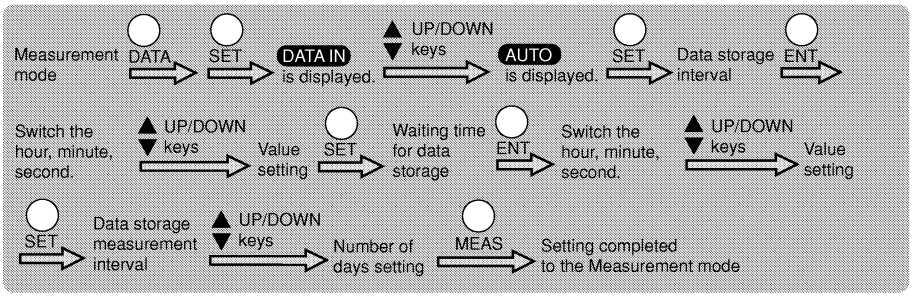
The data memory function can be used to store manualy measurement values with associated data numbers and to store automatically measurement values at fixed intervals (data logger).

4.1 Manual storage of data while monitoring the measurement data 34

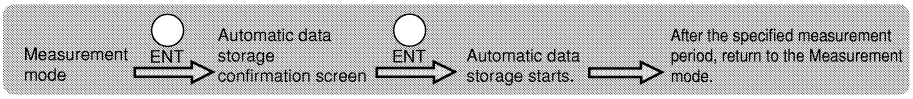


4.2 Automatic data storage 36

4.2.1 Data memory conditions settings 36

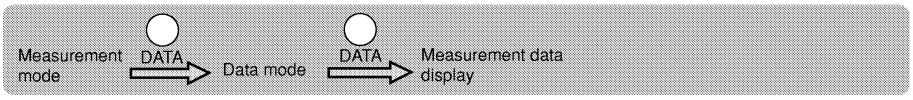


4.2.2 Start of automatic data storage 39

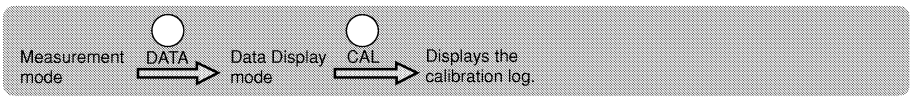


4.3 Calling up data from the memory 41

4.3.1 Calling up measurement data 41



4.3.2 Calling up the calibration log 43

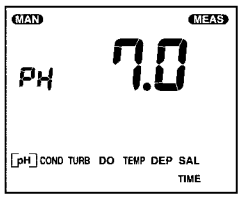


4.1 Manual storage of data while monitoring the measurement data

Make sure MAN is displayed on the measurement screen. → ENT → Start data storage.

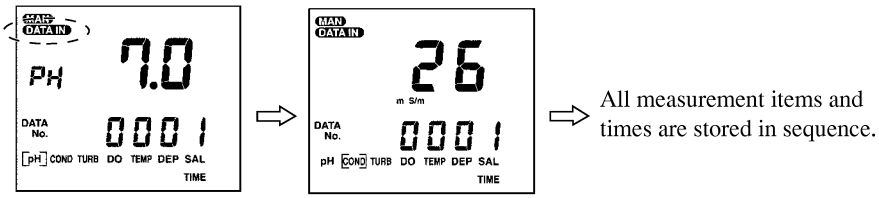
1. Make sure that **MAN** is displayed on the Measurement mode.

If **AUTO** is displayed, switch to **MAN** display.
(☞ page 35, Switch to **MAN** display on the measurement mode)



2. Press the **ENT** key.

Data storage starts, **DATA IN** and the data No. are displayed on the screen, and the measured value to be stored and the measurement item are displayed in order at approximately 0.5 second intervals.

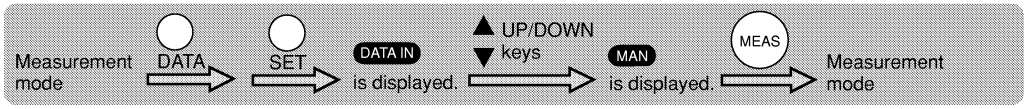


After the data is stored in memory, the screen returns to the original Measurement mode.

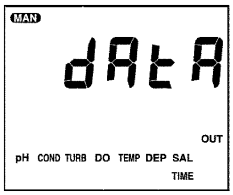
Note

- Up to 2880 sets of data can be stored in the memory.
  When 2880 sets of data have been stored in the memory, ERR 9 appears and no more data can be stored. In this case, "*Data memory clear*" while referring to page 78, and you can store new data in the memory.

When AUTO is displayed
Switch to MAN display on the measurement mode

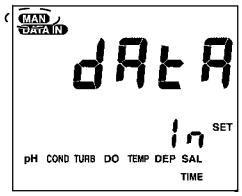


1. Press the **DATA** key in the Measurement mode.



2. Press the **SET** key.
DATA IN is displayed.

3. Press the **UP/DOWN** (▲ ▼) keys to display MAN.

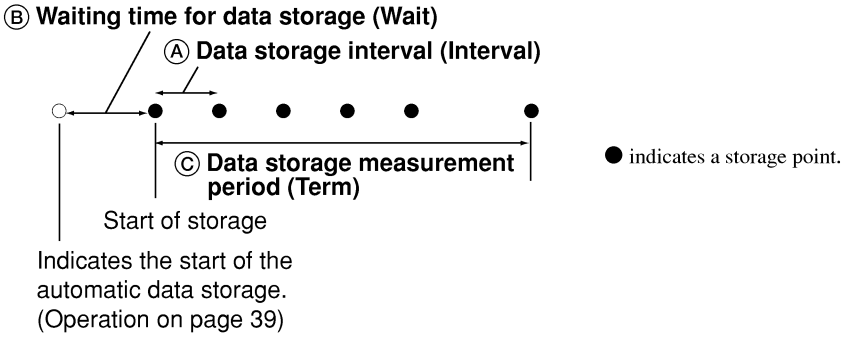


4. Press the **MEAS** key to return to the Measurement mode.

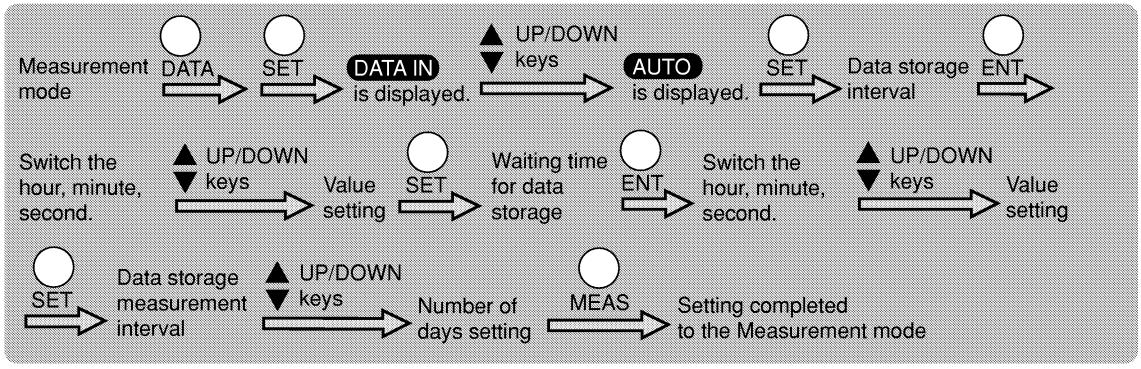
4.2 Automatic data storage

Measured values are stored automatically at constant time intervals. Before using the automatic storage, the following condition settings are required:

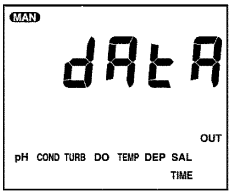
- Setting of data storage interval (4.2.1, step 4)
- Setting of waiting time for data storage (4.2.1, step 6)
- Setting of the data storage measurement period (4.2.1, step 8)



4.2.1 Data memory conditions settings



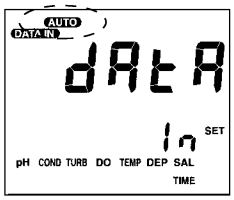
1. Press the **DATA** key in the Measurement mode.



2. Press the **SET** key.

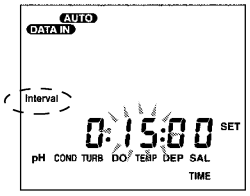
DATA IN is displayed.

3. Press the **UP/DOWN** (▲ ▼) keys to display **AUTO**.



4. Press the **SET** key to display the screen for setting the data storage interval Ⓐ.
"**Interval** " is displayed.

5. Press the **ENT** key to switch the among "hour", "minute" and "second" and set the value using the **UP/DOWN** (▲ ▼) keys.
(Data storage intervals can be set to 2 seconds to 24 hours.)
The current setting location will blink.

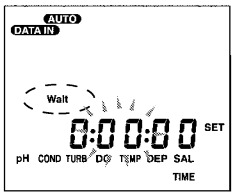


6. Press the **SET** key to display the screen for setting the waiting time for data storage Ⓑ.
"**Wait** " is displayed.

7. Press the **ENT** key to switch among "hour", "minute" and "second" and set the value using the **UP/DOWN** (▲ ▼) keys.
(The waiting time for data storage can be set to 2 seconds to 24 hours.)
The current setting location will blink.

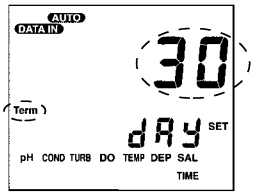
Important

- If wait time is set to "0", note that data is not stored in a memory the first time.



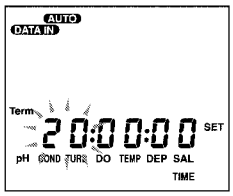
8. Press the **SET** key to display the screen for setting the data storage measurement period Ⓒ (number of days).
"**Term** " is displayed.

9. Use the **UP/DOWN** (▲ ▼) keys to set the value (number of days).



Setting of less than 24 hours

First set the number of days to 00 then press ENT key to select the "hour/minute/second" setting. Use the UP/DOWN (▲ ▼) keys to set the hour, the minute and second. During setting, the number to be set blinks.

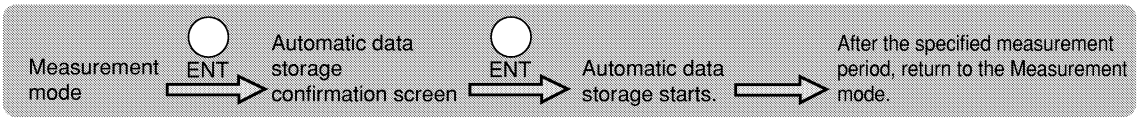


Note

- Press the SET key to return to step 4.

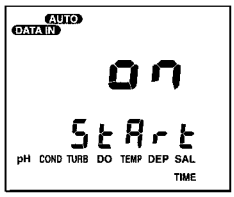
10. When the **MEAS** key is pressed, setting will be completed and the instrument will return to the Measurement mode.

4.2.2 Start of automatic data storage



1. Make sure that **AUTO** is displayed on the Measurement mode.

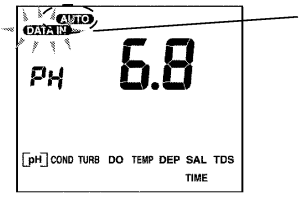
2. Press the **ENT** key. A confirmation screen will be displayed asking if you wish to start automatic data storage.



Note

- If you do not wish to proceed with automatic data storage, press the CAL key to return to the Masurement mode.

3. Press the **ENT** key to start automatic data storage.



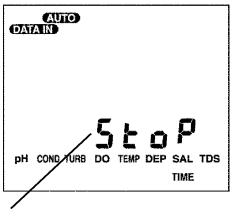
While **DATA IN** is blinking, the automatic data storage is being executed.

Note

- During the automatic data storage, measurement items can be switched by pressing the MEAS key.

Important

- During the automatic data storage, the ENT, SET, and DATA keys do not function and therefore calibration, setting change and stored data display cannot be performed.
- To stop automatic data storage, press the CAL key.



Confirmation display for canceling automatic data storage appears.
To stop the automatic data storage ..... Press the ENT key.
To return to the screen for the automatic data storage ... Press the DATA key.

4. After the specified measurement period, DATA IN disappears and the instrument returns to the normal Measurement mode.

Note

- When the instrument is turned on, AUTO lights up and DATA IN blink if automatic data storage is being performed with the sensor probe.

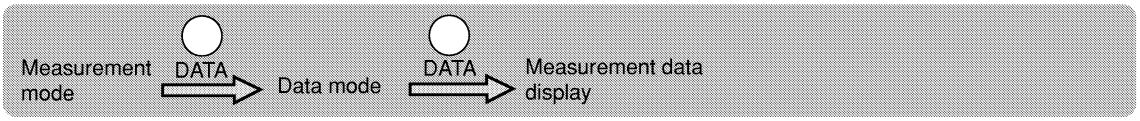
Notes for automatic data storage

- For long-term data storage, replace the sensor probe battery with a new one.
- You can remove the connector from the main unit. It can still be used for up to 60 hours at room temperature with the battery in the sensor probe (alkaline battery). Life is reduced by approximately one half when manganese batteries are used.
- If the sensor probe is connected to the instrument for monitoring, the instrument battery is first consumed to protect the memory of the sensor.
- When 2880 sets of data have been stored in the memory, ERR 9 appears and no more data can be stored. The automatic data storage is automatically ended and the instrument returns to the normal Measurement mode.

4.3 Calling up data from the memory

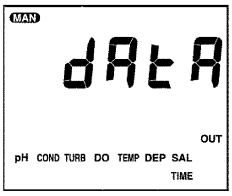
4.3.1 Calling up measurement data

Reading out data that has been stored manually or automatically.



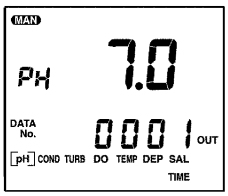
1. Press the **DATA** key in the Measurement mode.

The instrument goes to the DATA mode.



2. Press the **DATA** key.

The measurement data is displayed.
Data you want to call can be displayed by selecting a measurement item and data No.



DATA key ..... Selects switching of measurement item or memory data No.

When switching measurement items: Measurement item blinks.
When switching data No. : Data No. blinks.

UP/DOWN (▲▼) keys .... Switch measurement item or No. which has been selected with the DATA key.

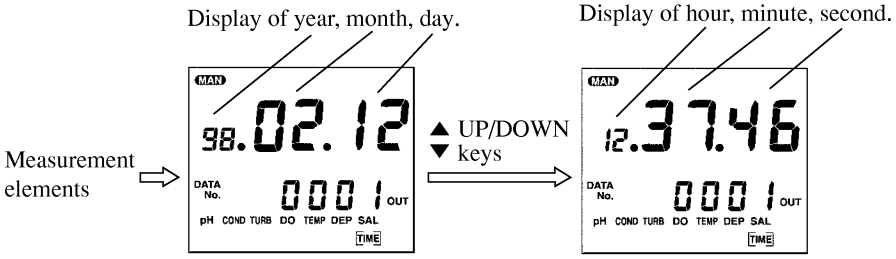
Note

- If you push the CAL key, only the data numbers will be displayed, allowing rapid changing of the numbers. Push the UP/DOWN (▲▼)keys to find the number, then press the SET key to display the data.

3. Press the **DATA** key.

TIME data

Use the UP/DOWN (▲▼) keys to switch between “Yer, Month, Day” and “Hour, Minute, Second”.

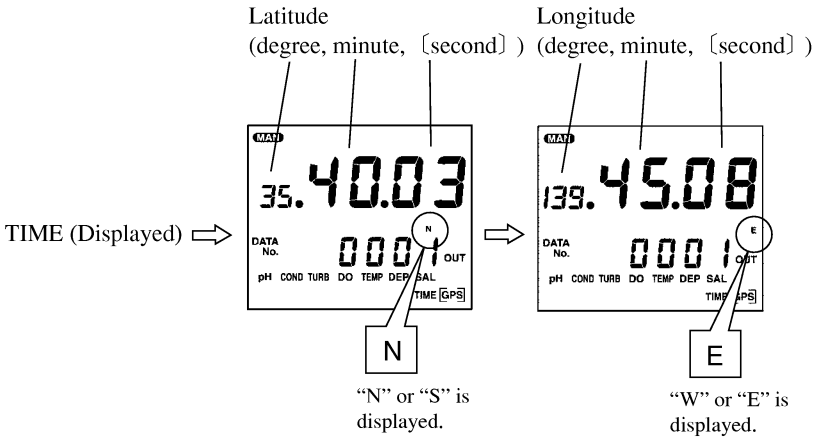


Note

- The time in the automatic memory can be out by about 2 seconds.

G.P.S. data (only when G.P.S. data is present)

Use the UP/DOWN (▲ ▼) keys to switch between “Latitude” and “Longitude”.



Latitude N → The North latitude S → The South latitude
Longitude E → The East longitude W → The West longitude

ENT key ..... Prints all measurement data for the displayed memory data item.
(when the printer is connected to the instrument)

Useful uses of keys in automatic storage

SET + UP (▲) key ..... Displays the first part of the next data automatically stored.
SET + DOWN (▼) key ..... Displays the first part of the previous data automatically stored.
If there is manual data, then the previous or next manual data is shown.

Display for automatic storage

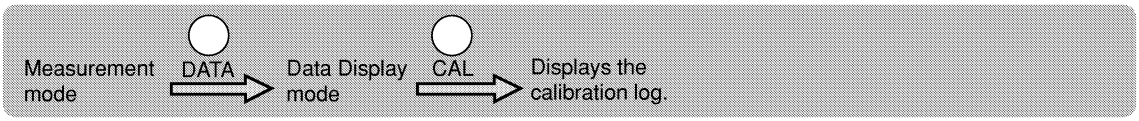
For the first and last data in one session of automatic storage the following identification marks are displayed in front of the values representing the data Nos.:
[: displayed for the first data in automatic storage.
] : displayed for the last data in automatic storage.

Note

- When the MEAS key is pressed, data calling is stopped and the instrument returns to the Measurement mode.
- Data is called from the sensor probe so to get one piece of data takes about one second.

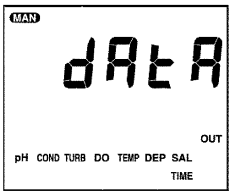
4.3.2 Calling up the calibration log

A calibration log is a record containing the “year, month, day” and “hour and minute” of the last calibration of individual measurement items and their calibration method. The instrument automatically stores the calibration log.



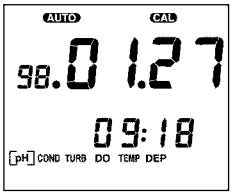
1. Press the **DATA** key in the Measurement mode.

The instrument goes to the DATA Display mode.



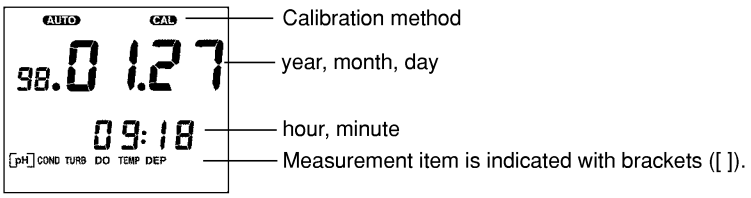
2. Press the **CAL** key.

The calibration log is displayed.



UP/DOWN (▲▼) keys: Switch the measurement item.
ENT key: Prints the entire calibration log. (when the printer is connected to the instrument)

Calibration log.



Calibration method

AUTO CAL	: AUTO calibration
MAN ZERO CAL	: Manual zero calibration
MAN SPAN CAL	: Manual span calibration
MAN ZERO SPAN CAL	: Manual zero calibration and span calibration

Note

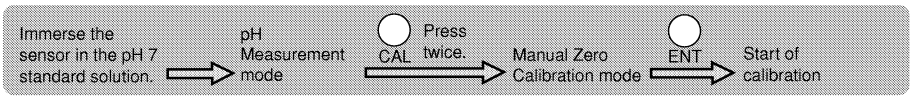
- Press the MEAS key to abort the data calling and return to the Measurement mode.

MEMO

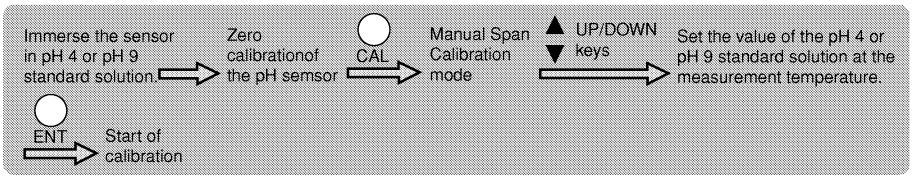
5. Techniques for more accurate measurement

In normal operation, calibration using the AUTO Calibration mode described earlier in the basic operation section provides sufficient accuracy. However, for more accurate measurement, manual calibration is effective. When measurement with high-accuracy extended display is needed, be sure to perform manual calibration.
Attention: The extended display mode is entered automatically when manual calibration is selected.

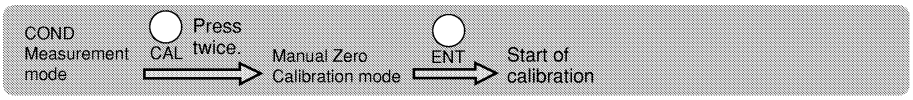
5.1 Manual pH calibration 47
5.1.1 Zero calibration 47



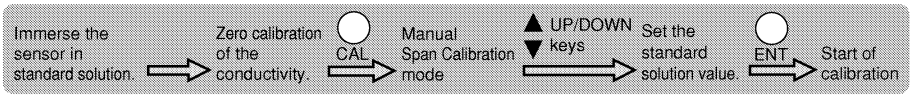
5.1.2 Span calibration 48



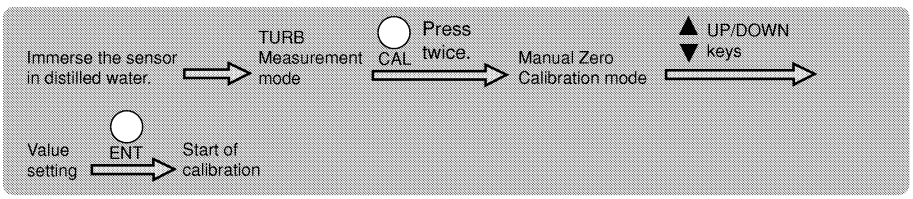
5.2 Manual conductivity (COND) calibration 49
5.2.1 Zero calibration 49



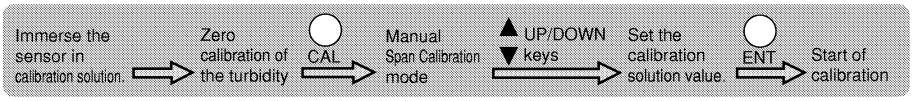
5.2.2 Span calibration 50



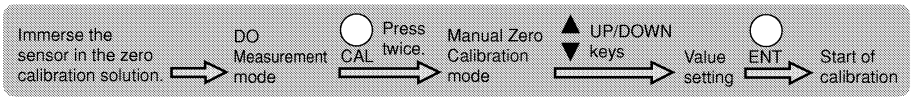
5.3 Manual turbidity (TURB) calibration 52
5.3.1 Zero calibration 52



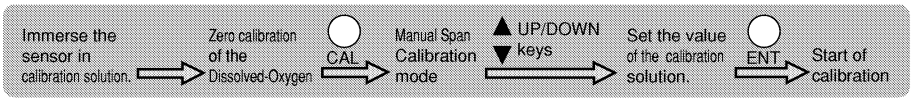
5.3.2 Span calibration 53



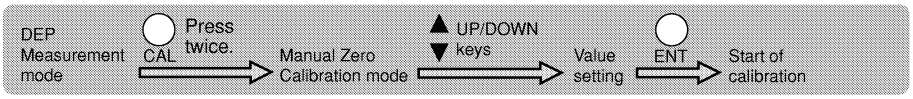
5.4 Manual Dissolved-Oxygen (DO) calibration 54
5.4.1 Zero calibration 54



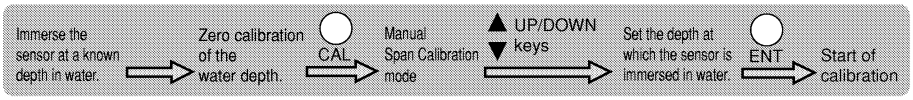
5.4.2 Span calibration 55



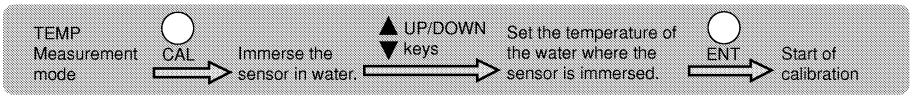
5.5 Water depth (DEP) calibration 57
5.5.1 Zero calibration 57



5.5.2 Span calibration 58



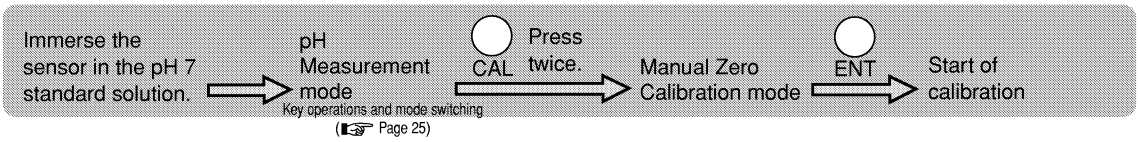
5.6 Temperature (TEMP) calibration 59



5.7 Operation check using ORP standard solution 60

5.1 Manual pH calibration

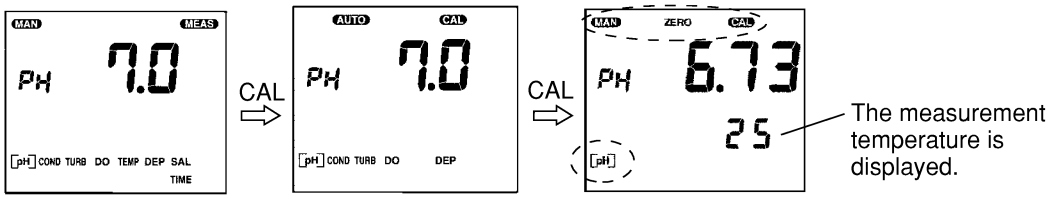
5.1.1 Zero calibration



1. Wash the sensor two or three times using distilled water, then pour some pH 7 standard solution into the calibration beaker, and immerse the sensor in it.

2. Press the **CAL** key twice in the pH Measurement mode.

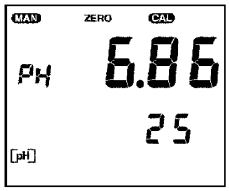
When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.



Manual Zero Calibration mode

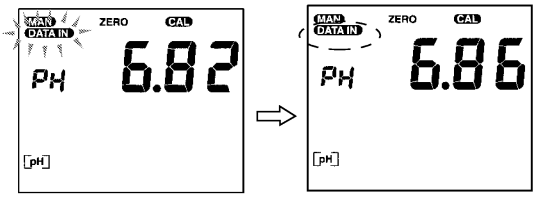
3. Use the **UP**/**DOWN** (▲ ▼) keys to input the value for the pH 7 standard solution at the measurement temperature.

(☞ *8. Reference data*, page 96.)



4. Press the **ENT** key.

The manual zero calibration starts.



End of calibration

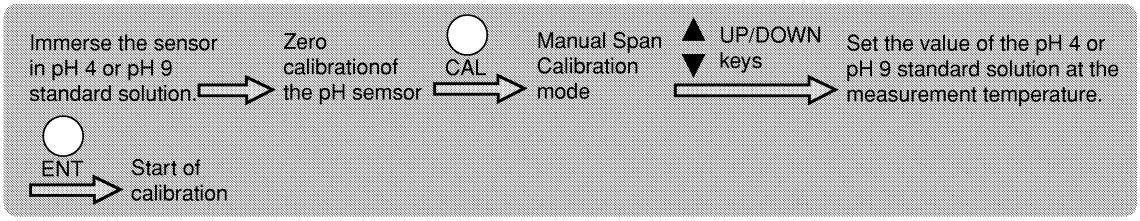
The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

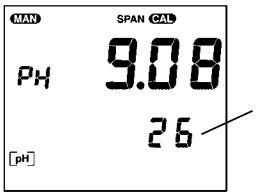
5.1.2 Span calibration



1. Wash the sensor two or three times using distilled water, then pour some pH 4 or pH 9 standard solution into the calibration beaker, and immerse the sensor in it.

2. After the zero calibration of the pH sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

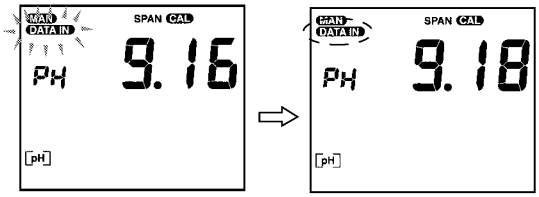
3. Use the **UP/DOWN** (▲ ▼) keys to set the value for the pH 4 or pH 9 standard solution at the measurement temperature.



The measurement temperature is displayed.

4. Press the **ENT** key.

The manual span calibration starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

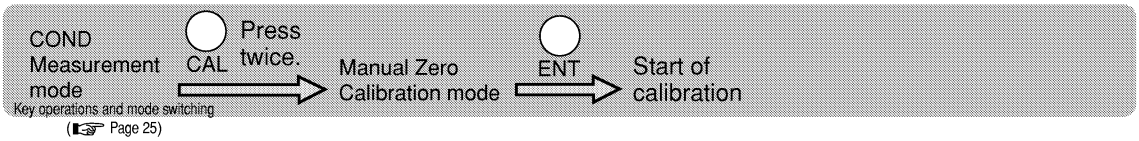
Note

- When the SET and CAL keys are pressed during the manual pH calibration mode, the calibration data for the pH sensor can be deleted.

5.2 Manual conductivity (COND) calibration

The U-20XD series models can measure conductivity (COND) in the range from 0.90 to 9.99 S/m. Depending on the concentration of the sample, these models automatically select the most suitable measuring range from three ranges: 0.0 to 99.9 mS/m, 0.090 to 0.999 S/m, and 0.90 to 9.99 S/m. The zero point is common to the three measuring ranges.

5.2.1 Zero calibration

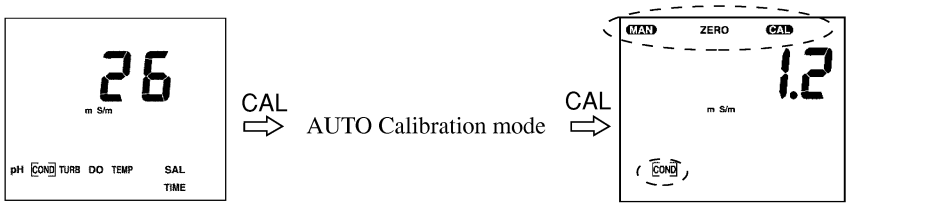


Key operations and mode switching
(☞ Page 25)

1. Wash the conductivity (COND) sensor two or three times using distilled water. Completely remove the water on the sensor and calibrate the instrument in the atmosphere.

2. Press the **CAL** key twice in the Conductivity (COND) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

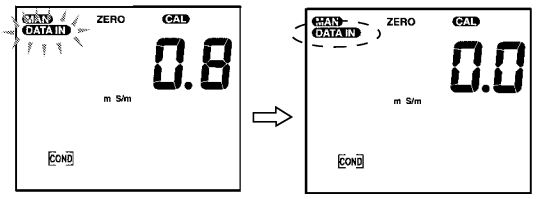


Manual Zero Calibration mode

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5.2.2 Span calibration

Preparation of calibration solution (Potassium chloride (KCl) standard solution)

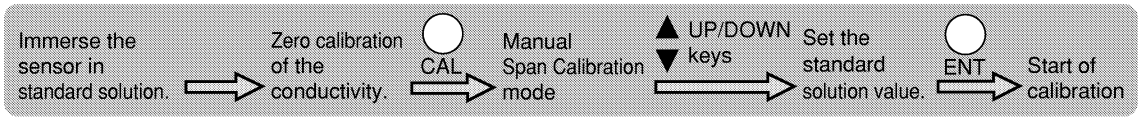
Dry Potassium chloride (KCl) powder (high-grade commercially available) at 105 °C for two hours, and leave it to cool in a desiccator.
Consult the following table and measure a portion of potassium chloride (KC1), then prepare standard potassium chloride (KC1) solution following the procedure below.

Potassium chloride (KCL) standard solution	Conductivity (COND) value	Potassium chloride (KCl) mass (g) at solution temperature of 25 °C	Calibration range
0.005 mol/L	71.8 mS/m	0.373	0.0 to 99.9 mS/m
0.050 mol/L	0.667 S/m	3.73	0.090 to 0.999 S/m
0.500 mol/L	5.87 S/m	37.2	0.90 to 9.99 S/m

1. Dissolve the weighed Potassium Chloride (KCl) in distilled water.
2. Put the dissolved Potassium Chloride (KCl) into a 1 L measuring flask, and fill to the 1 L mark with distilled water.

Calibration procedure

Perform the span calibration using the three types of standard solution as follows.

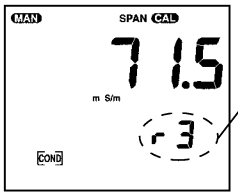


Important

- Set the temperature of the span standard solution to 25 ± 5 °C.
- The sensor should be calibrated in the three standard solutions in the order of increasing concentration.

1. Wash the sensor two or three times using distilled water, then pour some standard solution into the calibration beaker, and immerse the sensor in it.
2. After the zero calibration of the conductivity (COND) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
   MAN, **SPAN** and **CAL** light up.

3. Use the **UP/DOWN** (▲ ▼) keys to set the standard solution value.



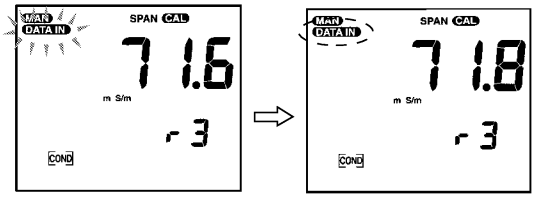
The calibration range is displayed.

Note

- The sensor automatically identifies the calibration solution and the relevant calibration range is displayed.
  - r 1 : 0.90 to 9.99 S/m
  - r 2 : 0.090 to 0.999 S/m
  - r 3 : 0.0 to 99.9 mS/m

4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **CAL** key and use each standard solution and perform steps 1 to 4 above for calibration.

6. Press the **MEAS** key to return to the Measurement mode.

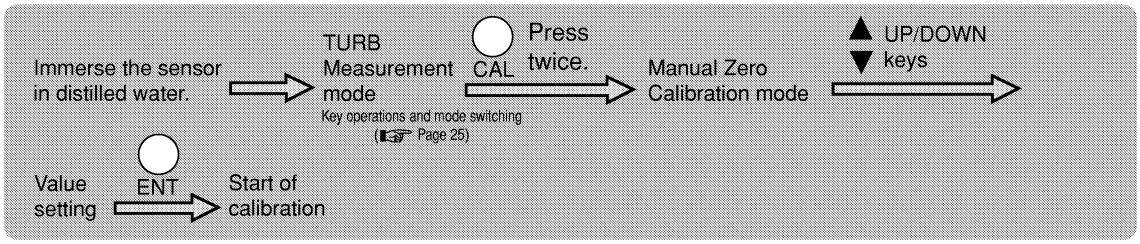
Note

- When the SET and CAL keys are pressed during the manual Conductivity (COND) Calibration mode, the calibration data for the conductivity (COND) sensor can be deleted.
- Perform the calibration again after deleting the present calibration data when calibration error occurs and the calibration cannot be performed.
- Perform the calibration again after deleting the present calibration data when the value cannot be read off because of unsettled digit of the measurement value.

5.3 Manual turbidity (TURB) calibration

5.3.1 Zero calibration

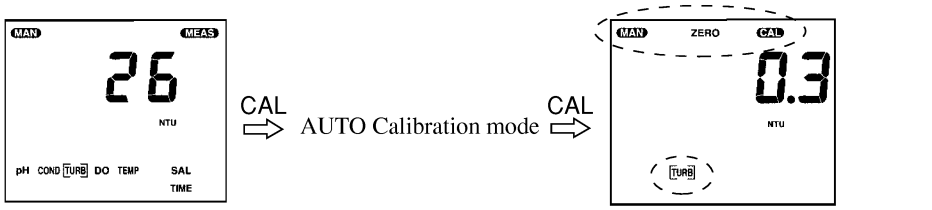
In zero calibration, distilled water is used as a calibration solution. If you cannot obtain distilled water, you may use ion exchange water, which can be considered to have a turbidity of zero.
When the turbidity (TURB) sensor is calibrated, it is particularly important that the probe is completely contamination-free. Do not use a contaminated probe. Otherwise unreliable calibration will result.



1. Wash the sensor two or three times using distilled water, then place some distilled water into the calibration beaker, and immerse the sensor in it.

2. Press the **CAL** key twice in the Turbidity (TURB) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

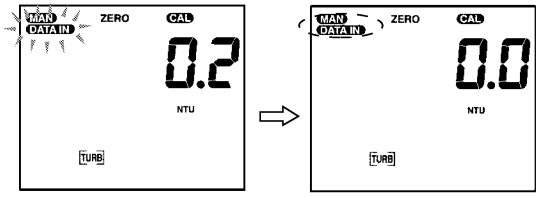


Manual Zero Calibration mode

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is started.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

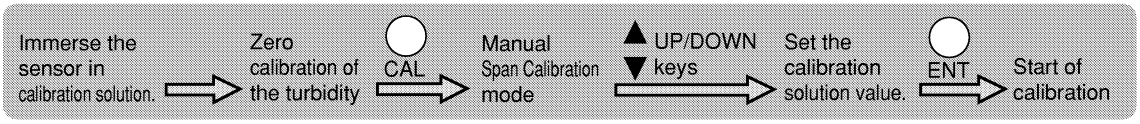
With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5.3.2 Span calibration

Preparation of calibration solution

Weigh out 5.0 g of hydrazine sulfate, and dissolve it in 400 mL of distilled water. Next dissolve 50 g of hexamethylene tetramine in 400 mL of distilled water, and mix the two solutions together. Finally add distilled water until the total solution volume is 1000 mL, and mix well. Store this solution at a temperature of 25 ± 3 °C for 48 hours. The turbidity value (TURB) of this solution is equivalent to 4000 NTU.
Use the solution as span calibration solution for turbidity (TURB) of 800 NTU by diluting this solution by a factor of 5 (use a pipette to measure 50 mL of the 4000 NTU solution and pour it into a 250 mL measuring flask, and add 200 mL of distilled water).

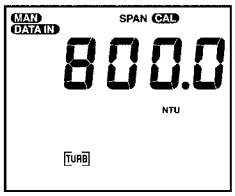
Calibration procedure



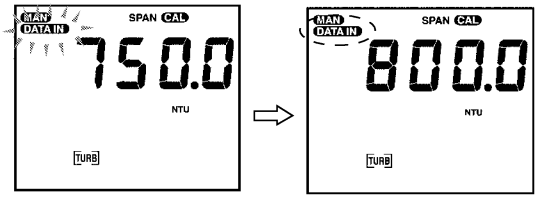
1. Wash the sensor two or three times using distilled water, then pour standard solution into a calibration beaker, and immerse the sensor in it.

2. After the zero calibration of the turbidity (TURB) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 800.0.



4. Press the **ENT** key.
The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Important

- When it is known beforehand that the solution for measurement has a low turbidity (0 to 100 NTU), calibrate the sensor in the span calibration solution of 80 NTU. To prepare an 80 NTU calibration solution, dilute the 4,000 NTU calibration solution with distilled water by a factor of 50.

Note

- When the SET and CAL keys are pressed during the manual Turbidity (TURB) Calibration mode, the calibration data for the turbidity (TURB) sensor can be deleted.

5.4 Manual Dissolved-Oxygen (DO) calibration

It is necessary to prepare a new calibration solution each time directly before calibration of the Dissolved-Oxygen (DO) sensor.

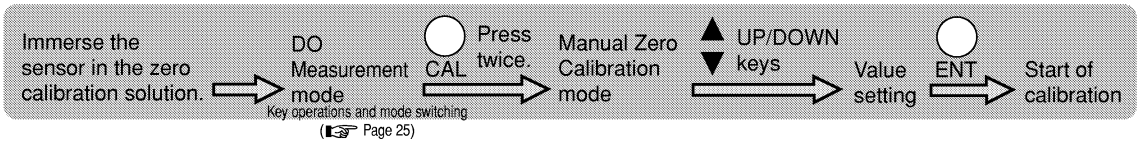
5.4.1 Zero calibration

Use ion exchange water or tap water with sodium sulfite dissolved in it.

Preparation of calibration solution

Add approximately 50 g of sodium sulfite to 1,000 mL of water (either ion exchange water or tap water) and stir the mixture to dissolve the sodium sulfite in it.
The calibration beaker (included) cannot be used to manually calibrate the DO sensor. Use a container that can immerse the DO sensor.

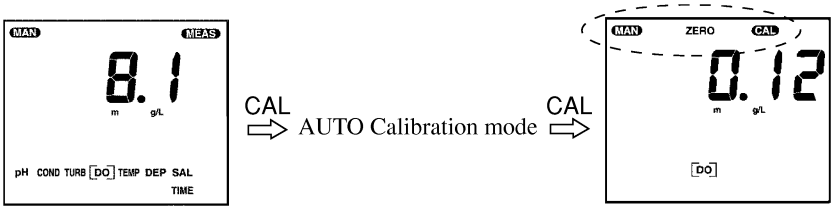
Calibration procedure



1. Wash the sensors 2 to 3 times with pure water and immerse the DO sensor completely in zero calibrated liquid.

2. Press the **CAL** key twice in the Dissolved-Oxygen (DO) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

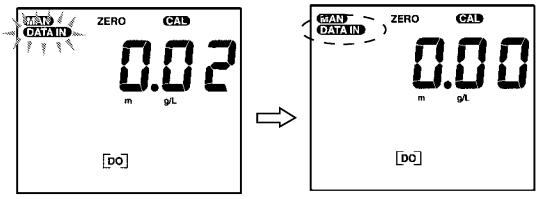


Manual Zero Caliburation mode

3. After the display has stabilized, use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

Important

- After calibration, use tap water to clean the sensor.

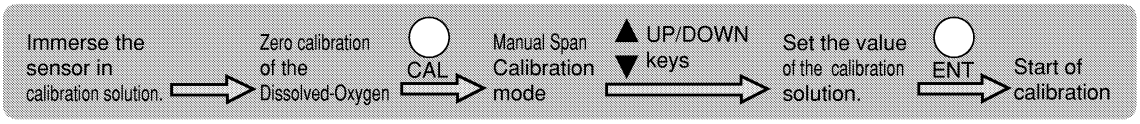
5.4.2 Span calibration

Use ion exchange water or tap water with saturated dissolved oxygen as the span calibration liquid.

Preparation of standard solution for span calibration

Pour 1 to 2 liters of water into a container (either ion exchange water or tap water).
Using a pneumatic pump, feed air into the water and froth up the solution until oxygen is saturated.
The calibration beaker (included) cannot be used to manually calibrate the DO sensor.
Use a container that can immerse the DO sensor.

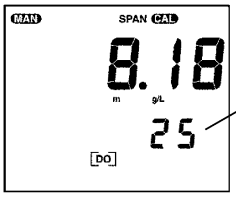
Calibration procedure



1. Wash the sensors 2 to 3 times with pure water and immerse the DO sensor completely in span calibrated liquid.

2. After the zero calibration of the Dissolved-Oxygen (DO) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

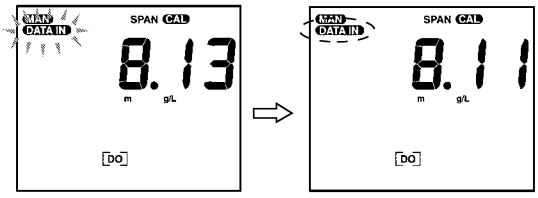
3. After the display has stabilized, use the **UP**/**DOWN** (▲ ▼) keys to set the amount of saturated dissolved oxygen in water at the temperature.



The temperature setting is displayed.
Refer to the table given on page 56 and set a value equivalent to the amount of saturated dissolved oxygen at the temperature.

4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

- When the SET and CAL keys are pressed during the manual Dissolved-Oxygen (DO) calibration mode, the calibration data for the dissolved-oxygen (DO) sensor can be deleted.

Amounts of saturated dissolved oxygen in water at various temperatures (salinity=0.0%)

JIS K0101

Temp. (˚C)	DO (mg/L)	Temp. (˚C)	DO (mg/L)	Temp. (˚C)	DO (mg/L)	Temp. (˚C)	DO (mg/L)
0	14.16						
1	13.77	11	10.67	21	8.68	31	7.42
2	13.40	12	10.43	22	8.53	32	7.32
3	13.04	13	10.20	23	8.39	33	7.22
4	12.70	14	9.97	24	8.25	34	7.13
5	12.37	15	9.76	25	8.11	35	7.04
6	12.06	16	9.56	26	7.99	36	6.94
7	11.75	17	9.37	27	7.87	37	6.86
8	11.47	18	9.18	28	7.75	38	6.76
9	11.19	19	9.01	29	7.64	39	6.68
10	10.92	20	8.84	30	7.53	40	6.59

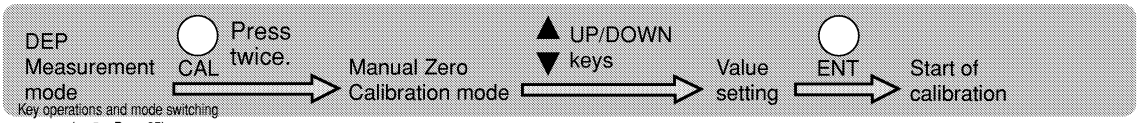
ISO5814

Temp. (˚C)	DO (mg/L)	Temp. (˚C)	DO (mg/L)	Temp. (˚C)	DO (mg/L)
0	14.62				
1	14.22	11	11.03	21	8.91
2	13.83	12	10.78	22	8.74
3	13.46	13	10.54	23	8.58
4	13.11	14	10.31	24	8.42
5	12.77	15	10.08	25	8.26
6	12.45	16	9.87	26	8.11
7	12.45	17	9.66	27	7.97
8	11.84	18	9.47	28	7.83
9	11.56	19	9.28	29	7.69
10	11.29	20	9.09	30	7.56

AUTO calibration is based on the JIS tables. When you need the measured data based on ISO, calibration should be done according to the procedure of span calibration.

5.5 Water depth (DEP) calibration

5.5.1 Zero calibration

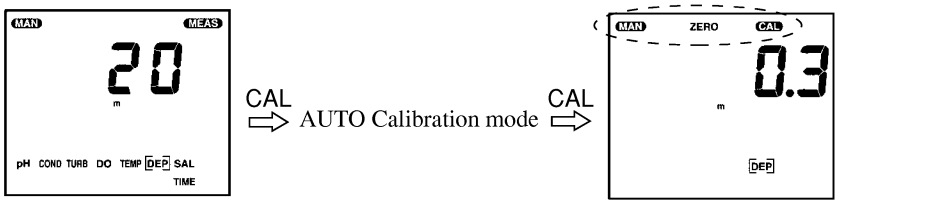


Key operations and mode switching (☞ Page 25)

1. Immerse the sensor in the sample water for approximately 30 minutes so that sensor probe and sample temperatures become the same.

2. Press the **CAL** key twice in the Water Depth (DEP) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.



Manual Zero Calibration mode

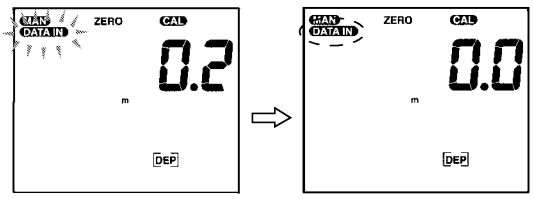
Important

- Sensor probe should be immersed to the depth where the battery cover comes level with the surface. And the level is uses as 0 m in depth.

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

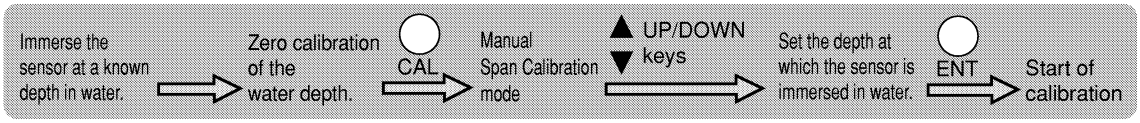
To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

Important

- Since the water depth (DEP) sensor depends greatly on temperature, calibrate the sensor at the same temperature as the sample for more accurate measurement.
- Use the AUTO Calibration mode because calibration error becomes large when using in a place with flow velocity or where it is shallow.

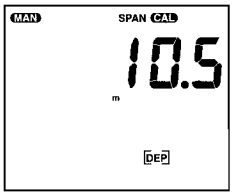
5.5.2 Span calibration



1. Immerse the sensor at a known depth in water. (Set the depth of the lid for memory backup battery as the depth setting.)

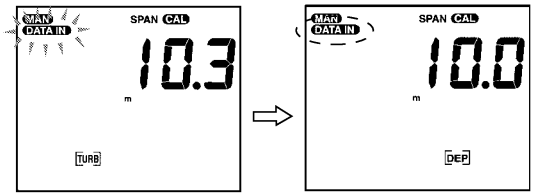
2. After the zero calibration of the water depth (DEP) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

3. Use the **UP/DOWN** (▲ ▼) keys to set the depth at which the sensor is immersed in water.



4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

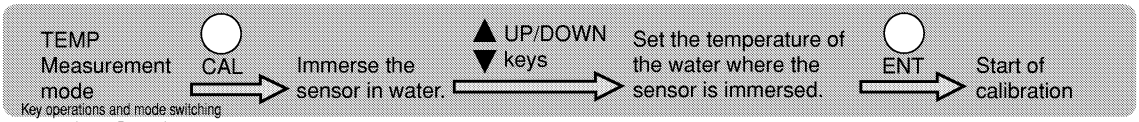
With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

- When the SET and CAL keys are pressed during the manual Water depth (DEP) Calibration mode, the calibration data for the water depth (DEP) sensor can be deleted.

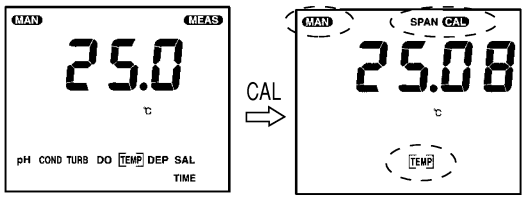
5.6 Temperature (TEMP) calibration



Key operations and mode switching
(☞ Page 25)

1. Press the **CAL** key in the Temperature (TEMP) Measurement mode.

Select the manual calibration mode.

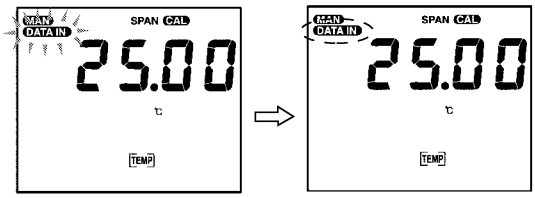


2. Immerse the sensor in water at a known temperature.

3. Use the **UP/DOWN** (▲ ▼) keys to set the temperature of the water where the sensor is immersed as a calibration value.

4. Press the **ENT** key.

The manual calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

- When the SET and CAL keys are pressed during the manual Temperature (TEMP) calibration mode, the calibration data for the temperature (TEMP) sensor can be deleted.

5.7 Operation check using ORP standard solution

Note

- Standard solution is not used only for calibration of the meter, but to confirm whether or not the condition of electrodes is good.

1. Add 250 mL pure (ion exchange) water to one packet of any of the below listed standard solutions and mix well.
   When mixing, the excess quinhydrone (a black powder) will float to the surface of the solution.
2. Immerse a washed and dried ORP electrode in the ORP standard solution and measure the mV value.
3. If the electrode and the meter, itself, are working correctly, numerical values within 15 mV or less of those listed in Table 1 should be obtained.
4. If measurements that fall within 15 mV of the values listed above are not obtained using this method, measure the solution again after replacing the reference electrode internal solution and removing the dirt from the surface of the metal electrode by moistening a cotton swab with alcohol or a neutral cleaning agent and lightly rubbing the electrode or by soaking the electrode in diluted nitric acid (1:1 nitric acid).
5. If measurements within 15 mV of the values listed above are still not obtained after re-measuring, the reference electrode or the meter may be faulty. Either replace the electrode or have the meter inspected.

Important

- If the prepared ORP standard solution is allowed to stand in open air for one hour or more, it may undergo transformation. For this reasons ORP standard solution that has finished being prepared cannot be stored.
- When measuring a solution that has low concentrations of oxidants and reductants after conducting an operational check using a standard substance, the measured values may not stabilize or the results of measurement might not be repeatable.
  If this is the case, use the meter after immersing the electrodes in the solution again and mixing it thoroughly.

Precautions when measuring actual samples

- Note that when measuring the ORP of solution that has extremely low concentrations of oxidants and reductants, such as tap water, well water, or water treated with purifying equipment, there may be less responsiveness, repeatability, and stability, in general.
- When alkaline water is allowed to stand, its ORP undergoes big changes. Always measure alkaline ion water promptly.

ORP standard solution

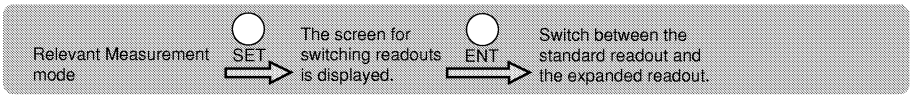
There are two kinds of standards substances. Under normal circumstances, it is sufficient to use only the one type of substance that is closest to the measured value.

Indicated value of ORP standard solution at various temperatures

Standard solution ℃	160-22 Phthalic-acid chloride + quinhydrone	160-51 Neutral phosphate + quinhydrone
5	+274.2	+111.9
10	+270.9	+106.9
15	+266.8	+101.0
20	+262.5	+95.0
25	+257.6	+89.0
30	+253.5	+82.7
35	+248.6	+76.2
40	+243.6	+69.0

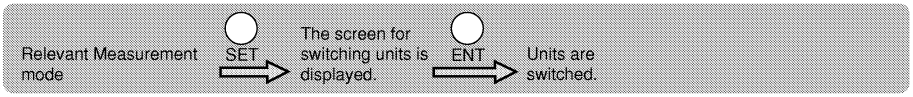
6. Using the various functions

6.1 Switching to Expanded readout (High-accuracy display) 63

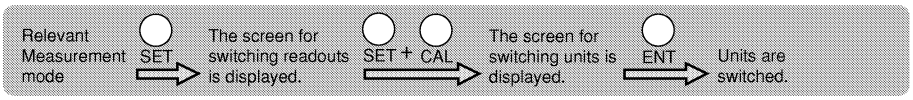


6.2 Switching measurement units 64

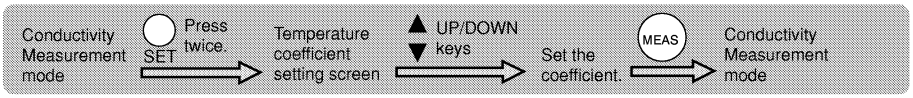
In the case of pH, ION, TURB, DO 64



In the case of COND and DEP 65

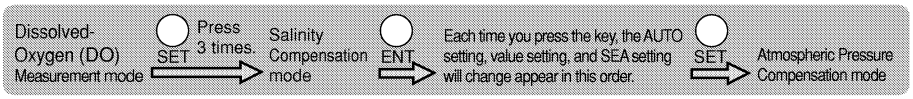


6.3 Temperature conversion for conductivity (COND) 67

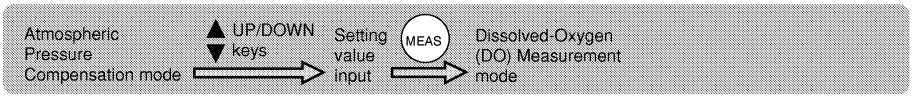


6.4 Dissolved-Oxygen (DO) environmental influence compensation 68

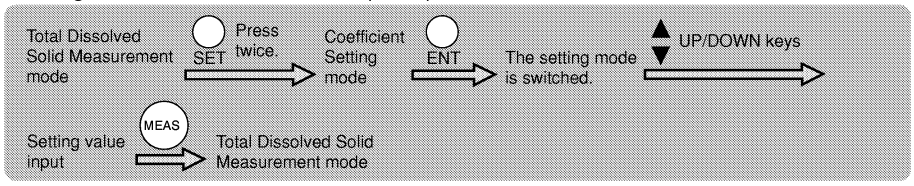
6.4.1 Salinity compensation 68



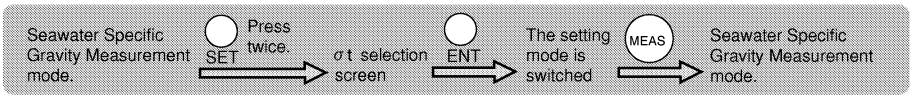
6.4.2 Atmospheric pressure compensation 69



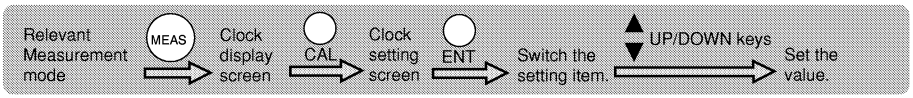
6.5 Setting a total dissolved solid (TDS) coefficient 70



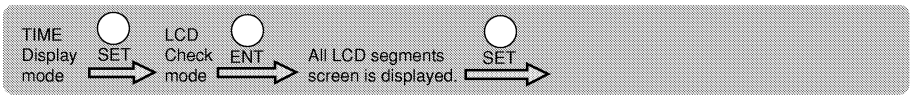
6.6 Displaying seawater specific gravity (σ_0, σ_{15}) 71



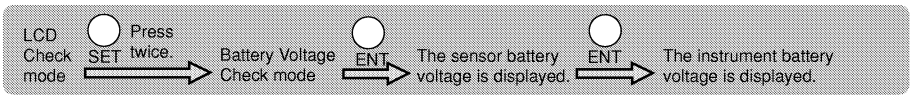
6.7 Setting the clock 72



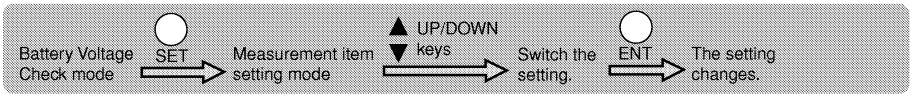
6.8 Key lock setting 73
6.9 Check mode 73
6.9.1 LCD check 74



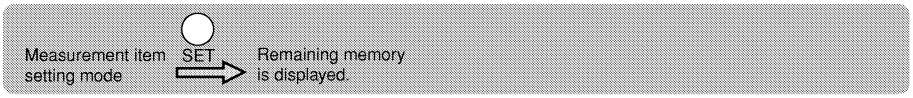
6.9.2 Battery voltage check 75



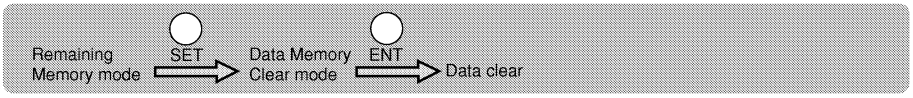
6.9.3 Measurement item setting 76



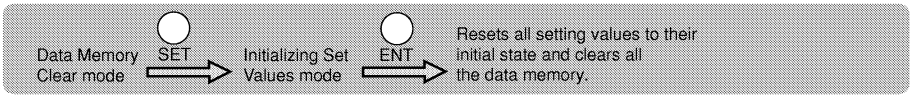
6.9.4 Remaining memory 77



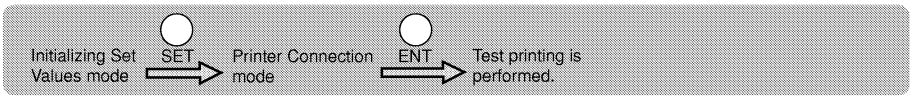
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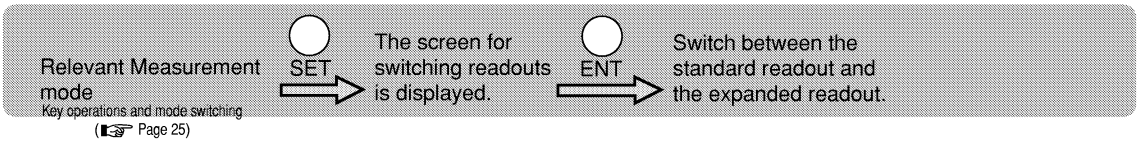


6.9.7 Printer connection, test print 81



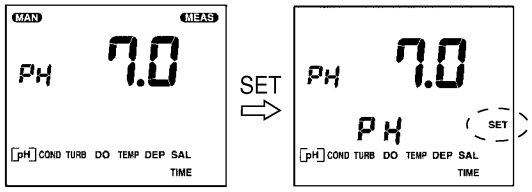
6.1 Switching to Expanded readout (High-accuracy display)

With the exception of oxidation-reduction potential (ORP), it is possible to switch between the Standard readout and the Expanded readout for the measurement value.



1. Press the **SET** key in the relevant Measurement mode.

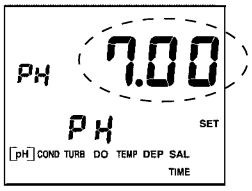
The screen for switching readouts is displayed.



Standard readout

2. Press the **ENT** key.

The screen can be switched between the standard readout and the expanded readout (High-accuracy display).



Expanded readout (High-accuracy display)

Note

- Switch readouts for each measurement items.
- Use the manual 2-point calibration (zero and span) when high accuracy is required for expanded readout (High-accuracy display).
- The expanded readout mode is automatically activated when the manual 2-point calibration mode is chosen.

3. Press the **MEAS** key to return to the Measurement mode.

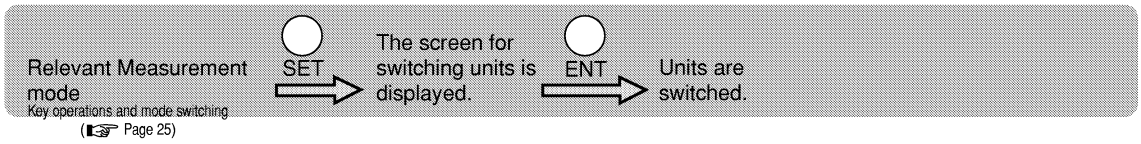
6.2 Switching measurement units

It is possible to switch between measurement units.

The units which can be switched are as follows:

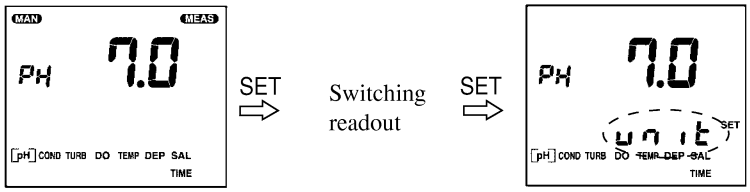
pH ..... pH or mV
Conductivity (COND) ..... S/m or mS/cm
Water depth (DEP) ..... m or ft
Turbidity (TURB) ..... NTU or mg/L
Dissolved Oxygen (DO) ..... mg/L or % (Oxygen saturation ratio)

In the case of pH, TURB, DO



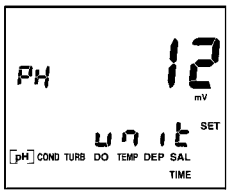
1. Press the **SET** key twice in the relevant Measurement mode.

Confirm that unit is displayed on the screen for switching units.



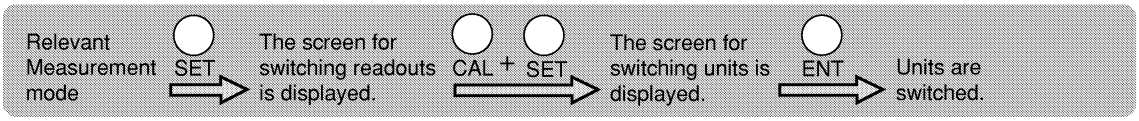
2. Press the **ENT** key.

Units are switched.



3. Press the **MEAS** key to return to the Measurement mode.

In the case of COND and DEP



1. Press the **SET** key in the Relevant Measurement mode.

The screen for switching readout is displayed.

2. Press the **SET** key while holding down the **CAL** key.

Confirm that **unit** is displayed on the screen for switching units.

3. Press the **ENT** key.

Units are switched.

4. Press the **MEAS** key to return to the Measurement mode.

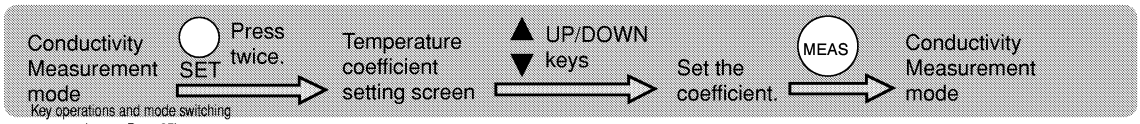
Measurement range

Measurement item		Measurement range		Measurement units
		Expanded	Standard	
pH		0.00 to 14.00	0.0 to 14.0	pH
		—	−1999 to 1999	mV in pH measurement
Conductivity (COND)	Range 1	0.90 to 9.99	0.9 to 9.9	S/m
		9.0 to 99.9	9 to 99	mS/cm
	Range 2	0.090 to 0.999	0.09 to 0.99	S/m
		0.90 to 9.99	0.9 to 9.9	mS/cm
	Range 3	0.0 to 99.9	0 to 99	mS/m
		0.000 to 0.999	0.00 to 0.99	mS/cm
Turbidity (TURB) *1		0.0 to 800.0	0 to 800	NTU (nephelometric turbidity units) or mg/L
Dissolved-oxygen (DO)		0.00 to 19.99	0.0 to 19.9	mg/L
		0.0 to 199.9	0 to 199	%
Temperature (TEMP)		0.00 to 55.00	0.0 to 55.0	°C
Water depth (DEP)		0.0 to 100.0	0 to 100	m
		0.0 to 330.0	0 to 330	ft
Salinity (SAL)		0.00 to 4.00	0.0 to 4.0	%
Total dissolved solids	Range 1	5.5 to 65.0	5 to 65	g/L
(TDS) *2	Range 2	0.55 to 6.50	0.5 to 6.5	g/L
	Range 3	0.000 to 0.650	0.00 to 0.65	g/L
Seawater specific gravity (σ_t)		0.0 to 50.0	0 to 50	–
Oxygen-reduction potential (ORP)		—	−1999 to 1999	mV

*1: Depending on the concentration range, the minimum turbidity is displayed as follows:
0 to 100 NTU ... 1 NTU for standard readout, 0.1 NTU for expanded readout.
100 to 800 NTU ... 10 NTU for standard readout, 1 NTU for expanded readout.
*2: The TDS range depends on the TDS factor settings. (Above ranges are given for a TDS coefficient of 0.65.)

6.3 Temperature conversion for conductivity (COND)

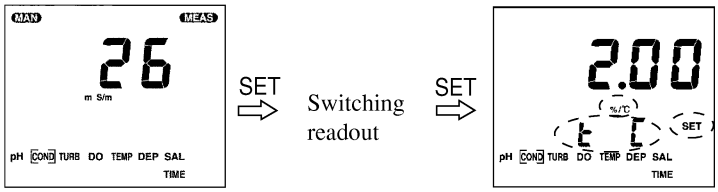
Sample conductivity (COND) varies with temperature, and this instrument uses a temperature conversion coefficient to automatically standardize the conductivity (COND) to the value at 25 °C. The initial setting value is 2 %/°C, which is the generally used value.



Key operations and mode switching (☞ Page 25)

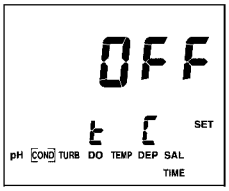
1. Press the **SET** key twice in the Conductivity (COND) Measurement mode.

The screen for setting temperature coefficients is displayed.



2. Use the **UP/DOWN** (▲ ▼) keys to set the coefficient.

The setting range is 0.00 to 3.00 %/°C.



With the ENT key, the temperature conversion is switched between ON and OFF.
The temperature conversion OFF mode is not a 25°C coefficient but a coefficient at the temperature of the sample.

3. Press the **MEAS** key.

The instrument returns to the Conductivity (COND) Measurement mode.

Note

- For temperature coefficients, refer to *Reference data*, page 98 to 99.

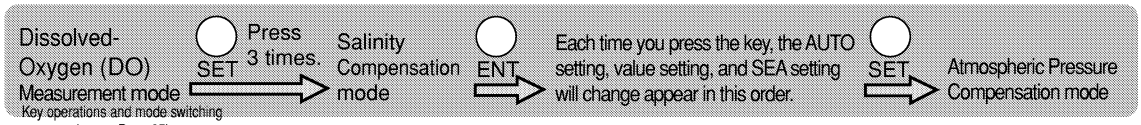
6.4 Dissolved-Oxygen (DO) environmental influence compensation

6.4.1 Salinity compensation

The indicated dissolved oxygen (DO) value can go over the actual value if salinity compensation isn't added because of the increase in salinity in the sample. To obtain a correct measured value for dissolved oxygen (DO) in the sample containing salinity, therefore, salinity compensation is needed. The following modes are available for calculation of salinity compensation.

AUTO..... Salinity compensation is performed automatically with salinity converted from a measured value for conductivity.

SEA ..... Compensation value appropriate for normal seawater is used.



Key operations and mode switching (☞ Page 25)

1. Press the **SET** key 3 times in the Dissolved-Oxygen (DO) Measurement mode.

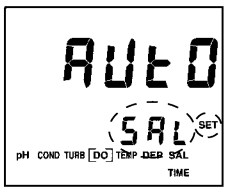
The salinity compensation mode currently set is displayed.

Important

- If you do not change the salinity compensation mode currently set, press the MEAS key to return to the Dissolved-Oxygen (DO) Measurement mode or press the SET key to select the Pressure Compensation mode.

2. Press the **ENT** key.

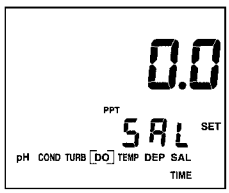
The following screens are displayed in sequence each time the ENT key is pressed: AUTO setting, value setting, SEA setting and AUTO setting.



3. From the screen on which the value is displayed, use the **UP/DOWN** (▲ ▼) keys to enter the setting value if the salinity is known.

For AUTO and SEA setting, this step need not be performed.

The setting range is 0.0 to 40.0 ppt (parts per thousand).

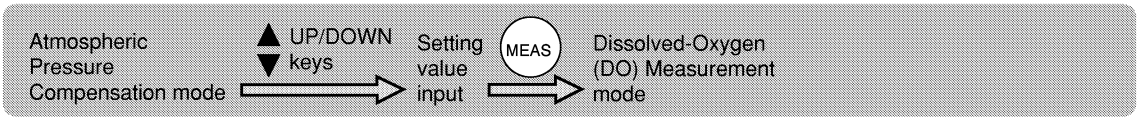


4. When the **SET** key is pressed, setting will be completed and the instrument will enter the Pressure Compensation mode.

5. Press the **MEAS** key to return to the Dissolved-Oxygen (DO) Measurement mode.

6.4.2 Atmospheric pressure compensation

Differences in the atmospheric pressure of the measurement location influence the Dissolved-Oxygen (DO) measurement. By setting (input) the actual atmospheric pressure of the measurement location into the instrument, it is possible to standardize the measured Dissolved-Oxygen (DO) value to a value at the standard atmospheric pressure (1013 hPa).



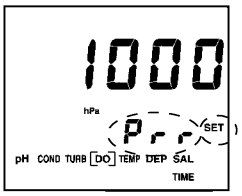
1. When the **SET** key is pressed on the salinity compensation screen, setting will be completed and the instrument will enter the Pressure Compensation mode.

Important

- If you do not change the Pressure Compensation mode currently set, press the MEAS key to enter the Dissolved-Oxygen (DO) Measurement mode.

2. Use the **UP/DOWN** (▲ ▼) keys to input a setting value.

The setting range is 100 to 1999 hPa.



3. When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Dissolved-Oxygen (DO) Measurement mode.

Relation between height (m) and atmospheric pressure (hPa)

Height (m)	0	200	400	600	800	1000	1200	1400	1600	1800	2000	3000	3400
Pressure (hPa)	1013	990	966	943	921	899	877	856	835	815	795	701	666

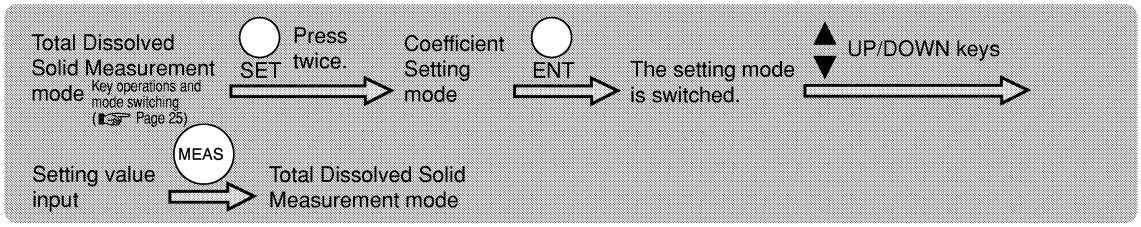
6.5 Setting a total dissolved solid (TDS) coefficient

The total dissolved solid amount (TDS) is a converted value obtained by multiplying the conductivity (COND) value by a known coefficient. Based on a conversion for KCl and $CaCO_3$ solutions, the coefficient initially set for the instrument depends on the conductivity (COND) value as shown below.

Conductivity (COND) (S/m)	Conversion coefficient
< 0.05	0.65
0.05 to 0.5	0.64
0.5 to 1	0.63
1 to 3	0.62
3 to 5	0.61
> 5	0.60

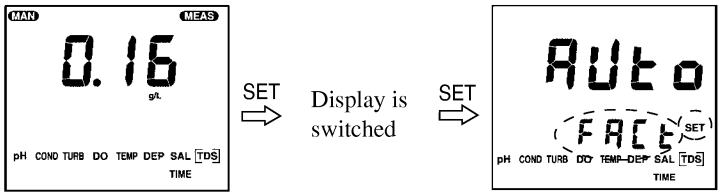
AUTO ..... Used to automatically calculate the total dissolved solid (TDS) amount with an initially set coefficient.

Setting value input ..... Used to determine the total dissolved solid (TDS) amount by setting any conversion coefficient irrespective of the conductivity (COND) value.



1. Press the **SET** key twice in the Total Dissolved Solid (TDS) Measurement mode.

The Coefficient Setting mode currently set is displayed.

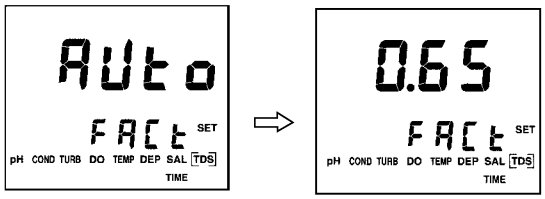


Important

- If you do not change the coefficient currently set, press the MEAS key to enter the Total Dissolved Solid (TDS) Measurement mode.

2. Press the **ENT** key.

The setting mode changes (AUTO/setting value input).



3. Use the **UP/DOWN** (▲ ▼) keys to input a setting value if required.

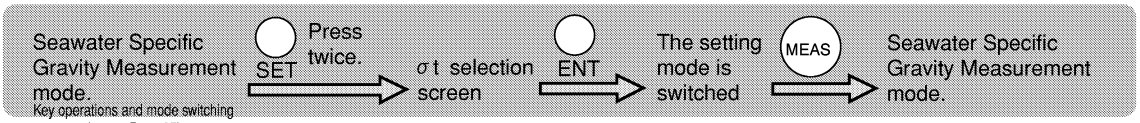
The setting range is 0.50 to 1.00.

4. When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Total Dissolved Solid (TDS) Measurement mode.

6.6 Displaying seawater specific gravity (σ_0, σ_{15})

The specific gravity of seawater varies with temperature. By converting the measured value based on the value for a reference temperature, it is possible to compare sample measurement values at different temperatures.

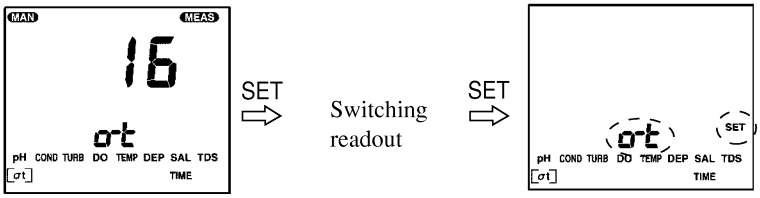
σ_t ..... Specific gravity of seawater at the measurement temperature.
σ_0 ..... Seawater specific gravity at 0 °C.
σ_{15} ..... Seawater specific gravity at 15 °C.



Key operations and mode switching
(☞ Page 25)

1. Press the **SET** key twice in the Seawater Specific Gravity (σ_t) Measurement mode.

Seawater specific gravity (σ_t) selection screen is displayed.

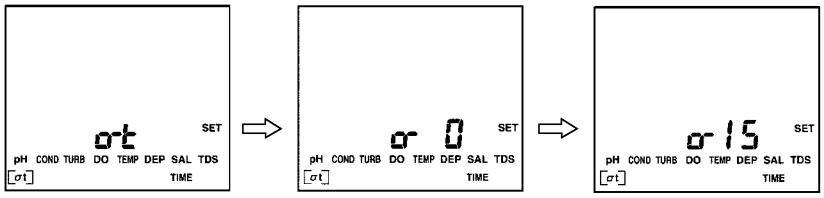


Important

- If you do not change the specific gravity currently set, press the MEAS key to enter the Seawater Specific gravity (σ_t) Measurement mode.

2. Press the **ENT** key.

The setting mode is switched.
($\sigma_0 \rightarrow \sigma_{15} \rightarrow \sigma_t \rightarrow \sigma_0 \ldots$)

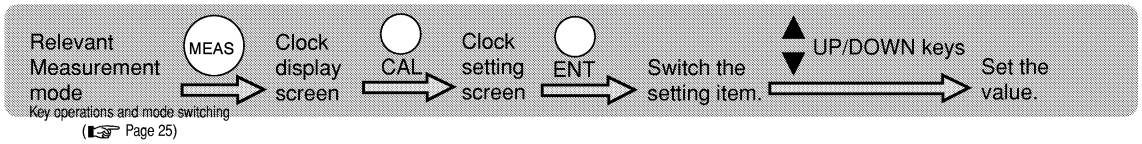


3. When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Seawater Specific Gravity (σ_t) Measurement mode.

Note

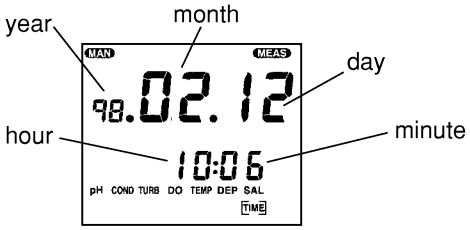
- See page 100 for more about seawater specific gravity.

6.7 Setting the clock



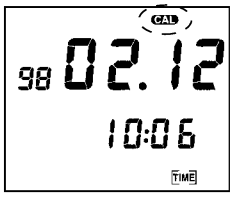
Key operations and mode switching (☞ Page 25)

1. Use the **MEAS** key in the measurement mode to switch to the clock display screen.



2. Press the **CAL** key.

CAL light up and clock setting screen is displayed.



3. Press the **ENT** key to switch the measuring item.

(year → month → day → hour → minute → year ...). The setting item will blink.



4. Use the **UP/DOWN** (▲ ▼) keys to set the value.

5. Press **SET** key to confirm the setting.

Note

- When the MEAS key is pressed, the instrument will return to the clock display.

Important

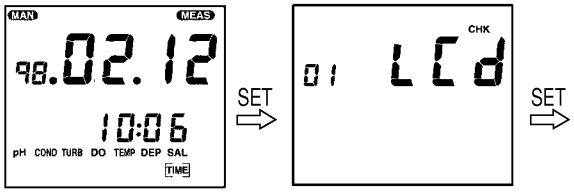
- When the MEAS key is pressed without pressing the SET key and the clock display is displayed again, settings are not changed.

6.8 Key lock setting

If you press the POWER key while pressing the UP (▲) key when the power is off, the instrument is then turned ON with the key locked and the key lock function works.
With the key locked, only the POWER and MEAS keys can be used and [LOCK] is displayed on the screen. To release this function, turn the instrument OFF first and then ON again.

6.9 Check mode

When the SET key is pressed in the measurement mode from the screen where "year, month, day and time" are displayed, the instrument performs self-diagnosis check.



Each time the SET key is pressed, the check mode item is switched sequentially.

Check mode items

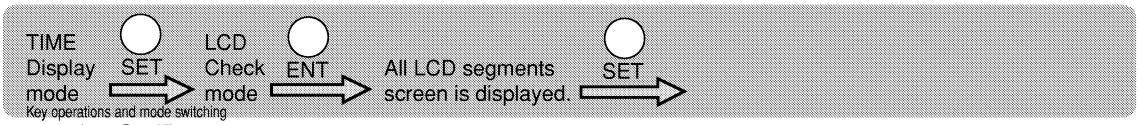
1 :	LCD check	Checks if all LCD segments will be displayed. (☞ Page 74)
2 :	Battery voltage check	Performs a simple battery voltage check for the instrument and sensors. (☞ Page 75)
3 :	Measurement item setting	Can set the measurement item to be stored. (☞ Page 76)
4 :	Remaining memory	Displays the number of data that can be stored now. (☞ Page 77)
5 :	Data memory clear	Clears the data memory. (☞ Page 78)
6 :	Initializing set values	Initializes all memory settings. (☞ Page 79)
7 :	Printer connection, test print	Performs a test print. (☞ Page 80)

Note

- In the check mode, it is possible to return to the Measurement mode by pressing the MEAS key.

6.9.1 LCD check

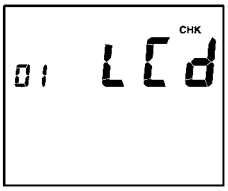
All LCD segments are displayed.



Key operations and mode switching
(☞ Page 25)

1. Press the **SET** key in the Clock Display mode.

LCD check mode screen is displayed.

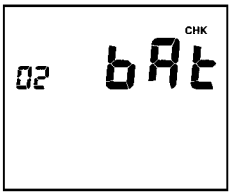


2. Press the **ENT** key.

3. Check to see if all LCD segments are displayed.



4. When the **SET** key is pressed, the instrument goes to the battery voltage check.

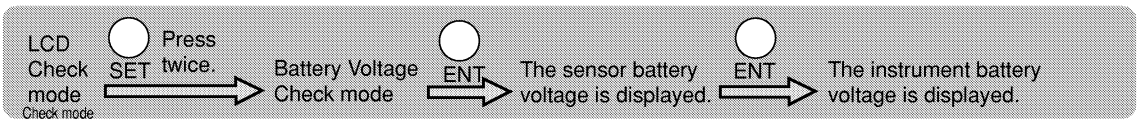


Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.2 Battery voltage check

The battery voltage in use is displayed.



Check mode
(☞ Page 73)

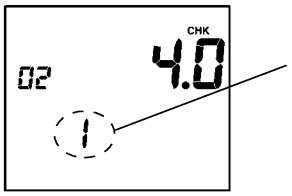
1. Press the **SET** key twice in the LCD Check mode.

Battery Voltage Check mode screen is displayed.



2. Press the **ENT** key.

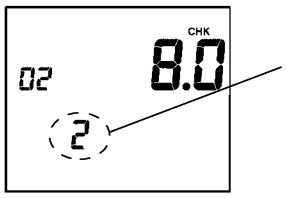
The sensor battery voltage is displayed.



1: Sensor battery voltage
Criteria
3.0 to 5.0 V : Normal
Less than 3.0 V: Replace the battery.
(Error No. 2 will blink.)

3. Press the **ENT** key.

The instrument battery voltage is displayed.



2: Instrument battery voltage
Criteria
5.5 to 11.0 V : Normal
Less than 5.5 V : Replace the battery.
(Error No.3 will blink.)

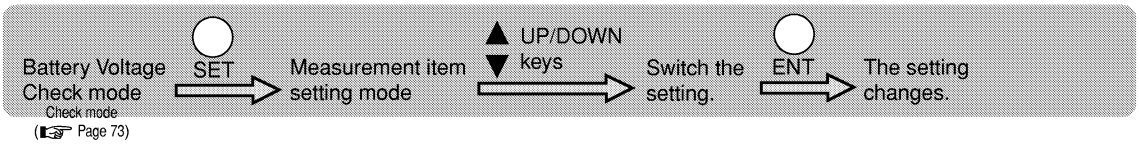
4. When the **SET** key is pressed, the instrument goes to the measurement item setting.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.3 Measurement item setting

Measuring items can be set.



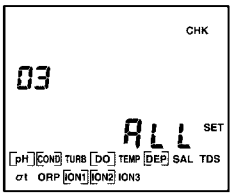
Check mode
(☞ Page 73)

1. Press the **SET** key in the Battery Voltage Check mode.

Display setting mode screen is displayed.

2. Use the **UP/DOWN** (▲ ▼) keys to switch the measurement item.

The selected item blinks.



3. Press the **ENT** key to switch between [set/ not set] for the blinking item.

An item for which "set" is selected is indicated with [ ].

Note

- If the temperature is "not set" data for each component is not temperature-compensated and is displayed as data at 25 °C.

4. When the **SET** key is pressed, the instrument goes to the remaining memory display.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.4 Remaining memory

The number of date that can be stored can be displayed.



Press the **SET** key in the Display Setting mode.

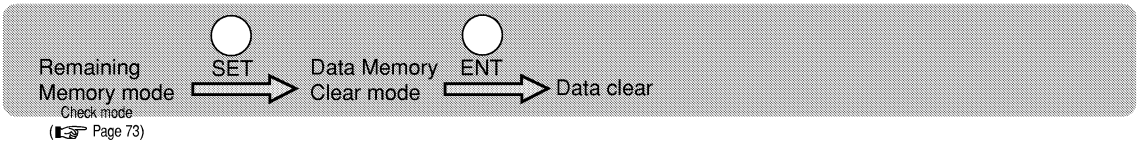
Remaining memory is displayed.

Note

- When the **SET** key is pressed, the instrument goes to the Data Memory Clear mode.
- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.5 Data memory clear

All the data memory is cleared.

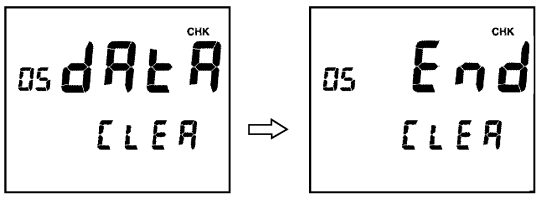


1. Press the **SET** key in the Remaining Memory mode.

Data memory clear mode screen is displayed.

2. Press the **ENT** key.

The data is cleared.



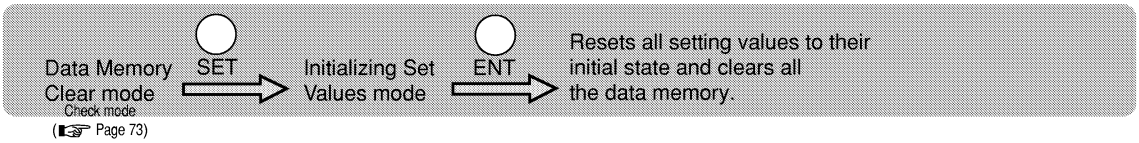
3. When the **SET** key is pressed, the instrument goes to the Memory Initialization mode.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.6 Initializing set values

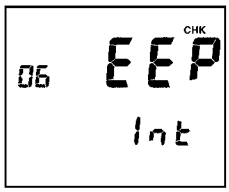
All setting values are reset to their initial state.



Check mode (☞ Page 73)

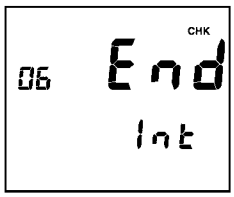
1. Press the **SET** key in the Data Memory Clear mode.

Initializing Set Values mode screen is displayed.



2. Press the **ENT** key.

All setting values are reset to their initial state.



3. When the **SET** key is pressed, the instrument goes to the Printer Connection mode.

Note

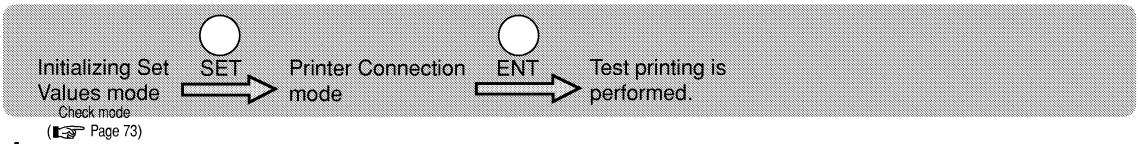
- When the MEAS key is pressed, the instrument return to the Clock Display mode.
- Data stored in the memory remains.

Initial setting

Item	Description	Initial value
Common	Display setting	Standard
	Data storage	Manual
pH	Unit	pH
COND	Unit	S/m
	Temperature coefficient	2.0 %/°C
DO	Salinity setting	AUTO
	Atmospheric pressure setting	1013 hPa
	Unit	mg/L
TURB	Unit	NTU
DEP	Unit	m
TDS	Coefficient	AUTO
σ_t	Unit	σ_t

6.9.7 Printer connection, test print

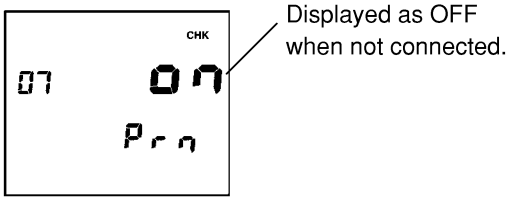
This mode only operates when the function expansion unit is connected. A test print is performed if a printer is connected.



Check mode (☞ Page 73)

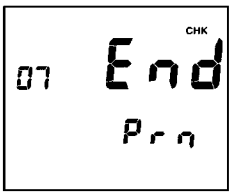
1. Press the **SET** key in the Initializing Set Values mode.

Printer Connection mode screen is displayed.



2. Press the **ENT** key to start printing.

Normally, "End" is displayed. If an error has occurred, "Err" is displayed.



3. When the **SET** key is pressed, the instrument will return to the first LCD check mode.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

7. Instrument specifications

7.1 Daily maintenance 82
7.2 Troubleshooting 85
7.3 Specifications 90
7.4 Spare parts 92
7.5 Option 94

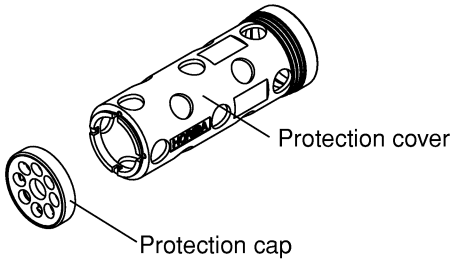
7.1 Daily maintenance

Sensor probe

● Storage method

After use, wash out with tap water and wipe off all contamination. Pour about 20 mL of pure water into the probe cap, install it on the sensor probe, and store in the carrying case.
In order to use the instrument regularly for a long time, store it after wiping off all contamination from the cable, sensor probe, and sensors.

Remove the protection cover once and completely wash out with tap water the left over sample on the screws. Reinstall the cover after having wiped off the drops of water. If there is any sample (especially sea water) left over on the screws, rust may form which may prevent the protection cover from being removed. (☞ Installation procedure, page 18.)
Depending on the level of contamination, remove the rubber protection cap from the tip of the protection cover and wash out with tap water. Reinstall it after wiping off the drops of water.

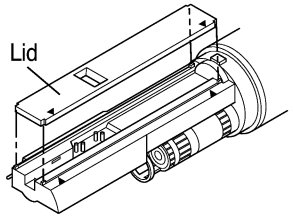


When storing with the pH/ORP and DO sensors attached to the probe, make sure to install the probe cap after having poured pure water into it.
Letting the pH/ORP and DO sensors get dry may cause deterioration of the instrument's performance. Should the sponge inside the probe cap be contaminated, replace it with a clean sponge (included).

TEMP/COND/TURB units

● To remove contamination

1. Remove the lid from the cell.
2. Clean the unit in tap water. If the unit is severely contaminated, use an absorbent cotton to remove contamination.
3. Attach the lid to the cell block before storage. (☞ page 29)



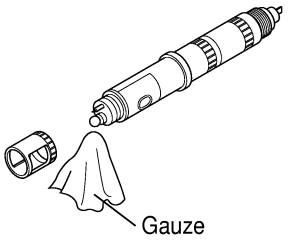
Important

- The cell has a window for turbidity measurement. Be careful to avoid damage to the window.
  In case of measurements, attach the lid to the cell in the correct direction.
- Don't remove the COND/TURB lid during calibration or measurement.
- Attach the lid to the cell with fitting four corners and facing ▲ marks each other.

pH/ORP sensors

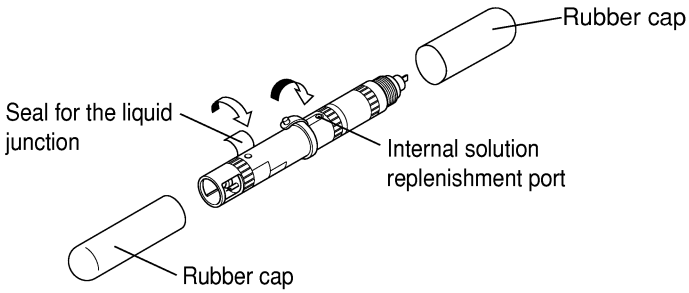
● To remove contamination

Use a piece of gauze dampened with detergent and wipe off contamination.



● Long-term storage

Remove the sensor from the sensor probe and check the internal solution replenishment port is closed. Then, attach a seal to the liquid junction and attach the rubber caps before storage.



● Monthly maintenance

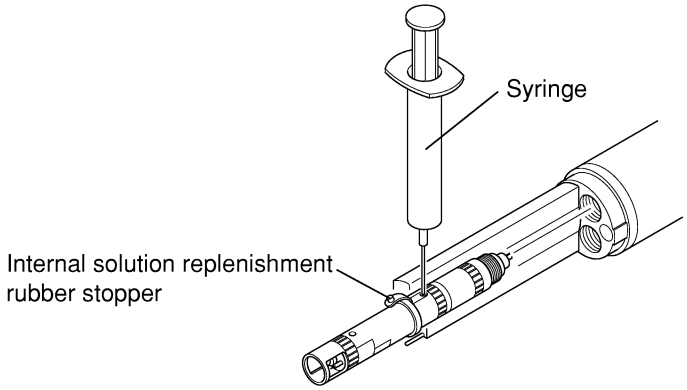
Replace the internal solution as described below:

1. Remove the sensor from the sensor probe using a sensor spanner.
2. Open the internal solution replenishment rubber stopper and remove the internal solution with a syringe.
3. Pour new internal solution (#330) to the level near rubber stopper. Be careful to avoid air bubbles from coming in the solition.
   Air bubbles in the internal solution will impair the sensors' pressure compensation function.

Important

- Shake the sensor to avoid bubbles in the internal solution from remaining at the bottom of the sensor.

4. Attach the sensor to the sensor probe.



DO sensor

● To remove contamination

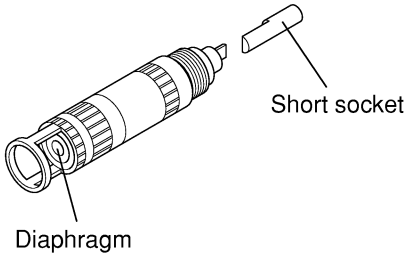
Wipe off contamination with gauze to avoid damage to the diaphragm.

● Long-term storage

Remove the sensor from the sensor probe using a sensor spanner. Set the supplied short socket and store the sensor in a cool(0 to 10℃), dark place.

Important

- Provide the DO sensor with a short socket or connect the sensor to the sensor probe for storage. Otherwise, the sensor may have a shorter life or stable instructions may not be obtained.
- The short socket is used when storing. Do not throw it away.



● Resetting the DO sensor when storing without having installed the short socket.

When leaving the DO sensor unattended for a brief period (1 or 2 days) without the short socket, the DO sensor can be reset by connecting it to the short socket or the probe. However, an amount of time corresponding to the period it was left unattended is necessary. If left unattended without being connected to the short socket or the probe for a long period (1 month), it cannot be reset.

● To replace the diaphragm.

Please read the instruction manual of the DO diaphragm replacement kit. (☞ page 89)

7.2 Troubleshooting

The instrument has a simple error message that informs users of operational errors and failure. Err No. is displayed at the bottom of the screen.

● Error message list

Err No.	Designation	Err No.	Designation
1	Sensor memory failure	6	Span calibration error
2	Sensor battery voltage drop	7	Calibration stability error
3	Instrument battery voltage drop	8	Printer error
4	Communications error	9	DATA IN error
5	Zero calibration error		

● Error and remedy

Important

- For err Nos. 5 to 7, the calibration err display disappears when a proper calibration is performed after the following action, or when the instrument is turned on again. For the other err Nos., the err display disappears after any of the following actions is taken.
- Error Nos. 2 and 3 are displayed even when using the AC adapter if the sensor probe battery voltage or instrument battery voltage drops is low on voltage.

Err NO.	Problem	Cause	Remedy
1	No data can be read from or written into the sensor probe memory.	Internal IC failure	Call your nearest store for sensor probe repair.
2	Sensor probe battery voltage drop	① Battery voltage drop ② Improper installation of the battery	① Replace the sensor probe battery. ② Set the batteries (LR03) in the correct direction.
3	Instrument battery voltage drop	① Battery voltage drop ② Improper installation of the battery	① Replace the instrument battery. ② Set the battery (6LR61) in the correct direction.
4	No communications possible between the instrument and the sensor probe	① Improper connection of the connector to the instrument ② Cable disconnection	① Connect the connector to the instrument properly and turn on the instrument again. ② Call your nearest store for cable repair.
5	No zero calibration possible	pH • The standard solution is contaminated. • Contamination on the pH glass membrane • Change in concentration of the internal solution for the reference electrode • Cracks in the pH glass electrode COND • The standard solution is contaminated. • The sensor is dirty. • The COND sensor is broken.	pH • Change the standard solution. • Clean the pH glass membrane. • Replace the internal solution for the reference electrode. • Replace the sensor. COND • Change the standard solution. • Clean the sensor. • Contact your nearest store.

Err NO.	Problem	Cause	Remedy
5	Zero calibration not possible	TURB	TURB
		• Air bubbles in the cell	• Swing the sensor probe while drawing a large arc.
		• Cell contamination	• Clean the cell.
		DO	DO
		• Damage to the diaphragm of the DO sensor	• Check the sensor and replace it if damaged.
		DEP	DEP
		• Contamination on the DEP sensor	• Clean the DEP sensor.
		• Damage to the DEP sensor	• Contact your nearest store.
6	Span calibration not possible	pH	pH
		• Contamination on the pH glass membrane	• Clean the pH glass membrane.
		• Change in concentration of the internal solution for the reference electrode	• Replace the internal solution for the reference electrode.
		• Cracks in the pH glass electrode	• Replace the sensor.
		• Damage to the connector pin	• Replace the sensor.
		COND	COND
		• The standard solution isn't correct.	• Calibrate with correct standard solution.
		• The standard solution value is set uncorrectly.	• Delete the calibration data for the conductivity, then calibrate the sensor again. (☞ Page 50)
		• The COND sensor is broken.	• Contact your nearest store.
		TURB	TURB
		• Air bubbles in the cell	• Swing the sensor probe while drawing a large arc.
		• Cell contamination	• Clean the cell.
		• The lid is attached uncorrectly.	• Confirm if the lid is attached correctly, then calibrate the sensor again. (☞ Page 29)
		DO	DO
		• Damage to DO sensor diaphragm	• Check the DO sensor and replace it if damaged.
		• DO sensor is unstable.	• Connect DO sensor to the sensor probe. Calibrate the sensor again 1 day later.
		• Damage to the connector pin	• Replace the sensor.
		DEP	DEP
		• Contamination on the DEP sensor	• Clean the DEP sensor.
		• Damage to the DEP sensor	• Contact your nearest store.
		TEMP	
		• Damage to the TEMP sensor	• Contact your nearest store.
7	The calibration value does not become stable within approximately three minutes.	① Sensor contamination	① Clean each sensor.
		② Dry sensor surface	② Pour the standard solution into the calibration beaker. Calibrate the sensor again 1 to 2 hours later.
		③ Severe temperature change	③ Calibrate the sensor in a place at a stable temperature or in a thermostatic oven.

Err NO.	Problem	Cause	Remedy
8	Printer unit failure		Turn OFF the instrument and use the remedy described below. Then turn ON the printer again.
		① Paper has jammed in the printer	① Remove the jammed sheet of paper
		② Improper printer unit connection	② Check to see if the printer is properl connected to the instrument.
		③ Printer failure	③ Replace the printer.
			* Contact your nearest store if the instrument does not recover after replacement of the printer.
9	Data cannot be stored because the memory is full.	No free space in the memory	Delete the data stored in the memory. (☞ Page 78)

● Other troubles

Remedies for various trouble with no Err No. displayed are described below.

Problem	Cause	Remedy
No data display with the power on	• No batteries	• Set new batteries.
	• Improper position of the positive and negative poles	• Set the batteries properly while paying attention to the positive and negative poles.
	• Battery voltage drop	• Replace the batteries with new ones.
	• Improper instrument battery contact	• Use radio pliers to narrow the positive terminal of the battery snap.
No setting change possible	• Automatic data storage is under way	• Press the CAL key to stop the automatic data storage.
No key operation possible	• The key lock function is working	• Turn OFF the instrument. Then turn ON the instrument again. (☞ Page 73)
	• Failure to calibrate the sensor or wrong calibration.	• Calibrate the sensor properly.
Blinking measured value	• Improper measurement sample	• Use a sample that is in the measurement range.
	• Sensor contamination	• Clean each sensor.
	• Poor calibration is possible. (The standard solution is contaminated.)	• Carry out correct calibration.
tYPE Err The Err is displayed and the operation cannot be performed.	• Improper connection of the cable connector to the instrument	• Connect the connector to the instrument properly and turn on the instrument again.
	• Cable disconnection	• Contact your nearest store.
	• Instrument inside failure	• Contact your nearest store.

● Troubleshooting for the TURB sensor

If an abnormal value such as -10, 800 or more is indicated, or indication does not become stable, follow as below instructions.

Remove the contamination of the sensor

Remove the cover of the turbidity (TURB) sensor, and clean the sensor with cotton swab.
Contamination or bubbles on the sensor may cause fluctuation of TURB values.

Remove bubbles around the sensor

When immersing sensor in the calibration cup, be sure lower it slowly. Quick immersion may cause bubbles on the sensor, which can have bad influence on calibration accuracy to give abnormal value indication.

Use of new standard solution

When calibration, clean the sensor before immersing it in the new standard solution. In case of zero calibration, when the standard solution is turbid or contaminated, calibrate again with the new standard one.

Points to be noted in making measurement

Immerse the sensor slowly in the sample. In case of abnormal measurement value observed, contamination or bubbles adhering may be suspected. So, shake greatly the sensor. Since immersion of the sensor in the sludge layer at the bottom of the sample can prohibit accurate measurement, shake greatly enough to remove the sludge.

● Maintenance of DO sensor

Durable life of DO sensor is generally one year, however, it may vary depending on the using condition. In case of the failure of calibration or breakage of the diaphragm, take either of the following steps according to the using period.

Within one year after purchasing :

Obtain diaphragm replacement kit (optional) to replace the used diaphragm and replenish the internal solution for restoration.

When exceeding one year after purchasing :

Replace by the new DO sensor.

● Materials

#5460 DO Sensor Diaphragm Replacement Kit Operation Manual

This operation manual explains how to replace the DO Sensor (#5460) Diaphragm.

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1 This kit comes with the following.

● Diaphragm sheets	10 sheets
● Diaphragm retaining ring	5 pcs.
● O−ring (S9)	5 pcs.
● Internal solution #305 (50 mL)	1 bottle
● Diaphragm retaining plate	1 pc.
● Replacement stand	1 pc.
● Syringe and needle	1 set
● Operation manual	1 sheet

2 Diaphragm Replacement

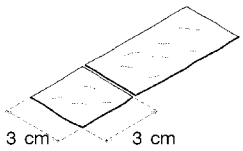
Chemical solution

Caution

The internal solution contains pottasium hydroxide (KOH) solution of high concentration.
If the solution comes in contact with hands or skin, wash immediately with water. If the solution comes in contact with the eyes, flush with ample amounts of water, then seek medical assistance.

1 Cut a diaphragm sheet to about 3 x 3 cm in size.

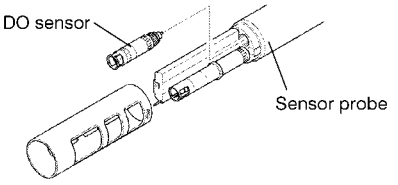
Note▶▶▶ Don't get any fingerprints or dust on the center part of the square.



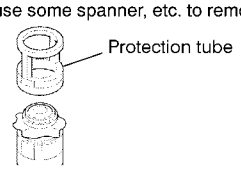
2 Detach the DO sensor from the sensor probe.

Ref▶▶▶ Refer to the U−21.22.23 Operation Manual, section 2.3.2 "Sensor Installation".

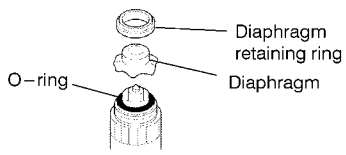
Note▶▶▶ If the short socket is not attached, instability may be seen when the sensor is used again.
Wear vinyl work gloves when handling the sensor. The KOH inside it is a strong alkaline and can irritate skin.



3 Detach the protection tube that holds the diaphragm in place. If it is difficult to remove, use some spanner, etc. to remove it.

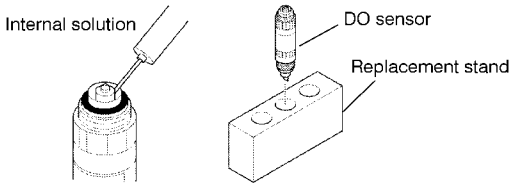


4 Detach the diaphragm retaining ring and diaphragm. Detach the diaphragm retaining ring and diaphragm if damaged or no longer functional.

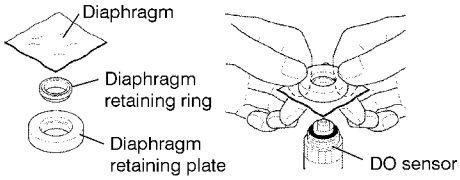


5 Set the sensor in the replacement stand ,and fill the internal solution with the attached syringe until the sensor tip is soaked with the solution.

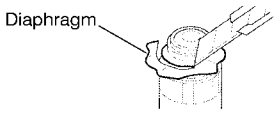
Note▶▶▶ Use the internal solution included in the kit for best performance.
In case some white crystals are seen, wipe them out completely with a Kimwipe®, etc.
In case the internal solution includes white crystals, remove the internal solution and fill up again. If the white crystals cannot be seen, just fill up.



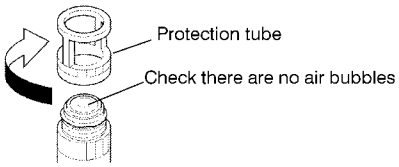
6 Fit the diaphragm retaining ring into the diaphragm retaining plate. Then, lay the diaphragm over the sensor and carefully cover with the ring and plate so that the diaphragm does not wrinkle. Finally, remove the retaining plate.



7 Cut the draped edge of the diaphragm to the shape of the sensor.

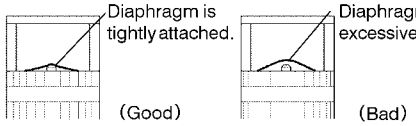


8 Check there are no air bubbles inside the sensor and tighten the protection tube securely.



9 Check the diaphragm bulges upward as shown on the below left and that it is not wrinkled.

Note▶▶▶ Check that span calibration can be made correctly. Sensitivity lowers and response speed slows if the diaphragm does not bulge sufficiently.



For any question regarding this product, please contact your local agency, or inquire from the Customer Registration website (www.horiba.co.jp/register).

HORIBA, Ltd.

7.3 Specifications

NOTE ○: Applicable
—: Unapplicable

		U-22XD
Instrument	Water-proof construction	IP67
	Mass	Approximately 500 g (including the grip holder)
Sensor *1	Use in 2-inch well	○
	Measurement temperature	0 to 55 °C
	Storage temperature	-5 to 60 °C
	Measurement depth	to 100 m
	Maximum sensor outside diameter	47 mm
	Sensor length	380 mm
	Continuous use available *2	30 days
	Automatic data gathering at set time	○
	Mass (Cable10 m)	Approximately 1.9 kg
pH ● Two-point calibration ● Automatic temperature compensation	Measuring principle	Glass electrode method
	Range	pH0 to 14
	Resolution	0.01 pH
	Repeatability	±0.05 pH
	Accuracy	±0.1 pH
Dissolved-Oxygen ● Salinity conversion (0 to 40 ppt/Auto) ● Automatic temperature compensation	Measuring principle	Diaphragm galvanic battery method
	Range	0 to 19.99 mg/L
	Resolution	0.01 mg/L
	Repeatability	±0.1 mg/L
	Accuracy	±0.2 mg/L
Conductivity ● Auto range ● Automatic temperature conversion (25 °C)	Measuring principle	4 AC electrode method
	Range	0 to 9.99 S/m
	Resolution	0.1 % of full scale
	Repeatability	±1 %
	Accuracy	±3 %
Salinity	Measuring principle	Conductivity conversion
	Range	0 to 4 %
	Resolution	0.01 %
	Repeatability	±0.1 %
	Accuracy	±0.3 %
Total Dissolved Solid(TDS) ● Conversion factor setting	Measuring principle	Conductivity conversion
	Range	0 to 99.9 g/L
	Resolution	0.1 % of full scale
	Repeatability	±2 g/L
	Accuracy	±5 g/L
Seawater specific gravity ● Display σ_t, σ_0, σ_{15}	Measuring principle	Conductivity conversion
	Range	0 to 50 σ_t
	Resolution	0.1 σ_t
	Repeatability	±2 σ_t
	Accuracy	±5 σ_t
Temperature	Measuring principle	Thermistor method
	Range	0 to 55 °C
	Resolution	0.01 °C
	Repeatability	±0.3 °C
	Accuracy	±1.0 °C

		U-22XD
Turbidity (TURB)	Measuring principle	Penetration and scattering method
● Unit selection	Range (NTU or mg/L)	0 to 800 NTU
	Resolution	0.1 NTU
	Repetability	±3 %
	Accuracy	±5 %
Water depth	Measuring principle	Pressure method
	Range	0 to 100 m
	Resolution	0.1 m
	Repetability	±3 %
	Accuracy	±5 %
Oxidation-reduction potential (ORP)	Measuring principle	Platinum electrode method
	Range	± 1999 mV
	Resolution	1 mV
	Repetability	± 5 mV
	Accuracy	±15 mV
Simultaneous measurement items		10

Note: The accuracy rating value is obtained from measurements at intermediate point of the standard solution after two-point calibration (at room temperature and pressure). The repeatability and accuracy rating percentages are based on the full scale (except for salinity).

*1: Organic solvents, strong acids, and strong alkaline solvents cannot be measured.

*2: Based on the data measured automatically at 15 minutes intervals. The battery life taken into account. Periodical maintenance and calibration is necessary when a lot of shellfishes and seaweeds exist at the measurement point.

7.4 Spare parts

Sensors

Sensor	Model	Spare part number
pH sensor	6230	9037-0056-00
pH/ORP sensor	6280	9037-0057-00
DO sensor	5460	9037-0058-00

Standard and internal solutions

Solution	Model	Spare part number	Remark
pH 4 standard solution (500 mL)	100-4	9003-0016-00	Standard solution for AUTO calibration, which is in addition used for manual pH span calibration.
pH 7 standard solution (500 mL)	100-7	9003-0017-00	Standard solution for pH zero calibration
pH 9 standard solution (500 mL)	100-9	9003-0018-00	Standard solution for manual pH span calibration
Powder for ORP standard solution (250 mL × 10)	160-51	9003-0031-00	Powdered standard solution to be used for checking ORP behavior
Powder for ORP standard solution (250 mL × 10)	160-22	9003-0030-00	
pH reference internal solution (250 mL)	330	9037-0052-00	Replenishment internal solution for pH reference electrode

Others

	Model	Spare part number	Remark
Calibration beaker XD	–	9037-0086-00	This is similar to the standard accessory, and used for sensor calibration.
Connector plug for the prove	–	9037-0071-00	When using the probe separately from the instrument, this is used to maintain waterproof of the connector.
Sensor spanner	–	9037-0088-00	This is used to connect the sensor to the probe. Similar to the standard accessory.
DO diaphragm replacement kit	–	9037-0074-00	In case of breakage of the DO sensor diaphragm, it is used in the replacement of the diaphragm to restore the sensor.
Battery cover packing	–	9096-0013-00	Replacement packing to be used for battery box of the main unit.
System unit cover O-ring	–	9096-0014-00	Replacement packing to be used for EXT cover of the main unit.
Sensor O-ring	–	9037-0076-00	Replacement O-ring to be used for connector of pH/ORP sensor and Do sensor.
Probe cap XD	–	9037-0087-00	This cap is to be used when storing the sensor probe.
Battery caver O-ring	–	9037-0084-00	This replacement O-ring is used for the sensor probe's battery cover.
Silicon grease	–	9037-0085-00	This silicon grease is applied on the sensors' O-rings. Similar to the standard accessory.
Protection cover packing	–	9037-0091-00	This is packing for when taking off the probe cap and seal after installing the protection cover. (board packing and O-ring set)
Sponge	–	9037-0089-00	This replacement sponge is used for the probe cap XD.
Protection cap	–	9037-0090-00	This cap is to be attached to the protection cover.

* The spare parts above are prepared by dealers.
Order the part by designating the parts name, model and spare parts number.

7.5 Option

Parts name	Model	Remark
Expansion adaptor	U-2001	This is applicable to AC adapter connection, RS-232C communication, GPS connection, printer output, and data-collecting software.
System unit *	U-2002-100V U-2002-110V U-2002-220V	This is applicable to AC adapter connection, RS-232C communication, GPS connection, printer output, and data-collecting software. GPS and printer are included in a complete set.
AC adaptor (for 100 V)	AC-10	AC adapter intended to drive the U-20 series by AC power supply. This should be used together with U-2001 and U-2002.
Carrying case	W-2010	Compact carrying case for cable below 10 m in length . Not large enough to hold flow cell or guard.
Carrying case	W-2030	Bigger-sized carrying case for cable exceeding 30 m in length. Large enough to hold flow cell.
Flow cell	W-2100	To be used for measurement at a pumping up sample.
Probe guard	W-2200	To be used for measurement at a location where there is a flow or a location with a thick sludge layer residing at the bottom.
PC connection cable	—	Nine-pin connection cable to PC.

* Specify the power source and voltage of the printer when ordering.

8. Reference Data

● pH measurement

1. Principle of pH measurement

U-20XD series use the glass electrode method for pH measurements. The glass electrode method measures a potential difference between the glass film for pH and the comparison electrode. For more information, refer to JIS Z 8802 pH measurement method.

2. Temperature compensation

The electromotive force generated by the glass electrode changes depending on the temperature of the solution. Temperature compensation is used to compensate for the change in electromotive force caused by temperature.
This function does not compensate the change in pH caused by the temperature of the solution. When pH is to be measured, the temperature of the solution when the pH is measured must be recorded along with that pH value, even if a meter that has automatic temperature compensation is used. If the solution temperature is not recorded, the results of the pH measurement may be meaningless.

3. Types of standard solutions

When measuring pH, the pH meter must be calibrated using a standard solution. There are five kinds of standard solutions specified in "JIS 28802 pH measurement". For normal measurement, two of standard solutions with a pH of 4, 7, and 9 are sufficient to accurately calibrate the meter.
For standard solutions, refer to "JIS Z 8802 pH measurement".

pH 4 standard solution ..... 0.05 mol/L potassium hydrogen phthalate aqueous solution
(Phthalate)
pH 7 standard solution ..... 0.025 mol/L potassium dihydrogenphosphate, 0.025 mol/L sodium phospate aqueous solution
(Neutral phosphate)
pH 9 standard solution ..... 0.01 mol/L tetra-sodium boric acid aqueous solution
(Borate)

pH values of pH standard solutions at various temperatures settings.

Temp. (°C)	pH 4 standard solution Phthalate	pH 7 standard solution Neutral phosphate	pH 9 standard solution Borate
0	4.01	6.98	9.46
5	4.01	6.95	9.39
10	4.00	6.92	9.33
15	4.00	6.90	9.27
20	4.00	6.88	9.22
25	4.01	6.86	9.18
30	4.01	6.85	9.14
35	4.02	6.84	9.10
40	4.03	6.84	9.07
45	4.04	6.84	9.04

4. Supplements for pH measurement

Pressure compensation diaphragm

U-20XD series can measure pH with high accuracy through the pressure compensation diaphragm without being affected by hydraulic pressure. Attention should be paid to the following points so that the diaphragm may function fully.
Before measurement, use a syringe and fill the reference electrode up to the replenish port with the internal solution. When injecting the polarity reference internal solution, be careful that air bubbles do not get into the solution.

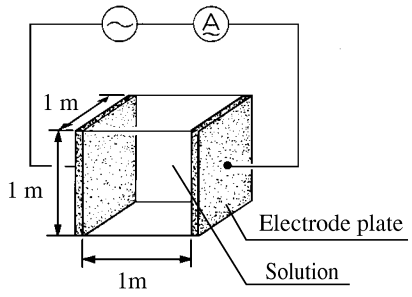
● COND measurement

1. Four-AC-electrode method

Conductivity is an index of the flow of electrical current in a substance.

Salts dissolved in water are separated into cations and anions. Such solution is called electrolytic solution. Electrolytic solution has the property of allowing the flow of current according to Ohm's law. This property is referred to as ionic conductivity, since current flow is caused by ion movement in electrolytic solution. Metals, on the other hand, allow the flow of current by means of electrons. This property is called electronic conductivity, which is distinguished from ionic conductivity.

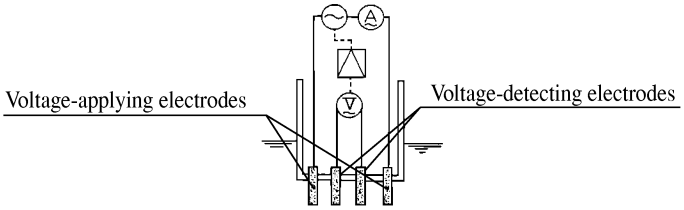
A cube with 1 m on each side, as shown in Fig. 1, is used to demonstrate an electrolytic solution. Two electrode plates are placed on opposite sides, and the cube is filled with a solution. If the resistance between these two electrode plates is represented by r (Ω), the conductivity of the solution L ($S.m^{-1}$) is represented as L=1/r. S stands for Siemens, a unit of measurement of conductance.



(Fig. 1 Definition of conductivity)

The most general method for measuring conductivity is based on the above principle, and is called the 2-electrode method. In the 2-electrode method the influence of polarization cannot be ignored for solutions with high conductivity and conductivity cannot be measure accurately. In addition, contamination on the surface of the electrode increases apparent resistance, resulting in inaccurate measurement of conductivity.

The U-20XD series has adopted the 4-electrode method to overcome these disadvantages of the the 2-electrode method. As shown in Fig. 2, the U-20XD series uses two voltage-detecting electrodes and two voltage-applying electrodes, for a total of four electrodes. The voltage-detecting electrodes are for detecting AC voltage, and the voltage-applying electrodes are for applying AC voltage.



(Fig. 2 Principle of the 4-electrode method)

Let us assume that the current, I (A), flows in a sample of conductivity L – under automatic control of the voltage-applying electrodes – so that the voltage at the voltage detecting-electrodes, E (V), remains constant at all times. Then, the resistance of the sample, R (Ω), across the voltage-detecting electrodes is represented as R=E/I. The resistance, R, of the sample is inversely proportional to its conductivity, L. Accordingly, a measurement of conductivity, Is, of a standard solution of known conductivity, Ls, enables calculation of conductivity of a sample according to the formula L = Ls (I/Is) from the ratio L : Ls = I : Is.

Even in the 4-electrode method, polarization occurs, since AC current flows in the voltage-applying electrodes. The voltage-detecting electrodes are, however, free from the effects of polarization, since they are separated from the voltage-applying electrodes, and furthermore, current flow is negligible. Therefore, the 4-electrode method is an excellent method to enable measurement of conductivity covering a very high range.

2. SI units

New measurement units, called SI units, have been in use from 1996. Accordingly, the U-20XD series also uses SI units. The following conversion table is provided for people who use the conventional kind of conductivity meter. Note that along with the change in unit systems, the measurement values and cell counts have also changed.

	Former units	→	SI units
Measurement	0.1 mS/cm	→	0.01 S/m
value	1 mS/cm	→	0.1 S/m
	100 mS/cm	→	10 S/m

3. Temperature coeffcient

In general, the conductivity of a solution varies largely with its temperature. The conductivity of a solution depends on the ionic conductivity, described earlier. As the temperature rises, conductivity becomes higher since the movement of the ions becomes more active. The temperature coefficient shows the change in % of conductivity per ˚C, with a certain temperature taken as the reference temperature. This is expressed in units of %/˚C. The temperature coefficient assumes the premise that the conductivity of a sample changes linearly according to temperature. Strictly speaking, with actual samples, however, conductivity changes along a curve. Furthermore, the cuve varies with the type of sample. In the ranges of smaller temperature changes, however, samples are said to have the temperature coefficient of 2 %/˚C (at reference tempreture 25 ˚C) this holds for most samples, except in certain special cases.
(The temperature coefficients for various types of solutions are listed on the next page.)
The U-20XD series uses an automatic temperature conversion function to calculate conductivity at 25 ˚C at a temperature coefficient of 2 %/˚C, based on the measured value of the temperature. Results are displayed on the readout.
The U-20XD series's temperature conversion function is based on the following formula.

$$L_{25} = L_t / \{ 1 + K (t - 25) \}$$

L_{25} : Conductivity of solution converted to 25 ˚C
(value displayed on U-20XD series)
t : Temperature of solution at time of measurement (˚C)
L_t : Conductivity of solution at t (˚C)
K : Temperature coeffcient (%/˚C)

Conductivity and temperature coefficient for various types of solutions

Conductivity and related temperature coefficients of representative substances (at 25 °C) are shown in the table below.

Substance	Concentration wt%	Conductivity S/m	Temperature coeffcient %/°C	Temperature °C
NaOH	5	19.69	2.01	15
	10	31.24	2.17	
	15	34.63	2.49	
	20	32.70	2.99	
KOH	25.2	54.03	2.09	15
	29.4	54.34	2.21	
	33.6	52.21	2.36	
	42	42.12	2.83	
NH_3	0.1	0.0251	2.46	15
	1.6	0.0867	2.38	
	4.01	0.1095	2.50	
	8.03	0.1038	2.62	
HCl	5	39.48	1.58	18
	10	63.2	1.56	
	20	76.15	1.54	
	30	66.20	1.54	
H_2SO_4	5	20.85	1.21	18
	10	39.15	1.28	
	20	65.27	1.45	
	40	68.00	1.78	
	50	54.05	1.93	
	60	37.26	2.13	
	100.14	1.87	0.30	
HNO_3	6.2	31.23	1.47	18
	12.4	54.18	1.42	
	31	78.19	1.39	
	49.6	63.41	1.57	
H_3PO_4	10	5.68	1.04	15
	20	11.29	1.14	
	40	20.70	1.50	
	45	20.87	1.61	
	50	20.73	1.74	
NaCl	5	6.72	2.17	18
	10	12.11	2.14	
	15	16.42	2.12	
	20	19.57	2.16	
	25	21.5	2.27	

Substance	Concentration wt%	Conductivity S/m	Temperature coeffcient %/°C	Temperature °C
Na_2SO_4	5	4.09	2.36	18
	10	6.87	2.49	
	15	8.86	2.56	
Na_2CO_3	5	4.56	2.52	18
	10	7.05	2.71	
	15	8.36	2.94	
KCl	5	6.90	2.01	18
	10	13.59	1.88	
	15	20.20	1.79	
	20	26.77	1.68	
	21	28.10	1.66	
KBr	5	4.65	2.06	15
	10	9.28	1.94	
	20	19.07	1.77	
KCN	3.25	5.07	2.07	15
	6.5	10.26	1.93	
NH_4Cl	5	9.18	1.98	18
	10	17.76	1.86	
	15	25.86	1.71	
	20	33.65	1.61	
	25	40.25	1.54	
NH_4NO_3	5	5.90	2.03	15
	10	11.17	1.94	
	30	28.41	1.68	
	50	36.22	1.56	
$CuSO_4$	2.5	10.90	2.13	18
	5	18.90	2.16	
	10	32.00	2.18	
	15	42.10	2.31	
CH_3COOH	10	15.26	1.69	18
	15	16.19	1.74	
	20	16.05	1.79	
	30	14.01	1.86	
	40	10.81	1.96	
	60	4.56	2.06	

● SAL conversion

The U-20XD series is designed to measure salinity as well as the other parameters.
Note that the "salinity" referred to here is the salinity of sea water. There is a constant relation between conductivity and salinity at certain temperatures.
Therefore, if data on the conductivity and temperature are available, the corresponding salinity is known. In other words, the salinity measurement of the U-20XD series is based on the principle of calculating the salt content, making use of the measured values of conductivity and temperature.
Note therefore, that measured results of all substances whose conductivity is detected are displayed as salinity. For example, the measured result is displayed as NaCl concentration, even if in fact the sample component is, for example, hydrochloric acid (HCl).

● TDS conversion

TDS is short for Total Dissolved Solids and means the total dissolved solid amount.
The conductivity of a solution is affected by the amount of salinity, minerals, and dissolved gases. That is, conductivity is an index that shows the total amount of all substances in the solution. Of these substances, TDS indicates only the amount of dissolved solids.
TDS can be used for a comparison of the state of substances composed of a single component such as NaCl. However, the use of TDS for the comparison of solutions of different types causes serious errors.
Conductivity and TDS are expressed by the following formulas:

Conductivity in SI units (S/m)	TDS(g/L) = L (S/m) × K × 10
	TDS(g/L) = L (mS/m) × K ÷ 100
Conductivity in the old units (mS/cm)	TDS(g/L) = L (mS/cm) × K
	K = TDS coefficient

Initial settings use the values listed in the table (☞ Page 70) that generally uses TDS coefficients.
For accurate TDS comparisons, find the TDS coefficient from measured conductivity values. Then set the value thus obtained and make measurements.

● σ_t conversion

Specific gravity of seawater

The density and specific gravity of seawater are equal numerically and generally are not distinguished strictly. Since seawater density ρ is between 1.000 and 1.031, 1 is subtracted from ρ and σ is obtained by multiplying the value by 1000.
The resultant value is used as the specific gravity of seawater.

$$\sigma = (\rho - 1) \times 1000$$

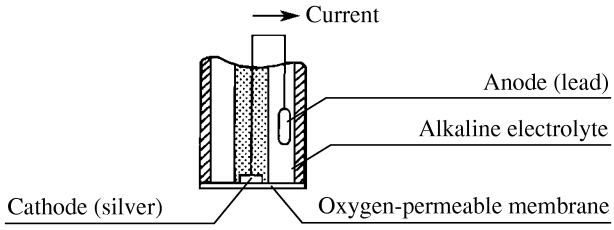
The density of seawater ρ is expressed by temperature, hydraulic pressure, and salinity functions. The density of seawater σ under the atmospheric pressure is expressed as σ_t. The density of seawater under the atmospheric pressure is determined by temperature and salinity.
The U-20XD series models make salinity measurement through temperature measurements and conductivity conversion and find σ_t through calculations.
In Japan σ_{15} at 15 °C is called a standard specific gravity and widely used while in foreign countries σ_0 at 0 °C is employed. σ_{15} and σ_0 are determined by the function of salinity.
In ocean surveys, in particular, these values σ_t, σ_{15}, and σ_0 are more widely used than conductivity and salinity and, in the U-20XD series models, newly added as measurement components.

● DO measurement

1. Principle of measurement

The "DO" referred to here means the concentration of oxygen dissolved in water. DO is essential to self-purification of river and sea and to water creatures such as fish. DO measurement is also essential to drainage and water quality control.

Fig. 3 shows the principle of measurement using a DO sensor.



(Fig. 3 Principle of DO sensor)

A noble metal (silver) is fitted closely to an oxygen-permeable membrane to make the cathode; a base metal (lead) is used as the anode. Both are immersed in an alkaline electrolyte with the anode-to-cathode external circuit closed. Oxygen diffusing through the oxygen-permeable membrane causes a reduction reaction at the cathode; this allows flow of current in the external circuit:

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$$

At the anode, oxidation reaction occur as follows:

$$2Pb \rightarrow 2Pb2^+ + 4e^-$$

The current is proportional to the quantity of oxygen diffusing through the oxygen-permeable membrane. Accordingly, measurement of the current makes the DO in a sample known.

The DO measuring method based on this principle is called the membrane-electrode method. This method allows convenient measurement of DO, especially when compared with chemical-analysis method, which needs complicated pre-treatment to eliminate the effects of oxidizing or reducing substances.

2. DO correction for salinity

When a solution and air are in contact and in complete equilibrium (saturated), DO: C [mg/L] in the solution, and the oxygen partial-pressure: Ps [MPa] in air are in the following relation:

$$C = Ps/H$$

H [MPa/ (mg/L)] is referred to as Henry's constant, which depends on the composition of the solution. In general, C becomes smaller as the salinity in the solution increases, since H becomes larger.

A DO sensor is intended to detect Ps in the above expression. Therefore, the DO measurement would be in error if the DO sensor were immersed either in air-saturated pure water or in solution with salt. To settle this problem, it is necessary to correct the DO reading based on the salinity of the sample using salinity correction.

Conventional DO meters make this salinity correction by inputting a known salinity value. This poses no problems if the salinity of the sample is known. In general, however, the salinity of the sample is usually not known, and the method is not practical even if the DO meters are equipped with the salinity correction function.

The U-20XD series is capable of measuring the salinity of a sample and automatically correcting the using this function.

3. Features of the U-20XD series DO sensors

In conventional DO measurements, it was necessary to keep the velocity of the flow constant because the velocity of flow led to fluctuation in indicated values. In our U-20XD series models, improvements in sensors have made it possible to make measurements with stable indications and with little influence of the velocity of flow.

● Turbidity (TURB) measurement

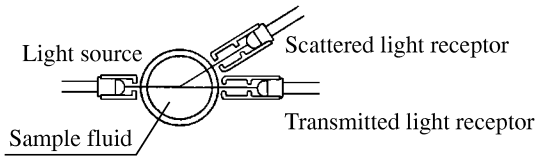
1. Principle of measurement

From among several types of turbidity-measuring methods available, the U-20XD series uses the light-transmission-scattering method, shown in Fig. 4.

Irradiation of a beam of light onto a sample brings about separation of the beam into (1) the light transmitted through the solution and (2) the light scattered by turbidity components in the sample. In the light-transmission-scattering method, the intensity of both transmitted light and the scattered light are measured using separate receptors, and the turbidity is obtained based on the ratio of the two.

With the U-20XD series, the light source is a pulse-lighting infrared-emission diode. The scattered light is measured at a point 60° offset from the light source. This light-absorption-scattering method has several advantages, including the fact that (1) the actual color of the sample fluid has little effect on the measurement of turbidity, (2) fluctuations in light quantity from the light source are easily compensated for, and (3) it allow the U-20XD series to be operated with relatively low-power consumption.

The turbidity value differs with the structure of the cell so changes with the instrument.



(Fig. 4 Principle of the light-transmission-scattering method)

2. Standard solution

U-20XD series can perform calibration using formazin (NTU) or kaolin standard solutions as a turbidity standard solution. However, units for the solution used for calibration should be displayed in measurements. Do not use more than 400 mg/L of kaolin standard solution because it increases precipitation speed, resulting in measurement error.

● DEP measurement

1. Depth (DEP) measurement

For the U-22XD model, depth measurement can be made through use of a pressure gauge. The principle of the depth measurement uses the relation between depth and pressure.

Although the measurement with the depth sensor is affected by atmospheric pressure, the depth sensor, however, makes zero-point adjustments through the automatic calibration before measurements.

2. Influence of temperature and calibration

The depth sensor depends greatly on temperature. For a wide difference between the temperature at which the sensor has been automatically calibrated and the temperature of the measurement sample, the sensor can make depth measurements with a higher accuracy by the following method:

Immerse the depth sensor of the sensor probe into the sample.

Keep the sensor immersed in the sample for approximately 30 minutes until the temperatures of the sensor and the sample are the same.

Then make the zero calibration of the sensor manually. (☞ Page 57)

● Measuring mV (oxidation-reduction potential (ORP))

ORP principles

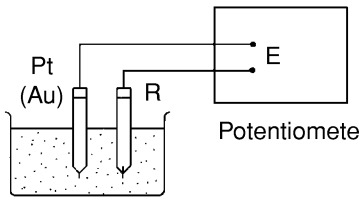
ORP (or "redox potential") is an abbreviation for oxidation-reduction potential. ORP is the energy level (potential) determined according to the state of equilibrium between the oxidants (M^{Z+}) and reductants $M^{(Z-N)+}$) that coexist within a solution.

$M^{z+} + ne^- \Leftrightarrow M^{(z-n)+}$.....①

If only ① exists within a solution, a metal electrode (platinum, gold, etc.) and a reference electrode are inserted into the solution, forming the ORP measuring system shown in Fig. 5. Measuring the potential (ORP) that exists between the two electrodes enables the potential to generally be expressed by the following equation.

$$E = E_0 - \frac{RT}{nF} \ln \frac{a_{M^{(z-n)+}}}{a_{M^{z+}}} \quad \text{..... ②}$$

E: Electric potential E_0 : Constant R : Gas constant T : Absolute temperature
n : Electron count F : Faraday constant a : Activity



(Fig. 5 Measuring mV)

For example, for a solution in which trivalent iron ions coexist with bivalent iron ions, equations ① and ② would be as follows.

$Fe^{3+} + e^- \Leftrightarrow Fe^{2+}$.....①

$$E = E_0 - \frac{RT}{F} \ln \frac{a_{Fe^{2+}}}{a_{Fe^{3+}}} \quad \text{.....②}$$

When only one type of state of equilibrium 1 exists in the solution, the ORP of the solution can be determined uniquely by equation 2. What is important here is that ORP is determined by the ratio of activity between the oxidant (Fe^{3+}) and the reductant (Fe^{2+}) (using the equation $a_{Fe^{2+}}/a_{Fe^{3+}}$). Actually, however many kinds of states of equilibrium exist simultaneously between various kinds of ions, in most solutions. This means that under actual circumstances, ORP cannot be expressed using the simple equation shown above and that the physical and chemical significance with respect to the solution is not very clear.

In this respect, the value of ORP must be understood to be only one indicator of the property of a solution. The measurement of ORP is widely used, however, as an important index in the analysis of solutions (potentiametric titration) and in the disposal and treatment of solutions.

Recently, there have appeared various claims regarding this matter, such as that a high degree of ORP is effective in sterilization or that drinking water that has a low ORP reduces the chance of illness by reacting with the activated oxygen in the cells of the body. ORP is used as an index for alkaline drinking water.

Standard electrode (reference electrode) types and ORP

The ORP of a solution that is obtained through measurement is a value that corresponds to the reference electrode employed.
If different kinds of reference electrodes are used for measurement, the ORP value of the same solution may appear to be different. HORIBA uses Ag/AgCl with 3.33 mol/L KCl as the reference solution for reference electrodes. According to general technical literature, standard hydrogen electrodes (N.H.E.) are often used as the standard electrode.
The relationship between N.H.E. and the ORP that is measured using an Ag/AgCl with 3.33 mol/L KCl electrode is expressed by the following equation.

$E_{N.H.E.} = E + 206 - 0.7\,(t - 25)\text{mV} \qquad t = 0 - 60\,℃$

$E_{N.H.E.}$: Measured ORP value using N.H.E. as the reference electrode

E: Measured ORP value using Ag/AgCl with 3.33 mol/L KCl as the reference electrode

Potential sign

Standard ORP is expressed in the following way, in literature related to electrochemistry and analytical chemistry.

A $Li^{+}+e^{-} \rightarrow Li$

E0=-3.024 V VS N.H.E.

However, in some literature, the "+" and "-" signs are reversed.

B $Li \rightarrow Li^{+}+e^{-}$

E0=+3.024 V VS N.H.E.

In expressions like B, above, the reaction is just reversed and there is no essential difference. But this kind of expression does invite confusion. The majority of the world, today, is consistent in its use of the signs as they are used in A, above. For this reason, HORIBA, too, uses signs concerning ORP that are consistent with A, above.

表 3

For any question regarding this product,
please contact your local agency,
or inquire from the Customer Registration
website (www.horiba.co.jp/register).

HORIBA, Ltd.

First edition: November 2001
CODE : I1000908000

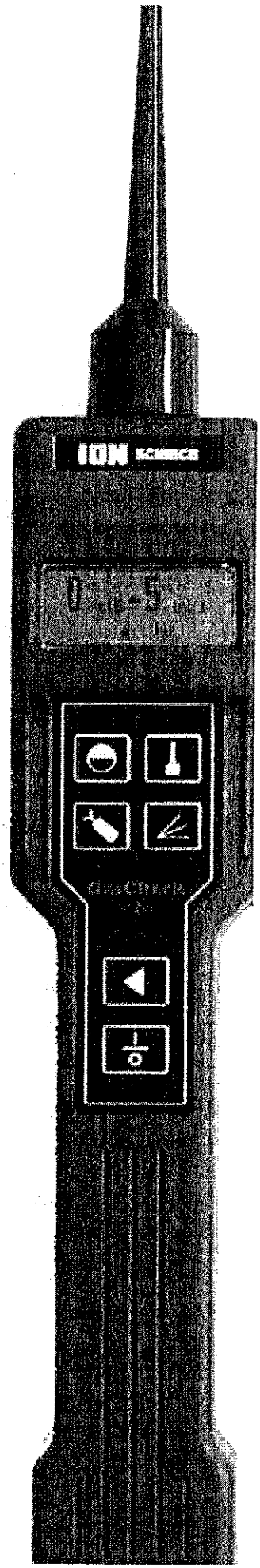
Attachment G

GasCheck 5000I Instruction Manual

GasCheck

Instruction Manual

Models Covered
5000 and 5000is



Declaration of Conformity

Ion Science Ltd

The Way

Fowlmere

Cambridge SG8 7UJ

England

Declare that the products

Gas Check 5000 and Gas Check 5000is

comply with the requirements of :

89/336/EEC	EMC Directive
76/117/EEC	Equipment and protective systems intended for use in potentially explosive atmospheres (Gas Check 5000is only).

in conformity with the following standards and specifications:

EN 50081-1 EMC Emissions - light industrial

EN 50082-1 EMC Immunity - light industrial

EN50014 Electrical equipment for use in potentially explosive atmospheres (Gas Check 5000is only)

EN50020 Electrical equipment for use in potentially explosive atmospheres (Gas Check 5000is only)

Product marking is as follows:

Gas Check 5000is carries the Intrinsic Safety Marking to class EEx ia IIC T4

Gas Check 5000 and Gas Check 5000is carry the CE-mark.

Signed:



M J STOCKDALE

Technical Director

Date: 19th May 1999

ION SCIENCE LTD

Fowlmere

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The 5000is version of this instrument is manufactured to intrinsically safe standards which are maintained and monitored by BASEEFA.

For more information about BASEEFA certification please visit www.ionscience.com/baseefa.htlm

1 INTRODUCTION

1.1 Scope and definitions

This manual provides installation, operation and maintenance instructions for the Gas Check 5000 and the Gas Check 5000is. You must use the Gas Check as specified in this manual.

Read this manual before you install and operate the Gas Check. Important safety information is highlighted as WARNING and CAUTION instructions; you must obey these instructions. The use of WARNINGS and CAUTIONS is defined below.

> WARNING
> Warnings are given where failure to observe the instruction could result in injury or death to people.

> CAUTION
> Cautions are given where failure to observe the instruction could result in damage to the equipment, associated equipment and process.

The units of measurement used throughout this manual conform to the SI international system of units of measurement

1.2 Description

Refer to Figure 1. The Gas Check is a portable, battery-powered, hand-held gas leak detector. Indications of a leak are shown on the digital display (2) and are also given by a front panel mounted LED flasher (3) and as audible clicks from the rear panel loud speaker (6). Two versions of Gas Check 5000 are available:

- The standard Gas Check 5000 is suitable for leak detection of all non-flammable gases other than air.
- The Gas Check 5000is is intrinsically safe and is suitable for leak detection of hydrogen and other flammable gases, and for leak detection in hazardous areas of EEx ia IIC T4 rating.

The Gas Check has a convenient storage and carrying case, together with a spare battery holder, a long flexible probe (for leak detection in areas where access is restricted) and a box-spanner for probe changing.

1.3 Principal of operation

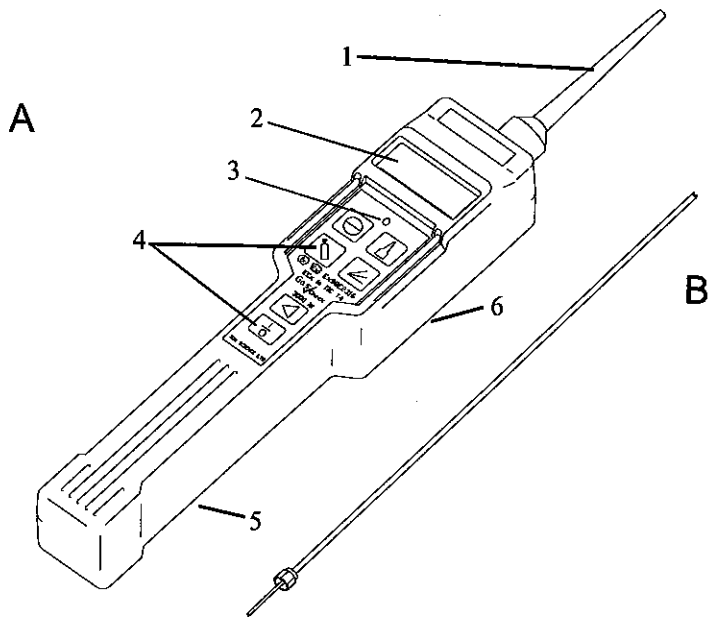
When the Gas Check is switched on, a small internal fan draws gas samples through the probe and into the Gas Check. The Gas Check measures the thermal conductivity of the gas samples.

When you first switch on the Gas Check (using the power I/O key) or when you press the zero key : see  
Section 4.1), the Gas Check zeros the leak rate display. If further samples have a different thermal conductivity from the zeroed sample, the Gas Check calculates and displays the difference as a leak rate. This means that you can use the Gas Check to detect any gas other than that in which the Gas Check is zeroed.

1.4 Applications

You can use the Gas Check on many types of applications, including those listed below.

- Quality assurance testing on manufactured component seals.
- Laboratory applications, such as the detection of leaks from gas chromatographs, from mass spectrometers, from gas cylinders and fittings.
- Industrial applications, such as the detection of leaks from gas installations, in cylinder receiving rooms, from pipeline assemblies, the detection of leaks from stored gases and the detection of vapours released from stored chemicals.
- Medical applications, such as the detection of leaks from anaesthetic gas bottles and pipelines and leak testing of membrane materials, glove boxes and so forth.
- Valve emission evaluation to EPA (Environmental Protection Agency) method 2.1
- Leak testing of pipeline joints, gaskets, chamber windows and so forth



A	Gas Check with short probe and nozzle fitted		
B	Long probe		
1	Nozzle	4	Tactile keys
2	Display	5	Battery compartment
3	LED flasher	6	Loudspeaker

Figure 1 – The Gas Check 5000 and 5000is

2 TECHNICAL DATA

2.1 General

Operating temperature range

Gas Check 5000	0 to 50 °C	32 to 122 °F.
Gas Check 5000is	0 to 40 °C	32 to 104 °F.

Storage temperature range

	-25 to 70 °C	-13 to 158 °F.

Materials of construction

Storage case	Polypropylene with polyester foam insert
Gas Check	Polyurethane casing with metal bracket on 5000is only

Dimensions

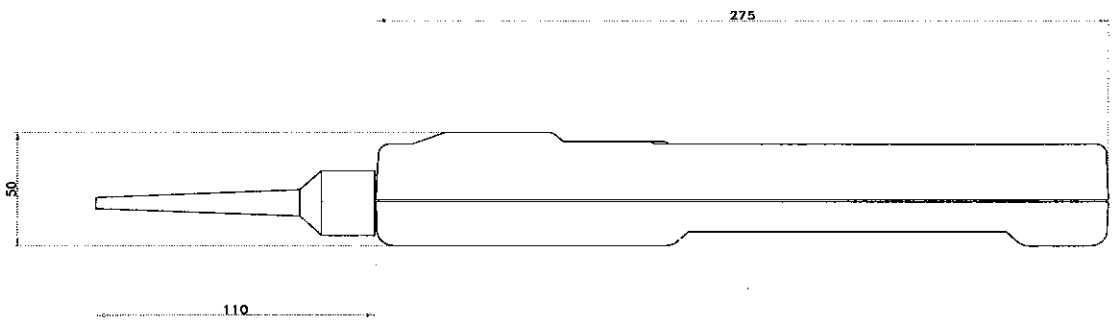
Storage case	420 x 320 x 97 mm	16.5" x 12.5" x 3.75 "
Gas Check	see Figure 2	

Mass

Complete storage case	1.6 kg	3.5 lb.
Gas Check	0.5 kg	1.0 lb.

Ingress ratings

Minimum	IP20



* Length of short probe and nozzle. Length of long probe = 300 mm (12")

Figure 2 – Dimensions (mm)

2.2 Performance

Detector type — Dual micro-volume thermal conductivity cell

Gases Detected:

All gases and vapours having a different thermal conductivity to the ambient air on which it was zeroed. The larger the difference the greater the sensitivity, so that Gas Check is not highly sensitive to the gases normally found in large concentrations in the ambient air, e.g. N2 (nitrogen) and O2 (oxygen).

For ease of use the Gas Check groups gases having similar thermal conductivity into one of the 5 gas groups shown in Table 1. Each group has a calibration curve stored in memory. Although the Gas Check will detect ALL gases with a different thermal conductivity to air, Table 1 lists those 12 common tracer gases that have been tested and assigned gas groups. If the gas or gas mixture that you seek is not included in these groups, select group 0 (which is the most sensitive). The Gas Check will then still find leaks quickly even though the measured values will not be displayed accurately. Some gases give a negative response.

Minimum response time

Short probe fitted	Less than 1 second
Long probe fitted	About 9 seconds

Recovery time — About 1 second

Maximum detection level with the short probe and nozzle fitted — 20 ml/sec

Data logging RTC — This is a nominal device for convenience and can be reset by a user if more accurate timings are required. While Gas Check 5000 operation does not depend on the RTC it has been tested for compliance with Y2K.

Minimum detection levels see Table 1

The nozzle when fitted provides automatic 10-fold dilution of the gas stream entering the Gas Check TCD, except when the ppm display is selected. The Gas Check nozzle should always be removed for assessing gas concentration levels in volume spaces smaller than about 1 litre when you are using the ppm display.

Note1: the minimum detection levels given in Table 1 apply when the Gas Check is used in clean laboratory air conditions. When used in conditions where the ambient atmosphere contains clouds of the gas for which you leak test, the minimum detection levels may be obscured.

Note2: An offset of up to 10 times the detection levels given in Table 1 may be experienced in the presence of electromagnetic interference @ 320 +- 30 MHz at the levels specified in EN 50082-1.

Table 1- Smallest Leak Detection Levels

Gas	Gas Group	Positive/Negative	Smallest Leak Detection Level in ml/sec
H_2	0	Positive	1×10^{-5} ml/sec
R12	0	Negative	1×10^{-5} ml/sec
R1301	0	Positive	1×10^{-5} ml/sec
R134a	0	Negative	1×10^{-5} ml/sec
He	1	Positive	2×10^{-5} ml/sec
SF_6	1	Negative	2×10^{-5} ml/sec
R22	1	Negative	2×10^{-5} ml/sec
CO_2	2	Negative	1×10^{-4} ml/sec
CH_4	2	Positive	1×10^{-4} ml/sec
Ar	2	Negative	1×10^{-4} ml/sec
R11	3	Positive	2×10^{-4} ml/sec
O_2	4	Positive	1×10^{-3} ml/sec

2.3 Battery data

WARNING

For Gas Check 5000is

Do not modify the Gas Check 5000is equipment to allow any recharging.

For intrinsic safety T4 rating in the Gas Check 5000is

Rechargeable batteries (e.g. Ni-Cad) must not be used.

There is no provision for the recharging of batteries within the Gas Check 5000is.

For alkaline types (MN1500 or LR6) type use only Duracell (Standard, Procell or Ultra) or Ever Ready (Energiser) batteries. Alternatively use any zinc carbon types R6PP or R6S

For Gas Check 5000

Do not modify the Gas Check 5000 equipment to allow any recharging.

Do not use in a hazardous area. Use the Gas Check 5000is.

The Gas Check 5000 is designed for use with primary (disposable) batteries.

Rechargeable batteries can be used for the Gas Check 5000 in safe areas.
There is no provision for the recharging of batteries within the Gas Check 5000.

Number of batteries required	4
Battery type	Alkaline, size AA
Recommended batteries	LR6 (R6) or MN1500
Average operating life	see Table 2

Note1: the energy storage capacity of batteries and even their T4 intrinsic safety rating is affected by ambient temperature. The data in Table 2 is for an ambient temperature of 20 °C (68 °F).
Note2: the use of the back light and the IR Data Port will dramatically shorten the battery life.

Battery type	Battery life (hours)	
	Gas Check 5000	Gas Check 5000is
LR6 (R6) *	40	20
MN1500 *	40	20
R6S	20	10
R6PP	20	10

- Recommended battery types

Table 2 – Average battery life

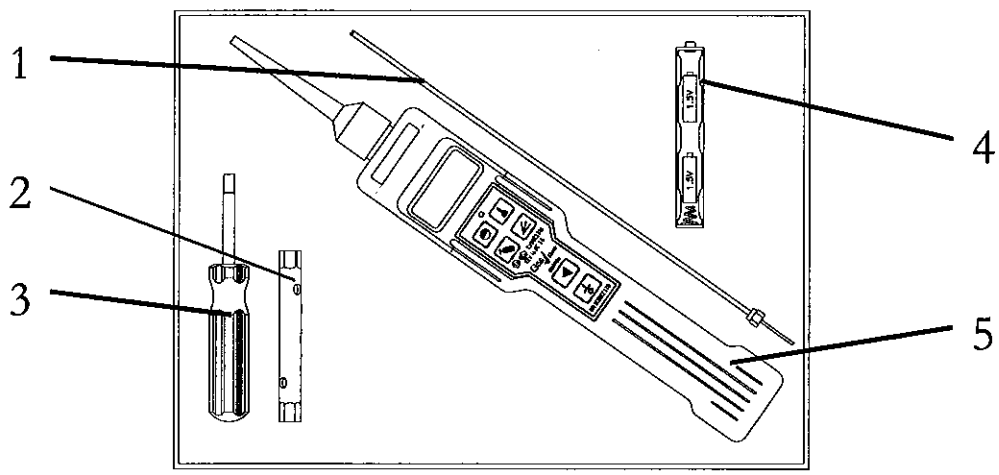
3 INSTALLATION

Remove all packaging materials, then open the storage case and inspect the equipment. If the storage case or any other item is damaged, notify your supplier and the carrier in writing within three days; state the Item Number and the Serial Number stamped on rear case of the Gas Check (refer to Figure6), together with your order number and your supplier's invoice number. Do not attempt to use the Gas Check if any item is damaged.
The batteries if provided are shipped in the spare battery clip and not inside the GasCheck instrument itself.

Refer to Figure 3 and check that the storage case contains the items listed in Table 3. If any item is missing, notify your supplier in writing within three days.

Qty	Description	Check ( )
1	Gas Check (with short probe and nozzle fitted)	
1	Long probe	
1	Box-spanner	
1	Special screwdriver for access to battery compartment (Gas Check 5000is only)	
1	Spare battery holder with 4 batteries	

Table 3 – Checklist of items in the storage case



1. Long probe
2. Box-spanner
3. Special screwdriver (Gas Check 5000is only)
4. Spare battery holder
5. Gas Check (with short probe and nozzle fitted)

Figure 3 – Items in the storage case

4 OPERATION

Gas Check 5000 and 5000is both have menu keys for user accessible settings. However normal use of the Gas Check would not involve any menus, because these are only needed to set up the instrument for your application, after which the instrument stores the settings internally when the instrument is switched off.

The display of information on the Gas Check 5000 and 5000is depends on whether the instrument has been keyed into the set up menus or is simply being used for leak detection. The Gas Check 5000 and 5000is menu displays utilise 3 lines of information while the normal display uses 2 or 3 lines; in each case the lowest line is for status information while the upper line(s) show(s) the menu topics or the leak rate values.

You may like to gain some confidence in the operation by simply using the control keys as in section 4.1.1 although if it has not yet been used you should carefully fit the battery clip in accordance with section 5.2. Please take note of the warnings and cautions when fitting batteries or when in potentially hazardous areas.

4.1 Controls

In normal use there is only one key to press, beyond the power ON/OFF control, and that is the ZERO key.

Refer to Figure 4. Use the control keys as described below. The complete menu structure, which you can access with menu key (3), is described in section 4.1.3. The display symbols are described in Section 4.2.2.

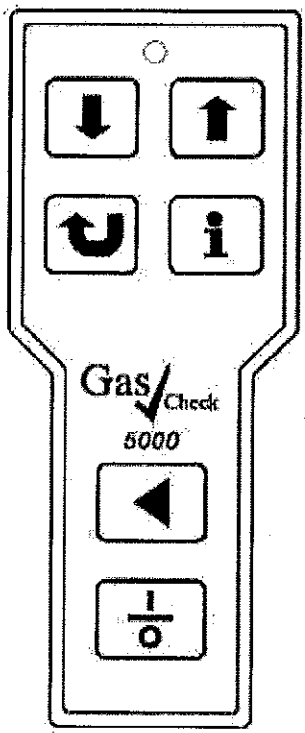


Figure 4 – Control keys (layout shown for both Gas Check 5000 and 5000is)

4.1.1 **Control keys**

POWER I/O (1)



Press this key to switch the Gas Check POWER on for use. When this key is pressed from normal display, Gas Check will turn its POWER off, remembering all last settings (except back light reverts to OFF state). When in menus, pressing this key brings Gas Check straight back into the normal display WITHOUT any of the settings changes being implemented.

ZERO/BACK (2)



Just press zero to adjust for background levels, reset the peak hold memory or after changing a gas group. In menus, successive presses of this key provides a simple step by step route BACK to the normal display.

PROCEED (3)



Press the proceed key to reach the menus that allow you to control special settings of the Gas Check 5000.

DOWN (4)



Press or hold the Down key to scroll up through menus or to raise a numerical value. Release when desired.

UP (5)



Press or hold the Up key to scroll down through menus or to lower a numerical value. Release when desired.

INFO (6)



Press the Info key to start data logging as per the parameters last set via the data control menu as described in section 4.1.2.

4.1.2 **Menu topics and corresponding displays**

The first pressing of the PROCEED key switches the display into the Gas Check 5000 series menus. These menus are shown by the relevant menu topic on the top line and the available choice and the current settings on the middle and bottom display lines. The UP and DOWN keys scroll round the menus choices. Some of the menus show a tick or cross at the right hand side to indicate the current settings or choices. The PROCEED key selects the current topic, and in some menus pressing this will change the tick for a cross or a cross for a tick and also alter the instrument running status symbols displayed on the lowest line. This has been found to help customers readily understand the menu selection for new instrument settings.

Basic Display Details:

Top Line:	*Name of Menu or Topic* ◀ *Back*
Middle Line:	*Operation. Keys* *Currently user adjustable*
Bottom Line:	*Instrument running status or Current Setting of Topic*

Where the menu structure has an underlying sub-menu structure for settings, the bottom line is blanked.

Main menu

ABC	Automatic Background Compensation (not normally used)
Alarm Level	Setting for the Audible/Visual Alarm
Audio	Mute the leak rate Audible/Visual
Backlight	Controls for the Back light display
Clock	Data logging clock functions
Data Control	Functions for data control including download via IR Link
Display	Bar graph or numeric display functions
Gas Group	Choice of Gas Group or named Gases
Language	Specialist option instead of English etc.
Nozzle	Switch for whether nozzle is fitted or not, for leak rate calibration purposes
Peak Hold	Switch for storing maximum reading on display between each zero key press
Power off	Controls whether or not Gas Check shuts down automatically after fixed time
Program	Specialist functions to set up Gas Check 5000 via IR Link and software
Range	Autoranging or fixed range displays
Units	Leak rate units

At this level the following key functions are available

Select the subsequent topic from the main menu by the PROCEED key.

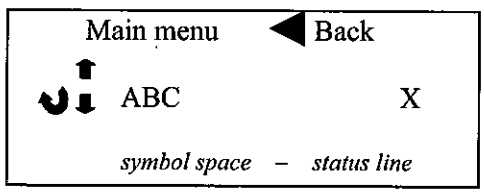
Leave the menu and go back to normal operation by the Zero/Back key ◀

Scroll through the menu by use of the up and down keys.

Escape back to the normal display without implementing any menu changes by pressing the 1/0 key once.

ABC

Display:



The X indicates that it hasn't been selected – The status line doesn't have the < symbol. If the Proceed key is pressed then the X turns to a ✓ and the < symbol appears on the status line. Press Proceed to reverse.

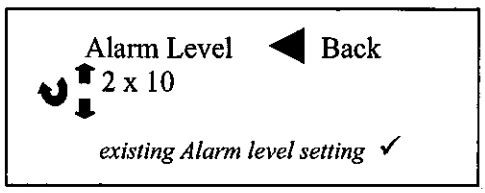
Alarm Level

The alarm level format is in the same style as the display type (see below for display style options).
The alarm level is linked to gas group setting such that if the Gas Group changes the alarm is not set, the

status symbol is removed from the display, although the last alarm level value is retained for reconfirmation. This is to ensure the Gas Check avoids incorrect alarm levels on a Gas Group change.

Press to jump down into Alarm Level setting.

Display:

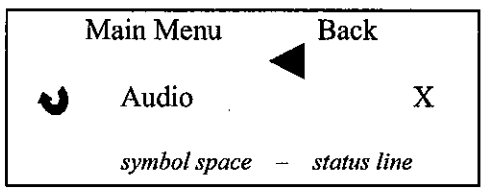


Note: When the alarm is set to maximum, the alarm function is disabled and the Main menu Alarm level display will show Not set and no bell will shown once the Gas Check is put back in the normal display.

Audio

When the audio feature is selected, the tick frequency of the Audio and Visual indicators is linked to the size of the leak rate displayed; when Audio is not selected there is no link.

Display



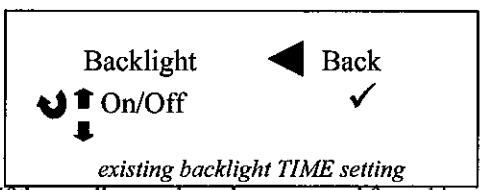
The X indicates that it hasn't been selected – The status bar doesn't have the < symbol. If Proceed key is pressed then the X turns to a ✓ and the loudspeaker symbol is placed down onto the third row.

Backlight

The backlight control allows the user to switch on the backlight or select a TIME setting.

Press to jump down into Backlight setting and then the PROCEED key or the UP and DOWN keys.

Display:

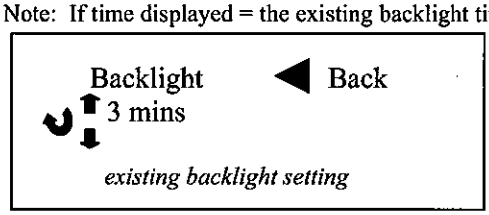


Status shown ✓ will put the Backlight ON

If the scroll up or down keys are used from this start point, then the following options are shown:-

20s 1min 3min 10min 30min

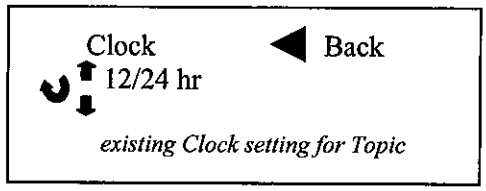
Note: If time displayed = the existing backlight time then a ✓ is shown at the end of the 2nd row, else a X .



Clock

Press ↩ *to jump down into Clock setting.*

Display:



Note: No X or ✓ at this level because we are not at the end of the tree. The full clock settings are shown.

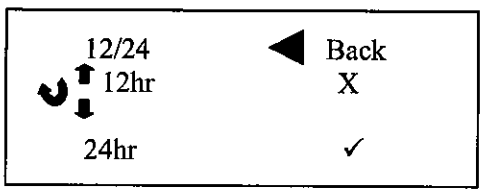
Operation of the up or down keys give access to the other topics at this level.

Date format
Set time
Set date

The following frames show the CLOCK sub-menus accessed by the ↩ key.

Clock - 12/24hr

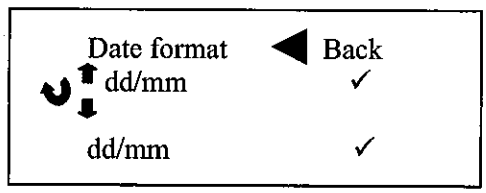
Display



Note: X because 12hr not selected but a ✓ always in the bottom row to show that this is currently selected.

Clock - Date Format

Display



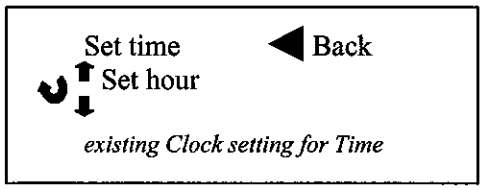
Note: Both ✓ in this example because of the same setting. This also denotes the end of the tree.

Other option within this topic is:-

mm/dd

Clock - Set Time

Display

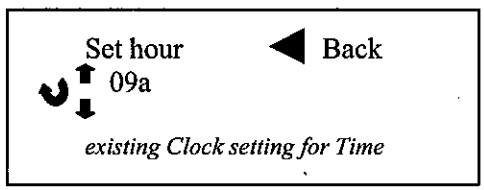


Other options within this topic are
Set min
Synch RTC

Clock - Set Time - Set hour

A press on the key takes us down into this next level. Use UP and DOWN keys to change hour value. Press PROCEED when this is what you require, and the clock will proceed to minutes then to seconds. If you are only changing hours for Daylight Saving Time changes then once PROCEED has been actioned the Back key can be used to return towards the normal display mode with the required changes stored.

Display



Clock - Set Time - Set min

As per Set hour

Clock - Set Time – Synch RTC

This allows users to confirm that the minutes setting conforms to expectations and allows set to 0 seconds. When the synchronisation time is shown and the minutes are incorrect, press Back to revert to set minutes. Pressing PROCEED will action the synchronisation to the displayed time and then revert to set time frame.

Data Control

This sub-menu has the following topics and are accessed by pressing the key.

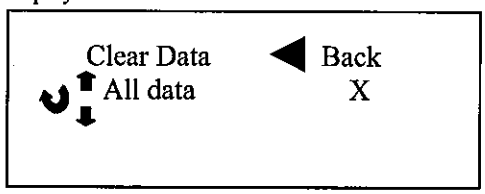
Clear data
Data transfer
Logging settings (only accessible in continuous logging mode)
Memory used
single/cont.

In most of these menus the third line shows current settings or values and also ✓ or X on the middle line.

Data Control - Clear Data

Enter this by a press.

Display



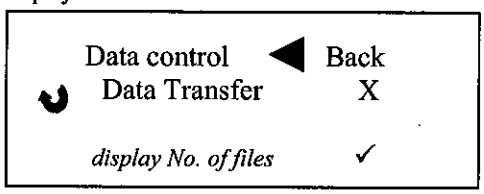
Note: An X or ✓ will be required following usual convention on the 2nd line.

Use the up or down scroll keys for other end tree selections:-

Last data X

Data Control – Data Transfer

Display



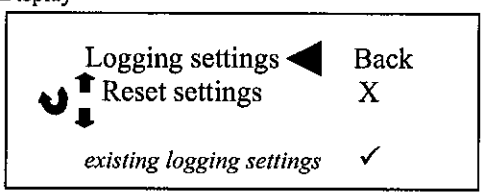
Note: An X or ✓ will be required following usual convention on the 2nd line.

When Data is being transferred, the X changes to a ✓ and then the Gas Check changes to a blank display but with the words 'Data transfer' for the duration of the transfer or time-out.

Data Control – Logging settings

Enter this by a press.

Display



Only if you want to return to default

No. Samples x Interval s ✓

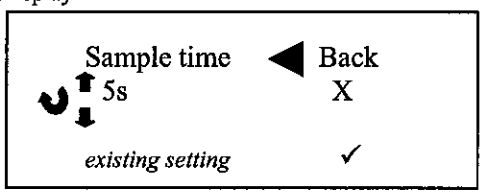
Use the up or down scroll keys for other Topic selections:-

Interval
No. Samples

Data Control – Logging settings- Interval

Enter this by a ↵ press and use the UP and DOWN keys to select a new repeat interval for data logging. Press PROCEED key to enter this value in seconds, BACK key to move back towards the normal display.

Display

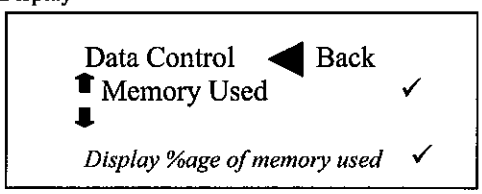


Data Control - Logging settings – No. Samples

As per Interval above. This sets up the total number of samples per record for each press of the INFO key. When the Single mode of data logging is used, this menu is NOT accessible, as only ONE sample is taken.

Data Control - Memory Used

Display

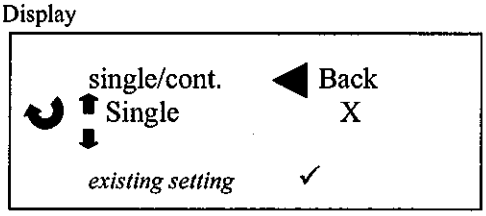


Note: This is an unusual case but we are still trying to follow the same format. This is just an information menu only but the ✓ serves as an indicator that it is an end tree situation. Also notice how the ↵ has been removed for this item.

Data Control - Single/cont.

Enter this by a ↵ press.

Display



The choice is given to toggle the 'Single' or Continuous mode of data taking (using the Logging Settings).

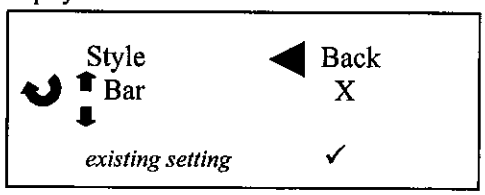
CAUTION
Please reset the Gas Check clock date and time settings required for data acquisition before proceeding to take data if this is to be used as a real time record rather than a logging sheet.

Display - Style

Gas Check 5000 allows the user to select a preferred style of presentation – either a bar graph with an exponent range indicated or a digital display with a similar exponent range. The style applies to both the normal display mode and the menu settings for alarm level.

Enter this by a ↵ press.

Display



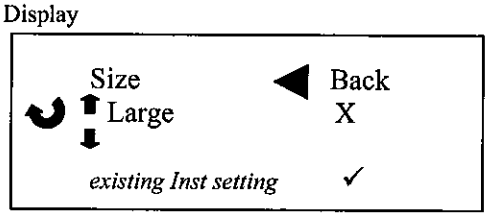
The option is given here (as per clearing Last data or All data) is for information purposes, to aid selection. The choice is given to toggle the 'Bar' at this level or to scroll through to see the other option: Numerals.

Display - Size

Gas Check 5000 allows the user to select a preferred size of the display style presentation as a larger or a smaller size for the bar graph or numerals and the exponent range value. The size only refers to the normal display settings not to the menu settings for the alarms.

Enter this by a ↵ press.

Display



The option is given here (as per clearing Last data or All data) is for information purposes, to aid selection. The choice is given to toggle the 'Large' at this level or to scroll through to see the other option: Small

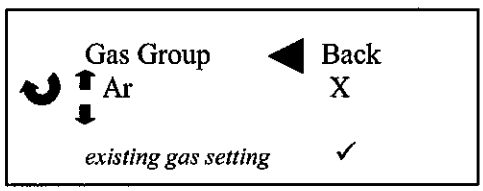
Note: Similar X and ✓ indications are followed in both Size and Style.

Gas Group

Enter this by a ↻ press.

-Ar
+CH_4
-CO_2
H_2
He
O_2
-R11
-R12
R22
-R134a
R1301
-SF_6
Group 0
Group 1
Group 2
Group 3
Group 4

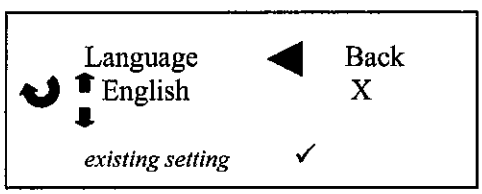
Named gases have a polarity feature that is NOT available for the Gas Groups on Gas Check 5000: the response after zeroing is ONLY allowed to be in the same sense as the polarity of the named gas.



Note: Similar X and ✓ convention is followed.

Language

Enter this by a ↻ press.

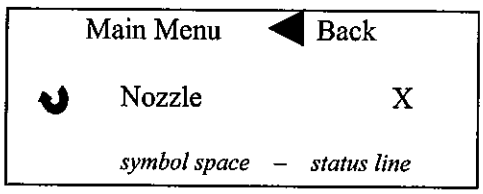


The option is given here (as per clearing Last data or All data) is for information purposes, to aid selection. The choice is given to toggle the 'English' at this level or to scroll through to see what the other options are - which in the instance is one! 2nd Programmed Language English (this is the normal factory alternative).

Note: Similar X and ✓ convention is followed.

Nozzle

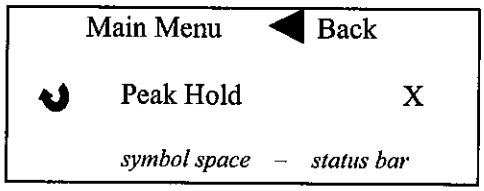
Display



The X indicates that it hasn't been selected – The status line doesn't have the nozzle symbol. If the PROCEED key pressed then the X turns to a ✓ and the nozzle symbol is placed down onto the third row

Peak Hold

Display

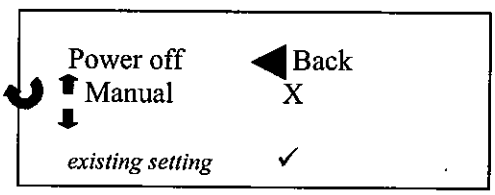


The X indicates that it hasn't been selected – The status bar doesn't have the Peak Hold symbol. If PROCEED key pressed then the X turns to a ✓ and the Peak Hold symbol is displayed in the third row.

Power off

This sub-menu has the following topics accessed by pressing the ↵ key.

Manual
Automatic



Manual will ensure the user has to press the I/O key to turn off Gas Check. Automatic waits approximately 10 minutes from last key press with signal below 50% of selected range before switching off Gas Check.

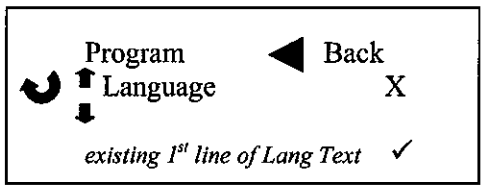
Manual will be useful for those users requiring continual operation for data logging purposes or leak ups.

Similar X and ✓ convention is followed as for other menus.

Program

This sub-menu has the following topics and are accessed by pressing the ↩ key.

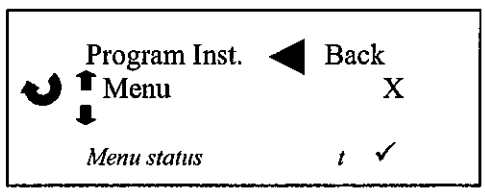
Language
Menu
Zone



Note: Similar X and ✓ convention is followed.

Pressing the PROCEED key without an IR Link communication will result in a delayed reset of the unit.

The other two selections will also be displayed because of different information content on the third line.

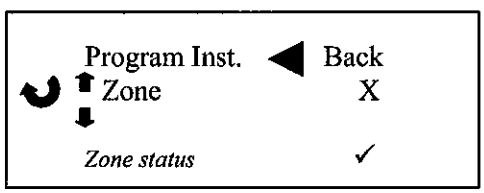


Menu status will only have a S/W test to indicate whether it is in the non programmed (normal default settings) or has any of these settings changed. THUS: the bottom line will have one of two messages:-

Factory Menu ✓
Custom Menu ✓

Pressing the PROCEED key without an IR Link communication will result in a delayed reset of the unit.

Note: Similar X and ✓ convention is followed.



Zone status will only have a S/W test to indicate whether it is in the non programmed zone or has the zone option programmed within it.
THUS: the bottom line will have one of two messages:-

Zone Set
No zone ✓

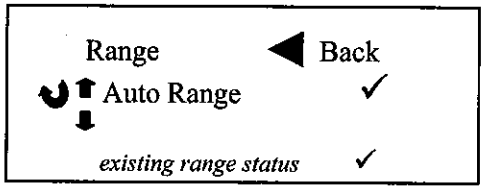
Pressing the PROCEED key without an IR Link communication will result in a timed our rest of the unit.

Note: Similar X and ✓ convention is followed.

Range

Press ↓ *to jump down into the range topic.*

Display:



Existing status shown ✓

This is Auto Range for ✓ above

If the scroll up or down keys are used from this start point, then the following options are shown:-

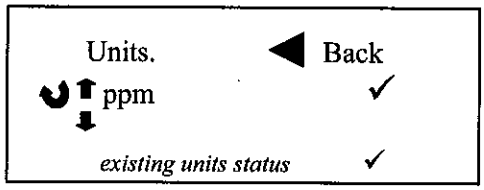
Manual $x10^3$
Manual $x10^2$
Manual $x10^1$

Note: The exponents are only an example for the units of ppm selected.

Units

Press ↓ *to jump down into the units topic.*

Display:



Existing status shown ✓

This is ppm for ✓ above

If the scroll up or down keys are used from this start point, then the following options are shown:-

ml/min
ml/s
ft^3/yr
ppm
sccm
mm^3/s
mm^3/min

Some Gas Check versions have a language selection where the units of ml are shown as cc instead, because this cc unit is more familiar than the SI based ml unit of identical volume.

4.2 Display

4.2.1 General

The Gas Check LCD matrix display is arranged to show the leak rate and other relevant status information in a consistent fashion during the normal use and also during user changes to the settings of the application.

- In normal use the upper portion of the LCD matrix is used for a large size digital representation of the leak rate (see Section 4.2.2) and status information is shown as symbols (see section 4.2.3) appearing in the lower portion.

- In response to Gas Check key presses, written text will appear across the middle of the matrix display, giving user confirmation of the changes to settings, while status symbols will also change consistently.

  Examples are

  Zeroing detector
  Nozzle On
  Peak hold On

4.2.2 Leak rate indication

Leakage is displayed as a single (signed) digit x 10 (signed) range for ease of interpretation and calibration. [Note that some gases (eg CO2) may cause a negative leak indication after Gas Check zeroing in clean air.]

Examples are

Leak rate for a positive gas: **5 x 10 -5 ml/s**
Leak rate for a negative gas **- 3 x 10 -4 ml/s**

Gas	Gas Group	Positive/Negative
H_2	0	Positive
R12	0	Negative
R1301	0	Positive
R134a	0	Negative
He	1	Positive
SF_6	1	Negative
R22	1	Negative
CO_2	2	Negative
CH_4	2	Positive
Ar	2	Negative
R11	3	Positive
O_2	4	Positive

Note that for the named gases the response of the Gas Check 5000 series after zeroing will be only permitted in the same sense as for that named gas, ie positive for helium, negative for R134a.

4.2.3 Other display symbols

Small display indicators show zeroing, nozzle fitted, peak hold and low battery voltage.

Zeroing — This symbol appears whilst the Gas Check is zeroing then it disappears.



Nozzle — This symbol indicates that the Gas Check is being used with the Nozzle on.



Peak hold — This symbol indicates that the Gas Check display is held to the maximum.



Low battery — This symbol indicates that the battery pack is lower than required for use.



Alarm bell — This symbol shows that the alarm level has been set up for use.



ABC — This symbol shows that the Automatic Background Compensation is applied



Auto Range — This symbol shows when the Gas Check leak rate is not in a fixed range.



Data Logging — This symbol shows continuous Data Logging is in progress



Data Logged — This symbol shows continuous Data Logging was completed (each dash =10%)



Audio — This shows that the audible/visual leak rate indication is ON



Exponent — This symbol lies below the leak range exponent in the bar graph mode



Negative — This shows the leak rate is negative

Bar graph — The leak reading shown below is 2 x 10- (Exponent range shown)



4.3 Audio and visual outputs

The Gas Check 5000 series have an internal audio sounder and a flashing LED indicator.
They both operate together when the detected leak rate climbs from the zero indication towards the maximum on the leak rate range (see Section 4.2.1). They also operate once to confirm a key press.

4.4 Prepare the Gas Check

4.4.1 Select which probe to use

Note: Whenever possible, use the Gas Check with the nozzle fitted. The nozzle helps to protect the short probe from blockage or damage. You cannot use the nozzle with the long probe.

Open the storage case and remove the Gas Check. The Gas Check is supplied with the short probe fitted and the nozzle fitted; a long probe is supplied in the storage case. Determine whether you want to use the short probe or the long probe and whether you want the nozzle fitted, as follows:

- Use the Gas Check with the short probe and without the nozzle to detect small leak rates of approximately 1×10^{-4} ml/sec and lower. Press the nozzle key to clear the nozzle fitted indicator: refer to Section 4.1

- For large leaks, use the Gas Check with the short probe and the nozzle fitted. The nozzle extends the detection range for more accurate measurement of large leaks. Press the nozzle key to set the nozzle fitted indicator: refer to Section 4.1.

- If the area you want to leak check has restricted access, remove the nozzle and the short probe and fit the long probe. The long probe is flexible and can be bent in a gentle arc so that it can reach areas that would otherwise be difficult to reach. Press the nozzle button to tell the Gas Check that the nozzle is not fitted when you fit the long probe: refer to Section 4.1/ Note that response time increases significantly when the long probe is fitted (see the note in Section 4.6.1).

The nozzle simply pulls off and pushes onto the end of the Gas Check. To remove the nozzle or to change the probe, refer to Section 4.4.2.

4.4.2 Remove the Gas Check nozzle or change the probe (if required)

1 Switch off the Gas Check.

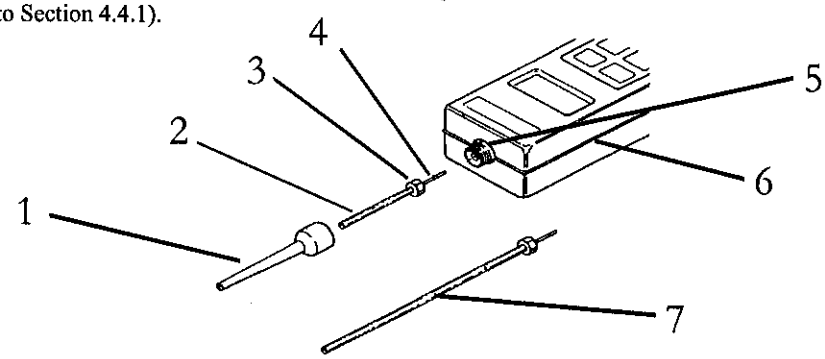
2 Refer to Figure 5. If the nozzle is fitted, hold the nozzle (1) by its wide base and gently twist and pull it to remove it from the Gas Check.

3 With the small end of the box spanner towards the Gas Check, guide the end of the probe (2 or 7) through the middle of the box spanner. Push the box spanner onto the nut (3) and turn the box spanner anticlockwise to undo and remove the probe.

4 Push the probe insert (4) of the other probe into the sample hole in the probe/nozzle fitting (5), then tighten the nut (3) one or two turns by hand to loosely secure the probe.

5 With the small end of the box spanner towards the Gas Check, guide the end of the probe through the middle of the box spanner. Push the box spanner onto the nut (3) and turn the box spanner clockwise to secure the probe on the Gas Check. Tighten the nut by hand only, but do ensure that the metal part of the capillary tube (2) under the plastic sheath is not loose – else a noisy detected signal will result.

6 If you have fitted the short probe to the Gas Check, push the nozzle onto the Gas Check if required (refer to Section 4.4.1).



1	Nozzle	5	Probe/nozzle fitting
2	Short probe	6	Gas Check
3	Nut	7	Long probe
4	Probe insert		

Figure 5 – Change the probe

4.5 Switch on the Gas Check

Press I/O key and wait for Gas Check to bleep & flash, the LCD to light up and show

Ion Science Ltd
Cambridge
England

Then show

Gascheck 5000
SW Version 1.49
Text Version 1.00

Then show

Zeroing Detector

After a short while the LCD display will then revert to the display format previously set up for the leak rate, which can be either the bar graph or the digital display, the latter in normal or large numeric display format.

When not detecting leaking gas, Gas Check still flashes and bleeps periodically to indicate that it is ON. Gas Check remembers the settings on instrument power off so that the operator rarely if ever has to alter any of them. The settings for gas groups, range, audio bleep, peak hold and nozzle are shown on the LCD.

The response of Gas Check can readily be checked against an Ion Science Portable Reference Leak (PRL). Alternatively the tracer gas supply can be suitably fitted with an Ion Science Ltd Standard Calibrated Leak.

4.6 Recommended leak search procedure

4.6.1 General procedure

Note: When you use the Gas Check, do not allow dirt or debris to enter the end of the probe or nozzle. If the probe or nozzle is blocked, the Gas Check will not operate correctly.

The following test procedures assume that you have fitted the short probe. The response time of the Gas Check increases from about 1 second or less when the short probe is fitted to about 9 seconds when the long probe is fitted. If you use the long probe for leak detection, ensure that you move the end of the probe much more slowly along the area you leak test.

Ensure the unit under test contains a reasonable percentage of a trace gas that is different from normal air!

Move the end of the probe (or nozzle) around the area of a suspected leak and look at the display (or listen to the audible clicks or note the visual LED flash rate on the Gas Check) to identify the location of a leak. Specific procedures for leak detection along seams and around pipe joints are described in the following sections.

When required:

- Use the peak hold and Auto-Ranging modes to continue to check for leaks, but to display the largest leak detected

- If you want to zero the Gas Check, move the end of the probe (or nozzle) away from any possible leak, into a clean air zone, then press the manual zero key: refer to Section 4.1.

Note that leak sensitivity depends on the ambient conditions. If you want to accurately measure a leak, compare the indicated leak with a calibration standard, such as a portable reference leak: see Section 7.3.

When the Gas Check indicates that a leak has been found, move the probe (or nozzle) back to determine the precise location of the leak. The leak is located where the largest leak size is indicated on the display and where highest frequency clicks and visual flashing rate are emitted by the Gas Check, on the current range.

4.6.2 Leak detection along seams

Move the end of the Gas Check probe (or nozzle) along the length of the seam, so that it just rests on the seam. The speed at which you move along the seam depends upon the size of the leak that you search for:

- If the leak is large, you can move fairly fast along the seam, at approximately 25 mm per second.

- If the leak is small, move slowly along the seam, at approximately 10 mm per second.

4.6.3 Leak detection at pipe joints

Use the same procedure as for leak detection along seams (see Section 4.6.2), however move the probe (or nozzle) around the joint at approximately half the speed specified in Section 4,.6.2. Light gases (such as hydrogen and helium) diffuse quickly into air, so an apparent leak on the side or top of a joint may actually be a leak at the bottom of the joint.

4.6.4 Data logging – interruption for menu access

Gas Check 5000 settings are protected during data logging. Press the INFO key to enter a menu dialogue.

4.7 Switch off the Gas Check

When the Gas Check 5000 has been left for a few seconds without any key press, then press the I/O key. The Gas Check will ignore I/O presses during the instrument power up sequence and during any zeroing.

4.7.1 Auto Shut Down

The unit will auto shut down after approximately 10 minutes.

5 MAINTENANCE

5.1 Inspect and clean the Gas Check

> CAUTION
> Do not clean the probe while it is attached to the Gas Check and do not try to clean the sample hole inside the probe/nozzle fitting which leads to the detector. If you do, you can damage the Gas Check.

1. Switch off the Gas Check.

2. Refer to Figure 5. If the nozzle is fitted, hold the nozzle (1) by its wide base and gently twist and pull it to remove it from the Gas Check.

3. With the small end of the box spanner towards the Gas Check, guide the end of the probe (2 or 7) through the middle of the box spanner. Push the box spanner onto the nut (3) and turn the box spanner anticlockwise to undo and remove the probe.

4. Inspect the Gas Check. If the case of the Gas Check is cracked is cracked or dented, we recommend that you do not continue maintenance, but return the Gas Check to your supplier for inspection and calibration.

5. Wipe the Gas Check with a clean, lint-free cloth moistened with a weak solution of detergent.

6. Carefully wipe the outer faces of the probe/nozzle fitting to remove any small particles of dirt or debris.

7. Use a suitable tool to push any blockage out of the end of the nozzle, then wipe the outside of and the inner face of the wide part of the nozzle.

8. Inspect the probe:
   - The hole in the short probe is approximately 0.47mm (0.0185inches) in diameter. We recommend that you push stiff wire of 0.25mm (0.01inches) diameter or less into the hole in the probe to remove any blockage.
   - The long probe is too long to be effectively cleaned. If the probe is blocked, we recommend that you obtain a new probe, available as a spare: refer to section 7.

9. Refit the required probe to the Gas Check: refer to Steps 4 to 6 in Section 4.4.2.

10. Place a light wipe of thin petroleum jelly around the inner face of the wide part of the nozzle, then fit the nozzle to the Gas Check.

11. Place the Gas Check and nozzle (and the long probe, if just removed) in its storage case. Store the case in suitable conditions: refer to Section 6.1.

5.2 Replace the batteries

> WARNING
> Replace the batteries in the Gas Check 5000is in a safe area where there are no flammable gases. If you do not, there may be a risk of fire or explosion.

> WARNING
> Use only dry cell (non-rechargeable) batteries in the Gas Check 5000is. Recommended batteries are specified in Section 2.3 Battery Data.

CAUTION

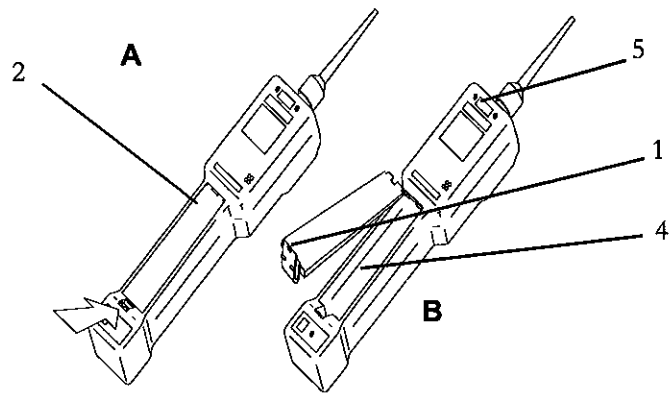
The Gas Check 5000is will blow a factory fitted internal protection fuse if the battery clip is reverse connected. The complete instrument will have to be returned to the supplier for fuse replacement.

Ensure that the rubber sleeve on the female part of the battery holder is correctly fitted when you fit new batteries. If you do not, you could refit the holder in the Gas Check 5000is with incorrect polarity, which will blow an internal fuse and make it inoperative.

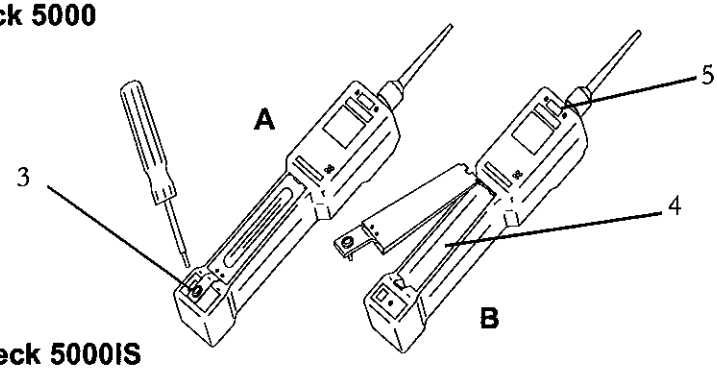
The Gas Check 5000 has an automatic re-settable fuse which will not be damaged.

Note: A spare battery holder is supplied in the case.

1. Refer to Figure 6. Turn over the Gas Check so that the battery compartment is at the top: see detail A.



Gas Check 5000



Gas Check 5000IS

1	Catch (5000is only)	4	Battery compartment
2	Battery compartment cover	5	Serial number
3	Captive screw (5000is only)		

Figure 6 – Replace the batteries (Gas Check 5000 and 5000is)

2. On the Gas Check 5000, use a small coin, screwdriver or your fingernail to press the catch (1) on the battery compartment cover (2), then pull up and remove the cover: see detail B.

   On the Gas Check 5000is, use the special screwdriver supplied to undo the captive screw on the batter compartment cover and remove the cover. This is located at the end of the Gas Check at position (5).

3. Remove the battery holder. If required, you can carefully unplug the battery holder so that you can detach the holder from the Gas Check.

4. Remove the old batteries (4) from the battery holder. Dispose of the batteries (see section 6.2).

5. Inspect the inside of the battery compartment and battery holder. If the batteries have leaked, use a cloth dampened with a weak solution of detergent to clean the battery compartment and battery holder.

> CAUTION
> The Gas Check 5000is will blow a factory fitted internal protection fuse if the battery clip is inadvertently reverse connected. The complete instrument will have to be returned to the supplier for fuse replacement.

6. Fit four new batteries in the battery holder. Ensure that you fit the batteries in the correct orientation. Refit the battery holder to the Gas Check.

7. On Gas Check 5000, refit the battery compartment cover (2) and ensure that the catch (1) is engaged.

   On the Gas Check 5000is, refit the battery compartment cover and use the special screwdriver to tighten the captive screw and secure the cover in place.

> CAUTION
> Ensure that the screw is located squarely to the threaded insert before tightening else the battery compartment cover may not be properly secured due to cross threading. The instrument will then have to be returned to the supplier for replacement of the Gas Check case and new serial number.

8. Switch on the Gas Check to ensure that the batteries are correctly fitted. If the display remains blank when you switch on the Gas Check, refer to section 5.3.

5.3 Fault finding

Fault symptom	Diagnosis	Action
Unit will not switch on	No/dead batteries fitted	Try another battery set (safely)
Unit will not switch off	Software locked out	Remove & replace battery clip (safely)
Battery symbol appears	Battery state low	Replace batteries (safely)
Back light drains battery life	Excessive use of backlight	Avoid or reduce use of backlight
Unit will not zero	Software locked out by hardware	Remove & replace batteries (safely)
LCD appears blank/dim	Contrast control setting wrong	Turn off & on; press & hold contrast
Bar graph/number do not change	Peak hold selected	Press zero or change Peak Hold state
Range does not change	Range not set in Auto-range	Press & hold Range until Auto-range
He range will not show –5	Unit with nozzle ON and/or range	Reset via Nozzle and Range keys
Gas group is wrong	Gas Group not set correctly	Press & hold Gases until shown OK
Nozzle/Peak not showing	Nozzle/Peak is not set correctly	Press & hold Nozzle until both correct
Calibration due message	Unit has exceeded 1 year on cal	Return to Ion Science for recalibration
Unit will not zero correctly	Sensor contaminated	Leave to stabilise
Blocked probe	Probe blocked by solid or liquid	Remove probe; clean; check now OK
Stalled motor	Motor failed & probe not blocked	Return to supplier for repair
Instrument readings noisy	Loose probe	Tighten nut slightly
Instrument readings noisy	Test environment contaminated	Move test into clean area
5000is will not switch on	Internal fuse blown	Return to supplier for repair

Table 5 – Fault finding

6 STORAGE AND DISPOSAL

6.1 Storage

If you will store the Gas Check for a long time, we recommend that you remove the batteries: refer to Section 5.2

Refit the Gas Check in its storage case and store in dry, cool conditions. When required for use, unpack as described in Section 3.

6.2 Disposal

Dispose of the Gas Check, components and used batteries safely in accordance with all local and national safety and environmental requirements. Some of the Gas Check carry case plastic material is recyclable.

7 SPARES AND ACCESSORIES

7.1 Introduction

Order spare parts from your local agent or distributor. When you order, please state for each part required:

- Serial Number stamped on your Gas Check - refer to Figure 6.
- Item Number and description of part.

7.2 Spares

Spare	Item Number
Long probe	A-8045
Short probe	A-8043
Replacement battery holder	1/BH-01
Nozzle 5000	08024CFC0
Nozzle 5000is	08024CPC8
Box spanner	3/BI-01
5000is screwdriver	A-8048
Case	28637C
Manual	SPGCMAN5K

7.3 Accessories

Accessories	Item Number
Portable reference leak	PRL
-Specifying: Gas, Leak rate and pressure	
IR Data Link	

7.4 Calibration

Ion Science offer a calibration service including issue of traceable certification to National Standards.

7.5 Refurbish program

Ion Science offer a refurbishment service for all customer repairs

Attachment H

U.S. EPA Compendia of Emergency Response Team (ERT) Sampling Procedures

GROUNDWATER WELL SAMPLING

SOP#: 2007
DATE: 01/26/95
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The objective of this standard operating procedure (SOP) is to provide general reference information on sampling of ground water wells. This guideline is primarily concerned with the collection of water samples from the saturated zone of the subsurface. Every effort must be made to ensure that the sample is representative of the particular zone of water being sampled. These procedures are designed to be used in conjunction with analyses for the most common types of ground water contaminants (e.g., volatile and semi-volatile organic compounds, pesticides, metals, biological parameters).

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

In order to obtain a representative groundwater sample for chemical analysis it is important to remove stagnant water in the well casing and the water immediately adjacent to the well before collection of the sample. This may be achieved with one of a number of instruments. The most common of these are the bailer, submersible pump, non-contact gas bladder pump, inertia pump and suction pump. At a minimum, three well volumes should be purged, if possible. Equipment must be decontaminated prior to use and between wells. Once purging is completed and the correct laboratory-cleaned sample containers have been prepared, sampling may proceed. Sampling may be conducted with any of the above instruments, and need not be the same as the device used for purging. Care should be taken when choosing the sampling device as some will affect the integrity of the sample. Sampling should occur in a progression from the least to most contaminated well, if this information is known.

The growing concern over the past several years over low levels of volatile organic compounds in water supplies has led to the development of highly sophisticated analytical methods that can provide detection limits at part per trillion levels. While the laboratory methods are extremely sensitive, well controlled and quality assured, they cannot compensate for a poorly collected sample. The collection of a sample should be as sensitive, highly developed and quality assured as the analytical procedures.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The type of analysis for which a sample is being collected determines the type of bottle, preservative, holding time, and filtering requirements. Samples should be collected directly from the sampling device into appropriate laboratory cleaned containers. Check that a Teflon liner is present in the cap, if required. Attach a sample identification label. Complete a field data sheet, a chain of custody form, and record all pertinent data in the site logbook.

Samples shall be appropriately preserved, labelled, logged, and placed in a cooler to be maintained at 4EC. Samples must be shipped well before the holding time is up and ideally should be shipped within 24 hours of sample collection. It is imperative that samples be shipped or delivered daily to the analytical laboratory in order to maximize the time available for the laboratory to perform the analyses. The bottles should be shipped with adequate packing and cooling to ensure that they arrive intact.

Sample retrieval systems suitable for the valid collection of volatile organic samples are: positive displacement bladder pumps, gear driven submersible pumps, syringe samplers and bailers (Barcelona, 1984; Nielsen, 1985). Field conditions and other constraints will limit the choice of appropriate systems. The focus of concern must remain to provide a valid sample for analysis, one which has been subjected to the least amount of turbulence possible.

Treatment of the sample with sodium thiosulfate preservative is required only if there is residual chlorine in the water that could cause free radical chlorination and change the identity of the original contaminants. It should not be used if there is no chlorine in the water.

Holding time for volatiles analysis is seven days. It is imperative that the sample be shipped or delivered daily to the analytical laboratory. The bottles must be shipped on their sides to aid in maintaining the airtight seal during shipment, with adequate packing and cooling to ensure that they arrive intact.

For collection of volatile organic samples, refer to the work plan to ensure that 40 mL glass sample vials with Teflon lined septa are ordered and in sufficient numbers. Check sampling supplies; field kit for chlorine, preservatives, Parafilm, foam sleeves and coolers. Due to the extreme trace levels at which volatile organics are detectable, cross contamination and introduction of contaminants must be avoided. Trip blanks are incorporated into the shipment package to provide a check against cross contamination.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 General

The primary goal in performing ground water sampling is to obtain a representative sample of the ground water body. Analysis can be compromised by field personnel in two primary ways: (1) taking an unrepresentative sample, or (2) by incorrect handling of the sample. There are numerous ways of introducing foreign contaminants into a sample, and these must be avoided by following strict sampling procedures and utilizing trained field personnel.

4.2 Purging

In a nonpumping well, there will be little or no vertical mixing of the water, and stratification will occur. The well water in the screened section will mix with the ground water due to normal flow patterns, but the well water above the screened section will remain isolated, become stagnant, and may lack the contaminants representative of the ground water. Persons sampling should realize that stagnant water may contain foreign material inadvertently or deliberately introduced from the surface, resulting in an unrepresentative sample. To safeguard against collecting nonrepresentative stagnant water, the following guidelines and techniques should be adhered to during sampling:

1. As a general rule, all monitor wells should be pumped or bailed prior to sampling. Purge water should be containerized on site or handled as specified in the site specific project plan. Evacuation of a minimum of one volume of water in the well casing, and preferably three to five volumes, is recommended for a representative sample. In a high-yielding ground water formation and where there is no stagnant water in the well above the screened section, evacuation prior to sample withdrawal is not as critical. However, in all cases where the monitoring data is to be used for enforcement actions, evacuation is recommended.

2. When purging with a pump (not a bailer), the pump should be set at the screened interval, or if the well is an open-rock well, it should be set at the same depth the sample will be collected. When sampling a screened well, the sample should also be collected from the same depth the pump was set at.

3. The well should be sampled as soon as possible after purging.

4. Analytical parameters typically dictate whether the sample should be collected through the purging device, or through a separate sampling instrument.

5. For wells that can be pumped or bailed to dryness with the equipment being used, the well should be evacuated and allowed to recover prior to collecting a sample. If the recovery rate is fairly rapid and time allows, evacuation of more than one volume of water

is preferred. If recovery is slow, sample the well upon recovery after one evacuation.

6. A non-representative sample can also result from excessive pre-pumping of the monitoring well. Stratification of the leachate concentration in the ground water formation may occur, or heavier-than-water compounds may sink to the lower portions of the aquifer. Excessive pumping can dilute or increase the contaminant concentrations from what is representative of the sampling point of interest.

4.3 Materials

Materials of construction for samplers and evacuation equipment (bladders, pump, bailers, tubing, etc.) should be limited to stainless steel, Teflon^{R}, and glass in areas where concentrations are expected to be at or near the detection limit. The tendency of organics to leach into and out of many materials make the selection of materials critical for trace analyses. The use of plastics, such as PVC or polyethylene, should be avoided when analyzing for organics. However, PVC may be used for evacuation equipment as it will not come in contact with the sample, and in highly contaminated wells, disposable equipment (i.e., polypropylene bailers) may be appropriate to avoid cross-contamination.

Materials of construction (bladders/ pumps, bailers, tubing, etc.) suitable for collecting and handling Volatile Organic Samples should be limited to stainless steel, Teflon and glass in areas which detection limit range concentrations are expected. The tendency of organics to leach into and out of many materials, make the selection of materials critical for these trace analyses. The use of plastics, e.g., PVC etc., should be avoided. There are numerous ways of introducing foreign contaminants into a sample, and these must be avoided by following strict sampling procedures and utlization of trained personnel.

4.4 Advantages/Disadvantages of Certain Equipment

4.4.1 Bailers

Advantages

C Only practical limitations on size and materials

C No power source needed

C Portable

C Inexpensive, so it can be dedicated and hung in a well, thereby reducing the chances of cross contamination

C Minimal outgassing of volatile organics while sample is in bailer

C Readily available

C Removes stagnant water first

C Rapid, simple method for removing small volumes of purge water

Disadvantages

C Time-consuming to flush a large well of stagnant water

C Transfer of sample may cause aeration

C Stoppers at the bottom of the bailer usually leak thus the bailer must be brought to the surface rapidly

C If the bailer is allowed to hit the bottom of the well boring, gravel can displace the ball valve not allowing the bailer to hold water

4.4.2 Submersible Pumps

Advantages

C Portable and can be transported to several wells

C Depending upon the size of the pump and the pumping depths, relatively high pumping rates are possible

C Generally very reliable and does not require priming

Disadvantages

- Potential for effects on analysis of trace organics
- Heavy and cumbersome to deal with, particularly in deeper wells
- Expensive
- Power source needed
- Sediment in water may cause problems with the pumps
- Impractical in low yielding or shallow wells

4.4.3 Non-Contact Gas Bladder Pumps

Advantages

- Maintains integrity of sample
- Easy to use
- Can sample from discrete locations within the monitor well

Disadvantages

- Difficulty in cleaning, though dedicated tubing and bladder may be used
- Only useful to about 100 feet
- Supply of gas for operation, gas bottles and/or compressors are often difficult to obtain and are cumbersome
- Relatively low pumping rates
- Requires air compressor or pressurized gas source and control box

4.4.4 Suction Pumps

Advantages

- Portable, inexpensive, and readily available

Disadvantages

- Restricted to areas with water levels within 20 to 25 feet of the ground surface
- Vacuum can cause loss of dissolved gasses and volatile organics
- Pump must be primed and vacuum is often difficult to maintain during initial stages of pumping

4.4.5 Inertia Pumps

Advantages

- Portable, inexpensive, and readily available
- Offers a rapid method for purging relatively shallow wells

Disadvantages

- Restricted to areas with water levels within 70 feet of the ground surface
- May be time consuming to purge wells with these manual pumps
- Labor intensive
- WaTerra pumps are only effective in 2-inch diameter wells

5.0 EQUIPMENT APPARATUS

5.1 Equipment Checklist

5.1.1 General

- Water level indicator
  - electric sounder
  - steel tape
  - transducer
  - reflection sounder
  - airline
- Depth sounder
- Appropriate keys for well cap locks
- Steel brush
- HNU or OVA (whichever is most appropriate)
- Logbook
- Calculator
- Field data sheets and samples labels
- Chain of custody records and seals
- Sample containers
- Engineer's rule

- C Sharp knife (locking blade)
- C Tool box (to include at least: screwdrivers, pliers, hacksaw, hammer, flashlight, adjustable wrench)
- C Leather work gloves
- C Appropriate Health & Safety gear
- C 5-gallon pail
- C Plastic sheeting
- C Shipping containers
- C Packing materials
- C Bolt cutters
- C Ziploc plastic bags
- C Containers for evacuation liquids
- C Decontamination solutions
- C Tap water
- C Non phosphate soap
- C Several brushes
- C Pails or tubs
- C Aluminum foil
- C Garden sprayer
- C Preservatives
- C Distilled or deionized water
- C Fire extinguisher (if using a generator for your power source)

5.1.2 Bailers

- C Clean, decontaminated bailers of appropriate size and construction material
- C Nylon line, enough to dedicate to each well
- C Teflon coated bailer wire
- C Sharp knife
- C Aluminum foil (to wrap clean bailers)
- C Five gallon bucket

5.1.3 Submersible Pump

- C Pump(s)
- C Generator (110, 120, or 240 volt) or 12 volt battery if inaccessible to field vehicle - amp meter is useful
- C 1" black PVC coil tubing - enough to dedicate to each well
- C Hose clamps
- C Safety cable
- C Tool box supplement
  - pipe wrenches
  - wire strippers
  - electrical tape
  - heat shrink
  - hose connectors
  - Teflon tape
- C Winch, pulley or hoist
- C Gasoline for generator/gas can
- C Flow meter with gate valve
- C 1" nipples and various plumbing (i.e., pipe connectors)
- C Control box (if necessary)

5.1.4 Non-Gas Contact Bladder Pump

- C Non-gas contact bladder pump
- C Compressor or nitrogen gas tank
- C Batteries and charger
- C Teflon tubing - enough to dedicate to each well
- C Swagelock fitting
- C Toolbox supplements - same as submersible pump
- C Control box (if necessary)

5.1.5 Suction Pump

- C Pump
- C 1" black PVC coil tubing - enough to dedicate to each well
- C Gasoline - if required
- C Toolbox
- C Plumbing fittings
- C Flow meter with gate valve

5.1.6 Inertia Pump

- C Pump assembly (WaTerra pump, piston pump)
- C Five gallon bucket

6.0 REAGENTS

Reagents may be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed. Decontamination solutions are specified in ERT SOP #2006, Sampling Equipment Decontamination.

7.0 PROCEDURE

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and

supplies needed (i.e, diameter and depth of wells to be sampled).

2. Obtain necessary sampling and monitoring equipment, appropriate to type of contaminant being investigated. For collection of volatile organic samples, refer to the work plan to ensure that 40 mL glass sample vials with Teflon lined septa are ordered and in sufficient numbers. Check sampling supplies; field kit for chlorine, preservatives, Parafilm, foam sleeves and coolers. Due to extreme trace levels at which volatile organics are detectable, cross contamination and introduction of contaminants must be avoided. Trip blanks are incorporated into the shipment package to provide a check against cross contamination.

3. Decontaminate or preclean equipment, and ensure that it is in working order.

4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Identify and mark all sampling locations.

7.2 Field Preparation

1. Start at the least contaminated well, if known.

2. Lay plastic sheeting around the well to minimize likelihood of contamination of equipment from soil adjacent to the well.

3. Remove locking well cap, note location, time of day, and date in field notebook or appropriate log form.

4. Remove well casing cap.

5. Screen headspace of well with an appropriate monitoring instrument to determine the presence of volatile organic compounds and record in site logbook.

6. Lower water level measuring device or equivalent (i.e., permanently installed transducers or airline) into well until water surface is encountered.

7. Measure distance from water surface to reference measuring point on well casing or protective barrier post and record in site logbook. Alternatively, if no reference point, note that water level measurement is from top of steel casing, top of PVC riser pipe, from ground surface, or some other position on the well head.

   If floating organics are of concern, this can be determined by measuring the water level with an oil/water interface probe which measures floating organics.

8. Measure total depth of well (at least twice to confirm measurement) and record in site logbook or on field data sheet.

9. Calculate the volume of water in the well and the volume to be purged using the calculations in Section 8.0.

10. Select the appropriate purging and sampling equipment.

11. If residual chlorine is suspected, use the Hach Field Test Kit for chlorine to determine if there is residual chlorine in the water to be sampled. If there is, treat the sample vial with a crystal of sodium thiosulfate prior to sample collection.

7.3 Purging

The amount of flushing a well receives prior to sample collection depends on the intent of the monitoring program as well as the hydrogeologic conditions. Programs where overall quality determination of water resources are involved may require long pumping periods to obtain a sample that is representative of a large volume of that aquifer. The pumped volume can be determined prior to sampling so that the sample is a collected after a known volume of the water is evacuated from the aquifer, or the well can be pumped until the stabilization of parameters such as temperature, electrical conductance, pH, or turbidity has occurred.

However, monitoring for defining a contaminant plume requires a representative sample of a small volume of the aquifer. These circumstances require that the well be pumped enough to remove the stagnant water but not enough to induce flow from other areas. Generally, three well volumes are considered effective, or calculations can be made to determine, on the basis of the aquifer parameters and well dimensions, the appropriate volume to remove prior to sampling.

During purging, water level measurements may be taken regularly at 15-30 second intervals. This data may be used to compute aquifer transmissivity and other hydraulic characteristics. The following well evacuation devices are most commonly used. Other evacuation devices are available, but have been omitted in this discussion due to their limited use.

7.3.1 Bailers

Bailers are the simplest purging device used and have many advantages. They generally consist of a rigid length of tube, usually with a ball check-valve at the bottom. A line is used to lower the bailer into the well and retrieve a volume of water. The three most common types of bailer are PVC, Teflon, and stainless steel.

This manual method of purging is best suited to shallow or narrow diameter wells. For deep, larger diameter wells which require evacuation of large volumes of water, other mechanical devices may be more appropriate.

7.3.1.1 Operation

Equipment needed will include a clean decontaminated bailer, Teflon or nylon line, a sharp knife, and plastic sheeting.

1. Determine the volume of water to be purged as described in 8.0, calculations.

2. Lay plastic sheeting around the well to prevent contamination of the bailer line with foreign materials.

3. Attach the line to the bailer and slowly lower until the bailer is completely submerged, being careful not to drop the bailer to the water, causing turbulence and the possible loss of volatile organic contaminants.

4. Pull bailer out ensuring that the line either falls onto a clean area of plastic sheeting or never touches the ground.

5. Empty the bailer into a pail until full to determine the number of bails necessary to achieve the required purge volume.

6. Thereafter, pour the water into a container and dispose of purge waters as specified in the site specific sampling plan.

7.3.2 Submersible Pumps

The use of submersible pumps for sample collection is permissible provided they are constructed of suitably noncontaminating materials. The chief drawback, however, is the difficulty avoiding cross-contamination between wells. Although some units can be disassembled easily to allow surfaces contacted by contaminants to be cleaned, field decontamination may be difficult and require solvents that can affect sample analysis. The use of submersible pumps in multiple well-sampling programs, therefore, should be carefully considered against other sampling mechanisms (bailers, bladder pumps). In most cases, a sample can be collected by bailer after purging with a submersible pump, however, submersible pumps may be the only practical sampling device for extremely deep wells (greater than 300 feet of water). Under those conditions, dedicated pump systems should be installed to eliminate the potential for cross-contamination of well samples.

Submersible pumps generally use one of two types of power supplies, either electric or compressed gas or air. Electric powered pumps can run off a 12 volt DC rechargeable battery, or a 110 or 220 volt AC power supply. Those units powered by compressed air normally use a small electric or gas-powered air compressor. They may also utilize compressed gas (i.e., nitrogen) from bottles. Different size pumps are available for different depth or diameter monitoring wells.

7.3.2.1 Operation

1. Determine the volume of water to be purged as described in 8.0 Calculations.

2. Lay plastic sheeting around the well to prevent contamination of pumps, hoses or lines with foreign materials.

3. Assemble pump, hoses and safety cable, and lower the pump into the well. Make sure the pump is deep enough so all the water is not evacuated. (Running the pump without water may cause damage.)

4. Attach flow meter to the outlet hose to measure the volume of water purged.

5. Use a ground fault circuit interrupter (GFCI) or ground the generator to avoid possible electric shock.

6. Attach power supply, and purge the well until the specified volume of water has been

evacuated (or until field parameters, such as temperature, pH, conductivity, etc, have stabilized). Do not allow the pump to run dry. If the pumping rate exceeds the well recharge rate, lower the pump further into the well, and continue pumping.

7. Collect and dispose of purge waters as specified in the site specific sampling plan.

7.3.3 Non-Contact Gas Bladder Pumps

For this procedure, an all stainless-steel and Teflon Middleburg-squeeze bladder pump (e.g., IEA, TIMCO, Well Wizard, Geoguard, and others) is used to provide the least amount of material interference to the sample (Barcelona, 1985). Water comes into contact with the inside of the bladder (Teflon) and the sample tubing, also Teflon, that may be dedicated to each well. Some wells may have permanently installed bladder pumps, (i.e., Well Wizard, Geoguard), that will be used to sample for all parameters.

7.3.3.1 Operation

1. Assemble Teflon tubing, pump and charged control box.

2. Procedure for purging with a bladder pump is the same as for a submersible pump (Section 7.3.2.1).

3. Be sure to adjust flow rate to prevent violent jolting of the hose as sample is drawn in.

7.3.4 Suction Pumps

There are many different types of suction pumps. They include: centrifugal, peristaltic and diaphragm. Diaphragm pumps can be used for well evacuation at a fast pumping rate and sampling at a low pumping rate. The peristaltic pump is a low volume pump that uses rollers to squeeze the flexible tubing thereby creating suction. This tubing can be dedicated to a well to prevent cross contamination. Peristaltic pumps, however, require a power source.

7.3.4.1 Operation

1. Assembly of the pump, tubing, and power source if necessary.

2. Procedure for purging with a suction pump is exactly the same as for a submersible pump (Section 7.3.2.1).

7.3.5 Inertia Pumps

Inertia pumps such as the WaTerra pump and piston pump, are manually operated. They are most appropriate to use when wells are too deep to bail by hand, or too shallow or narrow (or inaccessible) to warrant an automatic (submersible, etc.) pump. These pumps are made of plastic and may be either decontaminated or discarded.

7.3.5.1 Operation

1. Determine the volume of water to be purged as described in 8.0, Calculations.

2. Lay plastic sheeting around the well to prevent contamination of pumps or hoses with foreign materials.

3. Assemble pump and lower to the appropriate depth in the well.

4. Begin pumping manually, discharging water into a 5 gallon bucket (or other graduated vessel). Purge until specified volume of water has been evacuated (or until field parameters such as temperature, pH, conductivity, etc. have stabilized).

5. Collect and dispose of purge waters as specified in the site specific project plan.

7.4 Sampling

Sample withdrawal methods require the use of pumps, compressed air, bailers, and samplers. Ideally, purging and sample withdrawal equipment should be completely inert, economical to manufacture, easily cleaned, sterilized, reusable, able to operate at remote sites in the absence of power resources, and capable of delivering variable rates for sample collection.

There are several factors to take into consideration when choosing a sampling device. Care should be taken when reviewing the advantages or disadvantages of any one device. It may be appropriate to use a different device to sample than that which was used to purge. The most common example of this is the use of a submersible pump to purge and a bailer to sample.

7.4.1 Bailers

The positive-displacement volatile sampling bailer is perhaps the most appropriate for collection of water samples for volatile analysis. Other bailer types (messenger, bottom fill, etc.) are less desirable, but may be mandated by cost and site conditions.

7.4.1.1 Operation

1. Surround the monitor well with clean plastic sheeting. If using the GPI bailer, insert a vial into the claim and assemble the unit.

2. Attach a line to a clean decontaminated bailer.

3. Lower the bailer slowly and gently into the well, taking care not to shake the casing sides or to splash the bailer into the water. Stop lowering at a point adjacent to the screen.

4. Allow bailer to fill and then slowly and gently retrieve the bailer from the well avoiding contact with the casing, so as not to knock flakes of rust or other foreign materials into the bailer. If using the GPI bailer for collecting volatile organic samples, once at the surface, remove the bailer from the cable. Carefully open the GPI bailer unit and remove the vial. Begin slowly pouring from the bailer, and collect the duplicate samples from the midstream sample.

5. Remove the cap from the sample container and place it on the plastic sheet or in a location where it won't become contaminated. See Section 7.7 for special considerations on VOA samples.

6. Begin slowly pouring from the bailer.

7. Filter and preserve samples as required by sampling plan.

8. Cap the sample container tightly and place prelabeled sample container in a carrier.

9. Replace the well cap.

10. Log all samples in the site logbook and on field data sheets and label all samples.

11. Package samples and complete necessary

paperwork.

12. Transport sample to decontamination zone for preparation for transport to analytical laboratory.

7.4.2 Submersible Pumps

Although it is recommended that samples not be collected with a submersible pump due to the reasons stated in Section 4.4.2, there are some situations where they may be used.

7.4.2.1 Operation

1. Allow the monitor well to recharge after purging, keeping the pump just above screened section.

2. Attach gate valve to hose (if not already fitted), and reduce flow of water to a manageable sampling rate.

3. Assemble the appropriate bottles.

4. If no gate valve is available, run the water down the side of a clean jar and fill the sample bottles from the jar.

5. Cap the sample container tightly and place prelabeled sample container in a carrier.

6. Replace the well cap.

7. Log all samples in the site logbook and on the field data sheets and label all samples.

8. Package samples and complete necessary paperwork.

9. Transport sample to decontamination zone for preparation for transport to the analytical laboratory.

10. Upon completion, remove pump and assembly and fully decontaminate prior to setting into the next sample well. Dedicate the tubing to the hole.

7.4.3 Non-Contact Gas Bladder Pumps

The use of a non-contact gas positive displacement bladder pump is often mandated by the use of dedicated pumps installed in wells. These pumps are also suitable for shallow (less than 100 feet) wells. They are somewhat difficult to clean, but may be used with dedicated sample tubing to avoid cleaning. These pumps require a power supply and a compressed gas supply (or compressor). They may be operated at variable flow and pressure rates making them ideal for both purging and sampling.

Barcelona (1984) and Nielsen (1985) report that the non-contact gas positive displacement pumps cause the least amount of alteration in sample integrity as compared to other sample retrieval methods.

7.4.3.1 Operation

1. Allow well to recharge after purging.

2. Assemble the appropriate bottles.

3. Turn pump on, increase the cycle time and reduce the pressure to the minimum that will allow the sample to come to the surface.

4. Cap the sample container tightly and place prelabeled sample container in a carrier.

5. Replace the well cap.

6. Log all samples in the site logbook and on field data sheets and label all samples.

7. Package samples and complete necessary paperwork.

8. Transport sample to decontamination zone for preparation for transport to analytical laboratory.

9. On completion, remove the tubing from the well and either replace the Teflon tubing and bladder with new dedicated tubing and bladder or rigorously decontaminate the existing materials.

10. Nonfiltered samples shall be collected directly from the outlet tubing into the sample bottle.

11. For filtered samples, connect the pump outlet tubing directly to the filter unit. The pump

pressure should remain decreased so that the pressure build up on the filter does not blow out the pump bladder or displace the filter. For the Geotech barrel filter, no actual connections are necessary so this is not a concern.

7.4.4 Suction Pumps

In view of the limitations of these type pumps, they are not recommended for sampling purposes.

7.4.5 Inertia Pumps

Inertia pumps may be used to collect samples. It is more common, however, to purge with these pumps and sample with a bailer (Section 7.4.1).

7.4.5.1 Operation

1. Following well evacuation, allow the well to recharge.

2. Assemble the appropriate bottles.

3. Since these pumps are manually operated, the flow rate may be regulated by the sampler. The sample may be discharged from the pump outlet directly into the appropriate sample container.

4. Cap the sample container tightly and place prelabeled sample container in a carrier.

5. Replace the well cap.

6. Log all samples in the site logbook and on field data sheets and label all samples.

7. Package samples and complete necessary paperwork.

8. Transport sample to decontamination zone for preparation for transport to the analytical laboratory.

9. Upon completion, remove pump and decontaminate or discard, as appropriate.

7.4.6. Sample Retrieval - Syringe

A limited number of commercial syringe type samplers are available, (IEA, TIMCO, etc.) some are homemade devices. These devices are claimed to provide good quality samples for volatile analysis, but are severly limited in sample volume and are specific to sampling for volatiles. Essentially, they operated with an evacuated chamber that is lowered down the well, and allowed to fill with the pressure of the water. The entire mechanism is then brought to the surface with the sample. The sample may then be transferred to a sample vial, or the entire unit may be sent as the sample container.

1. Evacuate the syringe if necessary, and lower the sampling device to just below the well screen.

2. Remove the constriction from the device and allow the sample to fill the syringe, apply slight suction as necessary.

3. Bring unit to the surface. If necessary, transfer the sample to vials, as outlined in steps 2 through 7 above.

7.5 Filtering

For samples requiring filtering, such as total metals analysis, the filter must be decontaminated prior to and between uses. Filters work by two methods. A barrel filter such as the "Geotech" filter works with a bicycle pump, used to build up positive pressure in the chamber containing the sample which is then forced through the filter paper (minimum size 0.45 µm) into a jar placed underneath. The barrel itself is filled manually from the bailer or directly via the hose of the sampling pump. The pressure must be maintained up to 30 lbs/in^2 by periodic pumping.

A vacuum type filter involves two chambers; the upper chamber contains the sample and a filter (minimum size 0.45 µm) divides the chambers. Using a hand pump or a Gilian type pump, air is withdrawn from the lower chamber, creating a vacuum and thus causing the sample to move through the filter into the lower chamber where it is drained into a sample jar. Repeated pumping may be required to drain all the sample into the lower chamber. If preservation of the sample is necessary, this should be done after filtering.

7.6 Post Operation

After all samples are collected and preserved, the sampling equipment should be decontaminated prior to sampling another well to prevent cross-contamination of equipment and monitor wells between locations.

1. Decontaminate all equipment.

2. Replace sampling equipment in storage containers.

3. Prepare and transport ground water samples to the laboratory. Check sample documentation and make sure samples are properly packed for shipment.

7.7 Special Considerations for VOA Sampling

The proper collection of a sample for volatile organics requires minimal disturbance of the sample to limit volatilization and therefore a loss of volatiles from the sample.

Sample retrieval systems suitable for the valid collection of volatile organic samples are: positive displacement bladder pumps, gear driven submersible pumps, syringe samplers and bailers (Barcelona, 1984; Nielsen, 1985). Field conditions and other constraints will limit the choice of appropriate systems. The focus of concern must be to provide a valid sample for analysis, one which has been subjected to the least amount of turbulence possible.

The following procedures should be followed:

1. Open the vial, set cap in a clean place, and collect the sample during the middle of the cycle. When collecting duplicates, collect both samples at the same time.

2. Fill the vial to just overflowing. Do not rinse the vial, nor excessively overflow it. There should be a convex meniscus on the top of the vial.

3. Check that the cap has not been contaminated (splashed) and carefully cap the vial. Place the cap directly over the top and screw down firmly. Do not overtighten and break the cap.

4. Invert the vial and tap gently. Observe vial for at least ten (10) seconds. If an air bubble appears, discard the sample and begin again. It is imperative that no entrapped air is in the sample vial.

5. Immediately place the vial in the protective foam sleeve and place into the cooler, oriented so that it is lying on its side, not straight up.

6. The holding time for VOAs is seven days. Samples should be shipped or delivered to the laboratory daily so as not to exceed the holding time. Ensure that the samples remain at 4EC, but do not allow them to freeze.

8.0 CALCULATIONS

If it is necessary to calculate the volume of the well, utilize the following equation:

$$\textit{Well volume} \quad ' \quad nr^2h\ (cf) \qquad [\textit{Equation}\ 1]$$

where:

n	=	pi
r	=	radius of monitoring well (feet)
h	=	height of the water column (feet) [This may be determined by subtracting the depth to water from the total depth of the well as measured from the same reference point.]
cf	=	conversion factor (gal/ft^3) = 7.48 gal/ft^3 [In this equation, 7.48 gal/ft^3 is the necessary conversion factor.]

Monitor well diameters are typically 2", 3", 4", or 6". Knowing the diameter of the monitor well, there are a number of standard conversion factors which can be used to simplify the equation above.

The volume, in gallons per linear foot, for various standard monitor well diameters can be calculated as follows:

$$v(gal/ft) \quad ' \quad nr^2\ (cf) \qquad [\textit{Equation}\ 2]$$

where:

n	=	pi
r	=	radius of monitoring well (feet)
cf	=	conversion factor (7.48 gal/ft^3)

For a 2" diameter well, the volume per linear foot can be calculated as follows:

$$\textit{vol/linear ft} = nr^2 (cf) \quad [\textit{Equation } 2]$$
$$= 3.14\ (1/12\ ft)^2\ \ 7.48\ gal/ft^3$$
$$= 0.1632\ gal/ft$$

Remember that if you have a 2" diameter well, you must convert this to the radius in feet to be able to use the equation.

The conversion factors for the common size monitor wells are as follows:

Well diameter	2"	3"	4"	6"
Volume (gal/ft.)	0.1632	0.3672	0.6528	1.4688

If you utilize the conversation factors above, Equation 1 should be modified as follows:

$$\textit{Well volume} = (h)(cf) \quad [\textit{Equation } 3]$$

where:

h	=	height of water column (feet)
cf	=	the conversion factor calculated from Equation 2

The well volume is typically tripled to determine the volume to be purged.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following general QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented.

3. The collection of rinsate blanks is recommended to evaluate potential for cross contamination from the purging and/or sampling equipment.

4. Trip blanks are required if analytical parameters include VOAs.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA or REAC health and safety guidelines. More specifically, depending upon the site specific contaminants, various protective programs must be implemented prior to sampling the first well. The site health and safety plan should be reviewed with specific emphasis placed on the protection program planned for the well sampling tasks. Standard safe operating practices should be followed such as minimizing contact with potential contaminants in both the vapor phase and liquid matrix through the use of respirators and disposable clothing.

When working around volatile organic contaminants:

1. Avoid breathing constituents venting from the well.

2. Pre-survey the well head-space with an FID/PID prior to sampling.

3. If monitoring results indicate organic constituents, sampling activities may be conducted in Level C protection. At a minimum, skin protection will be afforded by disposable protective clothing.

Physical hazards associated with well sampling:

1. Lifting injuries associated with pump and bailers retrieval; moving equipment.

2. Use of pocket knives for cutting discharge hose.

3. Heat/cold stress as a result of exposure to extreme temperatures and protective clothing.

4. Slip, trip, fall conditions as a result of pump discharge.

5. Restricted mobility due to the wearing of protective clothing.

6. Electrical shock associated with use of submersible pumps is possible. Use a GFCI or a copper grounding stake to avoid this problem.

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U. S. EPA ENVIRONMENTAL RESPONSE TEAM

STANDARD OPERATING PROCEDURES

SOIL SAMPLING

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SUPERCEDES: SOP #2012; Revision 0.0; 11/16/94; U.S. EPA Contract 68-C4-0022.

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of representative soil samples. Sampling depths are assumed to be those that can be reached without the use of a drill rig, direct-push, or other mechanized equipment (except for a back-hoe). Analysis of soil samples may determine whether concentrations of specific pollutants exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or, if required, a backhoe.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Chemical preservation of solids is not generally recommended. Samples should, however, be cooled and protected from sunlight to minimize any potential reaction. The amount of sample to be collected and proper sample container type are discussed in ERT/REAC SOP #2003 Rev. 0.0 08/11/94, *Sample Storage, Preservation and Handling*.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

There are two primary potential problems associated with soil sampling - cross contamination of samples and improper sample collection. Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, disturbance of the matrix resulting in compaction of the sample, or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

5.0 EQUIPMENT

Soil sampling equipment includes the following:

- Maps/plot plan
- Safety equipment, as specified in the site-specific Health and Safety Plan
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan
- Appropriate size sample containers
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and custody seals
- Field data sheets and sample labels
- Cooler(s)
- Ice
- Vermiculite
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger
- Extension rods
- T-handle
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
  - Tubes
  - Points
  - Drive head
  - Drop hammer
  - Puller jack and grip
- Backhoe

6.0 REAGENTS

Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in ERT/REAC SOP #2006 Rev. 0.0 08/11/94, *Sampling Equipment Decontamination*, and the site specific work plan.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order.

4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations should be utility-cleared by the property owner or the On-Scene-Coordinator (OSC) prior to soil sampling; and utility clearance should always be confirmed before beginning work.

7.2 Sample Collection

7.2.1 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample.

This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed profiles are required. Tools plated with chrome or other materials should not be used. Plating is particularly common with garden implements such as potting trowels.

The following procedure is used to collect surface soil samples:

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.

2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.

3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix A). The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet.

The following procedure is used for collecting soil samples with the auger:

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole and proceed to Step 10.

5. Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.

7. Remove the tube sampler, and unscrew the drill rods.

8. Remove the cutting tip and the core from the device.

9. Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.

10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly.

    When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.

12. Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

7.2.3 Sampling with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth.

The following procedure is used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix A) into the material to be sampled at a 0^{o} to 45^{o} angle from horizontal. This orientation minimizes the spillage of sample.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler

Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should

be performed in accordance with ASTM D1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils".

The following procedures are used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.

2. Place the sampler in a perpendicular position on the sample material.

3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result.

4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.

5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. A larger barrel may be necessary to obtain the required sample volume.

6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

7.2.5 Test Pit/Trench Excavation

A backhoe can be used to remove sections of soil, when detailed examination of soil characteristics are required. This is probably the most expensive sampling method because of the relatively high cost of backhoe operation.

The following procedures are used for collecting soil samples from test pits or trenches:

1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities.

2. Review the site specific Health & Safety plan and ensure that all safety precautions including appropriate monitoring equipment are installed as required.

3. Using the backhoe, excavate a trench approximately three feet wide and approximately one foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than five feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.

4. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done.

5. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket.

6. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration

activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OHSA and corporate health and safety procedures, in addition to the procedures specified in the site specific Health & Safety Plan..

12.0 REFERENCES

Mason, B.J. 1983. Preparation of Soil Sampling Protocol: Technique and Strategies. EPA-600/4-83-020.

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ASTM D 1586-98, ASTM Committee on Standards, Philadelphia, PA.

APPENDIX A
Figures
SOP #2012
February 2000

FIGURE 1. Sampling Augers

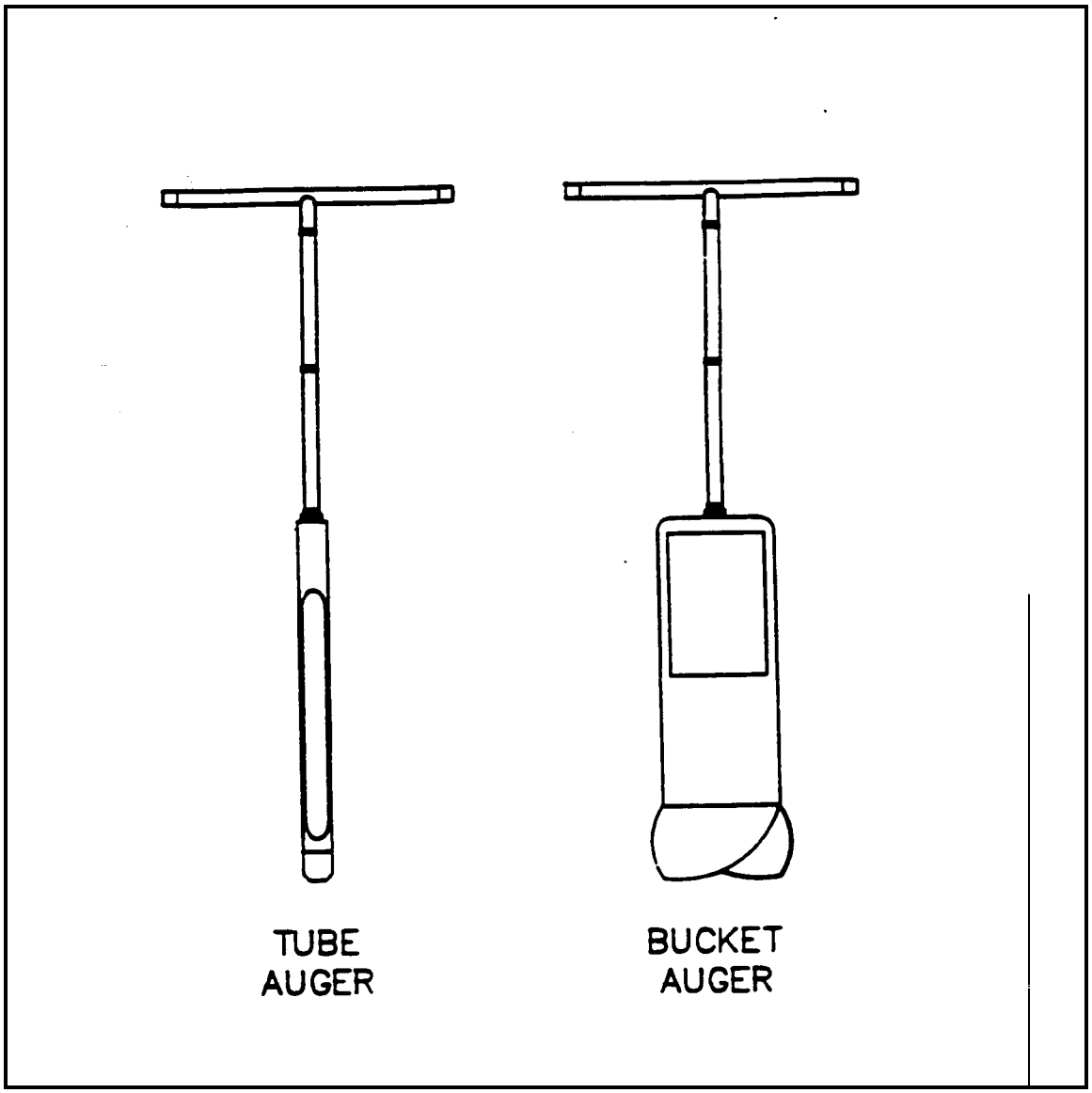
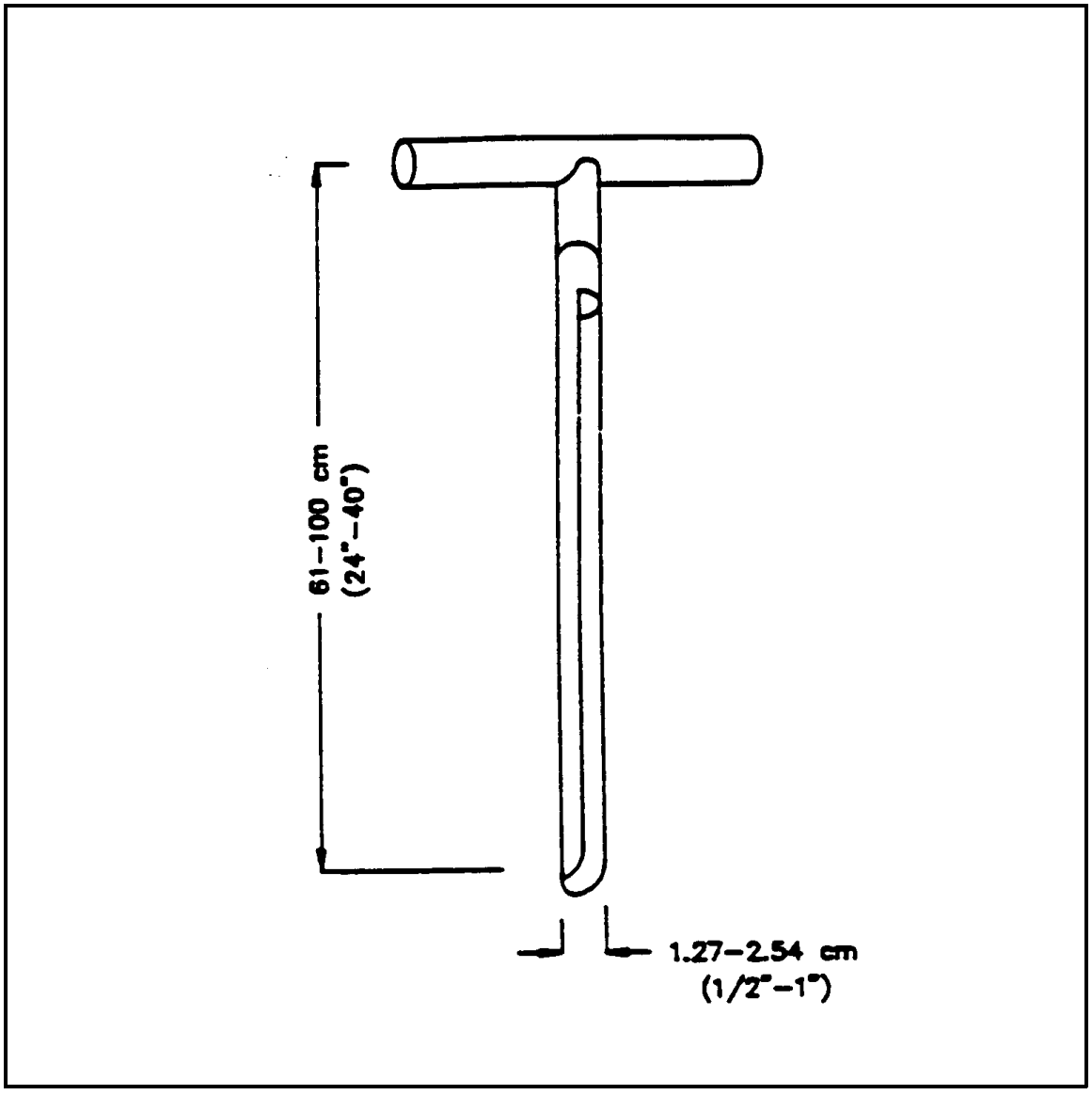


FIGURE 2. Sampling Trier



SEDIMENT SAMPLING

SOP#: 2016
DATE: 11/17/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is applicable to the collection of representative sediment samples. Analysis of sediment may be biological, chemical, or physical in nature and may be used to determine the following:

- toxicity;
- biological availability and effects of contaminants;
- benthic biota;
- extent and magnitude of contamination;
- contaminant migration pathways and source;
- fate of contaminants;
- grain size distribution.

The methodologies discussed in this SOP are applicable to the sampling of sediment in both flowing and standing water. They are generic in nature and may be modified in whole or part to meet the handling and analytical requirements of the contaminants of concern, as well as the constraints presented by site conditions and equipment limitations. However, if modifications occur, they should be documented in a site or personal logbook and discussed in reports summarizing field activities and analytical results.

For the purposes of this procedure, sediments are those mineral and organic materials situated beneath an aqueous layer. The aqueous layer may be either static, as in lakes, ponds, and impoundments; or flowing, as in rivers and streams.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Sediment samples may be collected using a variety of methods and equipment, depending on the depth of the aqueous layer, the portion of the sediment profile required (surface vs. subsurface), the type of sample required (disturbed vs. undisturbed), contaminants present, and sediment type.

Sediment is collected from beneath an aqueous layer either directly, using a hand held device such as a shovel, trowel, or auger; or indirectly, using a remotely activated device such as an Ekman or Ponar dredge. Following collection, sediment is transferred from the sampling device to a sample container of appropriate size and construction for the analyses requested. If composite sampling techniques are employed, multiple grabs are placed into a container constructed of inert material, homogenized, and transferred to sample containers appropriate for the analyses requested. The homogenization procedure should not be used if sample analysis includes volatile organics; in this case, sediment, or multiple grabs of sediment, should be transferred directly from the sample collection device or homogenization container to the sample container.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

1. Chemical preservation of solids is generally not recommended. Cooling to 4 °C is usually the best approach, supplemented by the appropriate holding time for the analyses requested.

2. Wide mouth glass containers with Teflon lined caps are utilized for sediment samples. The sample volume is a function of the analytical requirements and will be specified in the Work Plan.

3. If analysis of sediment from a discrete depth or location is desired, sediment is transferred directly from the sampling device to a labeled sample container(s) of appropriate size and construction for the analyses

requested. Transfer is accomplished with a stainless steel or plastic lab spoon or equivalent.

4. If composite sampling techniques or multiple grabs are employed, equal portions of sediment from each location are deposited into a stainless steel, plastic, or other appropriate composition (e.g., Teflon) containers. The sediment is homogenized thoroughly to obtain a composite representative of the area sampled. The composite sediment sample is transferred to a labeled container(s) of appropriate size and construction for the analyses requested. Transfer of sediment is accomplished with a stainless steel or plastic lab spoon or equivalent. Samples for volatile organic analysis must be transferred directly from the sample collection device or pooled from multiple areas in the homogenization container prior to mixing. This is done to minimize loss of contaminant due to volatilization during homogenization.

5. All sampling devices should be decontaminated, then wrapped in aluminum foil. The sampling device should remain in this wrapping until it is needed. Each sampling device should be used for only one sample. Disposable sampling devices for sediment are generally impractical due to cost and the large number of sediment samples which may be required. Sampling devices should be cleaned in the field using the decontamination procedure described in the Sampling Equipment Decontamination SOP.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Substrate particle size and organic matter content are a direct consequence of the flow characteristics of a waterbody. Contaminants are more likely to be concentrated in sediments typified by fine particle size and a high organic matter content. This type of sediment is most likely to be collected from depositional zones. In contrast, coarse sediments with low organic matter content do not typically concentrate pollutants and are generally found in erosional zones. The selection of a sampling location can, therefore, greatly influence the analytical results and should be justified and specified in the Work Plan.

5.0 EQUIPMENT/APPARATUS

Equipment needed for collection of sediment samples may include:

- Maps/plot plan
- Safety equipment
- Compass
- Tape measure
- Survey stakes, flags, or buoys and anchors
- Camera and film
- Stainless steel, plastic, or other appropriate composition bucket
- 4-oz., 8-oz., and one-quart wide mouth jars w/Teflon lined lids
- Ziploc plastic bags
- Logbook
- Sample jar labels
- Chain of Custody records, field data sheets
- Cooler(s)
- Ice
- Decontamination supplies/equipment
- Spade or shovel
- Spatula
- Scoop
- Trowel
- Bucket auger
- Tube auger
- Extension rods
- "T" handle
- Sediment coring device (tube, drive head, eggshell check value, nosecone, acetate tube, extension rods, "T" handle)
- Ponar dredge
- Ekman dredge
- Nylon rope or steel cable
- Messenger device

6.0 REAGENTS

Reagents are not used for preservation of sediment samples. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP.

7.0 PROCEDURES

7.1 Preparation

1. Determine the objective(s) and extent of the sampling effort. The sampling methods to be employed, and the types and amounts of equipment and supplies required will be a function of site characteristics and objectives of the study.

2. Obtain the necessary sampling and monitoring equipment.

3. Prepare schedules, and coordinate with staff, client, and regulatory agencies, if appropriate.

4. Decontaminate or preclean equipment, and ensure that it is in working order.

5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations. Specific site factors including flow regime, basin morphometry, sediment characteristics, depth of overlying aqueous layer, contaminant source, and extent and nature of contamination should be considered when selecting sample locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.2 Sample Collection

Selection of a sampling device is most often contingent upon: (1) the depth of water at the sampling location, and (2) the physical characteristics of the sediment to be sampled. The following procedures may be utilized:

7.2.1 Sampling Surface Sediment with a Trowel or Scoop from Beneath a Shallow Aqueous Layer

For the purpose of this method, surface sediment is considered to range from 0 to six inches in depth and a shallow aqueous layer is considered to range from 0 to 12 inches in depth. Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with tools such as spades, shovels, trowels, and scoops. Although this method can be used to collect both unconsolidated/consolidated sediment, it is limited somewhat by the depth and movement of the aqueous layer. Deep and rapidly flowing water render this method less accurate than others discussed below. However, representative samples can be collected with this procedure in shallow sluggish water provided care is demonstrated by the sample team member. A stainless steel or plastic sampling implement will suffice in most applications. Care should be exercised to avoid the use of devices plated with chrome or other materials; plating is particularly common with garden trowels.

The following procedure will be used to collect sediment with a scoop, shovel, or trowel:

1. Using a decontaminated sampling implement, remove the desired thickness and volume of sediment from the sampling area.

2. Transfer the sample into an appropriate sample or homogenization container. Ensure that non-dedicated containers have been adequately decontaminated.

3. Surface water should be decanted from the sample or homogenization container prior to sealing or transfer; care should be taken to retain the fine sediment fraction during this procedure.

7.2.2 Sampling Surface Sediment with a Bucket Auger or Tube Auger from Beneath a Shallow Aqueous Layer

For the purpose of this method, surface sediment is considered to range from 0 to six inches in depth and a shallow aqueous layer is considered to range from 0 to 24 inches in depth. Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with a system consisting of bucket auger or tube auger, a series of extensions, and a "T" handle (Figure 1, Appendix A). The use of additional extensions in conjunction with a bucket auger can increase the depth of water from which sediment can be collected from 24 inches to 10 feet or more. However, sample handling and manipulation increases in difficulty with increasing depth of water. The bucket auger or tube auger is driven into the sediment and used to extract a core. The various depths represented by the core are homogenized or a subsample of the core is taken from the appropriate

depth.

The following procedure will be used to collec t sediment samples with a bucket auger or tube auger:

1. An acetate core may be inserted into th e bucket auger or tube auger prior to sampling if characteristics of the sediments o r waterbody warra nt. By using this technique, an intact core can be extracted.

2. Attach the auger head to the required length of extensions, then attach the "T" handle t o the upper extension.

3. Clear the area to be sampled of any surfac e debris.

4. Insert the buc ket auger or tube auger into the sediment at a 0º to 20º angle from vertical . This orientation minimizes spillage of th e sample from the sampler upon extractio n from the sediment and water.

5. Rotate the auger to cut a core of sediment.

6. Slowly withdraw the auger; if using a tub e auger, make sure that the slot is facin g upward.

7. Transfer the sample or a specified aliquot of sample into an appropriate sample o r homogenization container. Ensure that non-dedicated containers have been adequatel y decontaminated.

7.2.3 Sampling Deep Sediment with a Bucket Auger or Tube Auger fro m Beneath a Shallow Aqueous Layer

For the purpose of this method, deep sediment i s considered to ran ge from six to greater than 18 inches in depth and a shallow aqueous layer is considered to range from 0 to 24 inches. Collection of dee p sediment from beneath a shallow aqueous layer can be accomplished with a system consisting of a bucke t auger, a tube auger, a series of extensions and a "T" handle. The use of additional extensions ca n increase the depth of water from which sediment can be collected from 24 inches to five feet or more . However, water clarity mu st be high enough to permit the sampler to directly observe the samplin g operation. In addition, sample handling an d manipulation increases in difficulty with increasin g depth of water. The bucket auger is used to bore a hole to the upper range of the desired sampling depth and then withdrawn. The tube auger is then lowered down the borehole, and driven into the sediment to the lower range of the desired sampling depth. The tube is then withdrawn and the sample recovered from the tube. This method can be used to collect firml y consolidated sediments, but is somewhat limited b y the depth of the aqueo us layer, and the integrity of the initial borehole.

The following procedure will be used to collect deep sediment samples with a bucket auger and a tub e auger:

1. Attach the bucket auger bit to the require d lengths of extensions, then attach the "T " handle to the upper extension.

2. Clear the area to be sampled of any surfac e debris.

3. Begin augering, periodically removing an y accumulated sediment (i.e., cuttings) fro m the auger bucket. Cuttings should b e disposed of far enough from the samplin g area to minimize cross contamination o f various depths.

4. After reaching the upper range of the desired depth, slowly and carefully remove bucke t auger from the boring.

5. Attach the tube auger bit to the require d lengths of extensions, then attach the "T " handle to the upper extension.

6. Carefully lower tube auger down borehol e using care to avoid making contact with the borehole sides and, thus , cross contaminating the sample. Gradually force tube auger into sediment to the lower range of the desire d sampling depth. Hammering of the tub e auger to facilitate coring should be avoide d as the vibrations may cause the boring walls to collapse.

7. Remove tu be auger from the borehole, again taking ca re to avoid making contact with the borehole sides and, thus , cross contaminating

the sample.

8. Discard the top of core (approximately 1 inch); as this represe nts material collected by the tube auger before penetration to the layer of concern.

9. Transfer sample into an appropriate sampl e or homogenization container. Ensure tha t non-dedicated containers have bee n adequately decontaminated.

7.2.4 Sampling Surface Sediment with a n Ekman or Ponar Dredge fro m Beneath a Sh allow or Deep Aqueous Layer

For the purpose of this method, surface sediment i s considered to range from 0 to six inches in depth . Collection of surface sediment can be accomplishe d with a system consisting of a remotely activate d device (dredge) and a deployment system. Thi s technique consists of lowering a sampling devic e (dredge) to the surface of the sediment by use of a rope, cable, or extended handle. The mechanism i s activated, and the device entraps sediment in sprin g loaded or lever operated jaws.

An Ekman dredge is a lightweight sediment sampling device with spring activate d jaws. It is used to collect moderately consolid ated, fine textured sediment. The following procedure will be used for collectin g sediment with an Ekman dredge (Figure 2, Appendix A):

1. Attach a sturdy nylon rope or stainless steel cable through the hole on the top of th e bracket, or secure t he extension handle to the bracket with machine bolts.

2. Attach springs to both sides of the jaws. Fix the jaws so that they are in open position by placing trip cables over the release studs . Ensure that the hinged doors on the dredg e top are free to open.

3. Lower the sampler to a point 4 to 6 inche s above the sediment surface.

4. Drop the sampler to the sediment.

5. Trigger the jaw release mechanism b y lowering a messenger down the line, or b y depressing the but ton on the upper end of the extension handle.

6. Raise the sampler and slowly decant any free liquid through the top of the sampler. Car e should be taken to retain the fine sedimen t fraction during this procedure.

7. Open the dredge jaws and tran sfer the sample into a stainless steel, plastic or othe r appropriate composition (e.g., Teflon) container. Ensure that non-dedicate d containers have been adequatel y decontaminated. If necessary, continue t o collect additional sediment grabs unti l sufficient mat erial has been secured to fulfill analytical requirements. Thoroughl y homogenize and then transfer sediment t o sample containers appropriate for th e analyses requested. Samples for volatil e organic analysis must be collected directl y from the bucket before homogenization t o minimize volatilization of contaminants.

A Ponar dredge is a heavyweight sediment sampling device with weighted jaws that are lever or sprin g activated. It is used to collect consolidated fine t o coarse textured sediment. The following procedur e will be used for collecting sediment with a Pona r dredge (Figure 3, Appendix A):

1. Attach a sturdy nylon rope or steel cable t o the ring provided on top of the dredge.

2. Arrange the Ponar dredge with the jaws i n the open position, setting the trip bar so th e sampler remains open when lifted from th e top. If the dredge is so equipped, place th e spring loaded pin into the al igned holes in the trip bar.

3. Slowly lower the sampler to a poin t approximately two inches above th e sediment.

4. Drop the sampler to the sediment. Slack on the line will release the trip bar or sprin g loaded pin; pull up sharply on the lin e closing the dredge.

5. Raise the dredge to the surface and slowl y

decant any free liquid through the screens on top of the dredge. Care should be taken to retain the fine sediment fraction during this operation.

6. Open the dredge and transfer the sediment to a stainless steel, plastic or other appropriate composition (e.g., Teflon) container. Ensure that non-dedicated containers have been adequately decontaminated. If necessary, continue to collect additional sediment until sufficient material has been secured to fulfill analytical requirements. Thoroughly homogenized and then transfer sediment to sample containers appropriate for the analyses requested. Samples for volatile organic analysis must be collected directly from the bucket before homogenization to minimize volatilization of contaminants.

7.2.5 Sampling Subsurface Sediment with a Coring Device from Beneath a Shallow Aqueous Layer

For purposes of this method, subsurface sediment is considered to range from 6 to 24 inches in depth and a shallow aqueous layer is considered to range from 0 to 24 inches in depth. Collection of subsurface sediment from beneath a shallow aqueous layer can be accomplished with a system consisting of a tube sampler, acetate tube, eggshell check valve, nosecone, extensions, and "T" handle, or drivehead. The use of additional extensions can increase the depth of water from which sediment can be collected from 24 inches to 10 feet or more. This sampler may be used with either a drive hammer for firm sediment, or a "T" handle for soft sediment. However, sample handling and manipulation increases in difficulty with increasing depth of water.

The following procedure describes the use of a sample coring device (Figure 4, Appendix A) used to collect subsurface sediments.

1. Assemble the coring device by inserting the acetate core into the sampling tube.

2. Insert the "egg shell" check valve into the lower end of the sampling tube with the convex surface positioned inside the acetate core.

3. Screw the nosecone onto the lower end of the sampling tube, securing the acetate tube and eggshell check valve.

4. Screw the handle onto the upper end of the sampling tube and add extension rods as needed.

5. Place the sampler in a perpendicular position on the sediment to be sampled.

6. If the "T" handle is used, place downward pressure on the device until the desired depth is reached. After the desired depth is reached, rotate the sampler to shear off the core at the bottom. Slowly withdraw the sampler from the sediment and proceed to Step 15.

7. If the drive hammer is selected, insert the tapered handle (drive head) of the driven hammer through the drive head.

8. Drive the sampler into the sediment to the desired depth.

9. Record the length of the tube that penetrated the sample material, and the number of blows required to obtain this depth.

10. Remove the drive hammer and fit the keyhole-like opening on the flat side of the hammer onto the drive head. In this position, the hammer serves as a handle for the sampler.

11. Rotate the sampler to shear off the core at the bottom.

12. Lower the sampler handle (hammer) until it just clears the two ear-like protrusions on the drive head, and rotate about 90 °.

13. Slowly withdraw the sampler from the sediment. If the drivehead was used, pull the hammer upwards and dislodge the sampler from the sediment.

14. Carefully remove the coring device from the water.

15. Unscrew the nosecone and remove the

eggshell check valve.

16. Slide the acetate core out of the sample r tube. Decant surface water, using care t o retain the fine sediment fraction. If hea d space is present in the upper end, a hacksaw may be used to shear the acetate tube off a t the sediment surface. The acetate core may then be capped at both ends. Indicate on the acetate tube the appropr iate orientation of the sediment core using a waterproof marker . The sample may be used in this fashion, o r the contents transferred to a sample o r homogenization container.

17. Open the acetate tube and transfer th e sediment to a stainless steel, plastic or other appropriate composition (e.g., Teflon) container. Ensure that non-dedicate d containers have been adequatel y decontaminated. If necessary, continue t o collect additional sediment until sufficien t material has been s ecured to fulfill analytical requirements. Thoroughly homogenize an d then transfer sediment to sample container s appropriate for the analyses requested . Samples for volatile organ ic analysis must be collected directly from the bucket befor e homogenization to min imize volatilization of contaminants.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality as surance (QA) activities which apply to the implementation of thes e procedures. However, the following QA procedures apply:

1. All data must be documented on field dat a sheets or within site logbooks.

2. All instrumentation must be operated i n accordance with operating instructions a s supplied by the manufacturer, unles s otherwise specified in the work plan . Equipment checkout and calibratio n activities must occur prior t o sampling/operation, and they must b e documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials , follow U.S. EPA/OSHA and Corporate health an d safety procedures.

More specifically, when sampling sediment fro m waterbodies, physical hazards must be identified and adequate precautions must be taken to ensure th e safety of the sampling team. The team membe r collecting the sample should not get too close to th e edge of the waterbody, where bank failure may cause loss of balance. To prevent this, the perso n performing the sampling should be on a lifeline, and be wearing adequate protective equipment. I f sampling from a vess el is determined to be necessary, appropriate protective measures m ust be implemented.

12.0 REFERENCES

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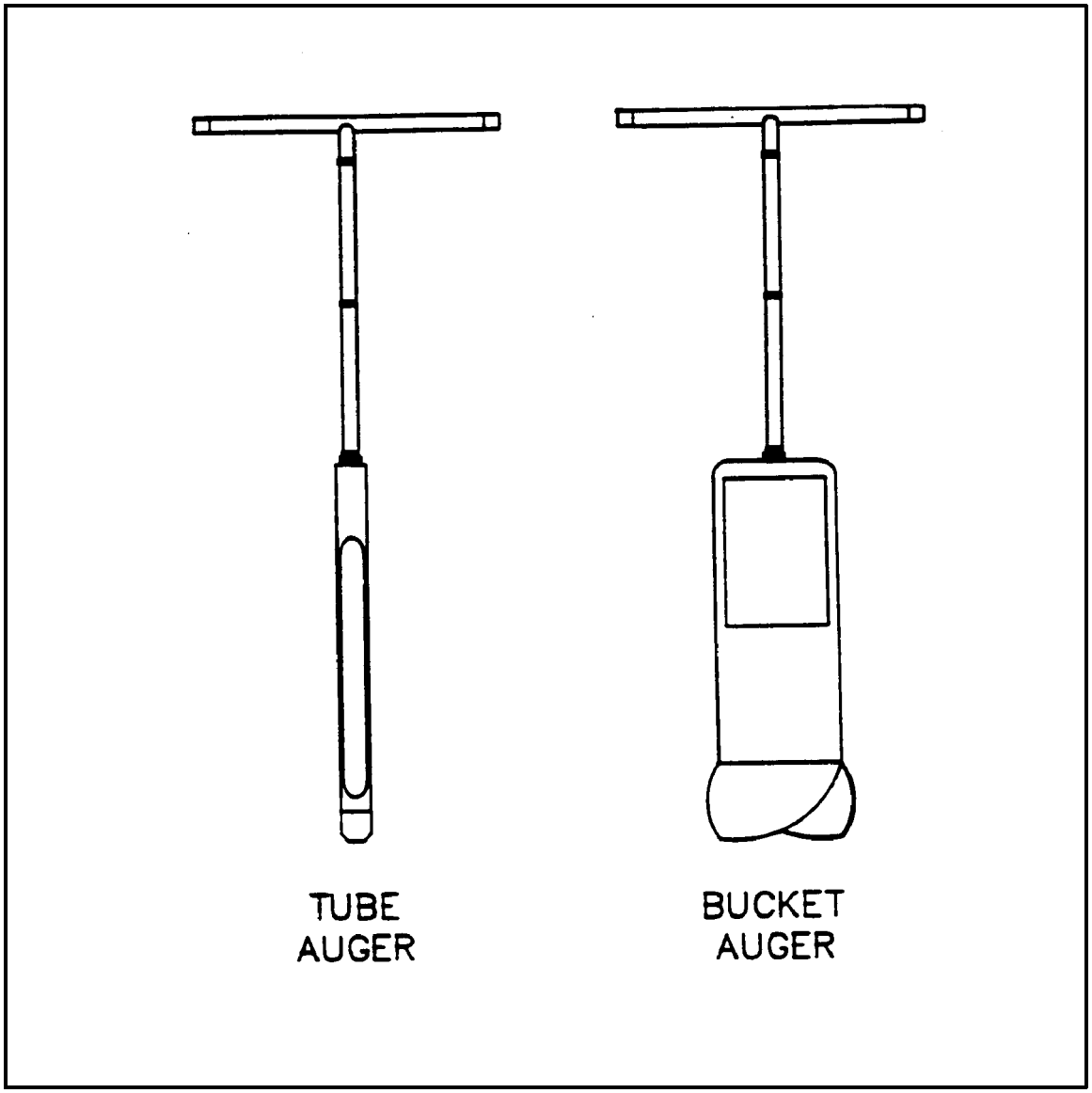
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APPENDIX A

Figures

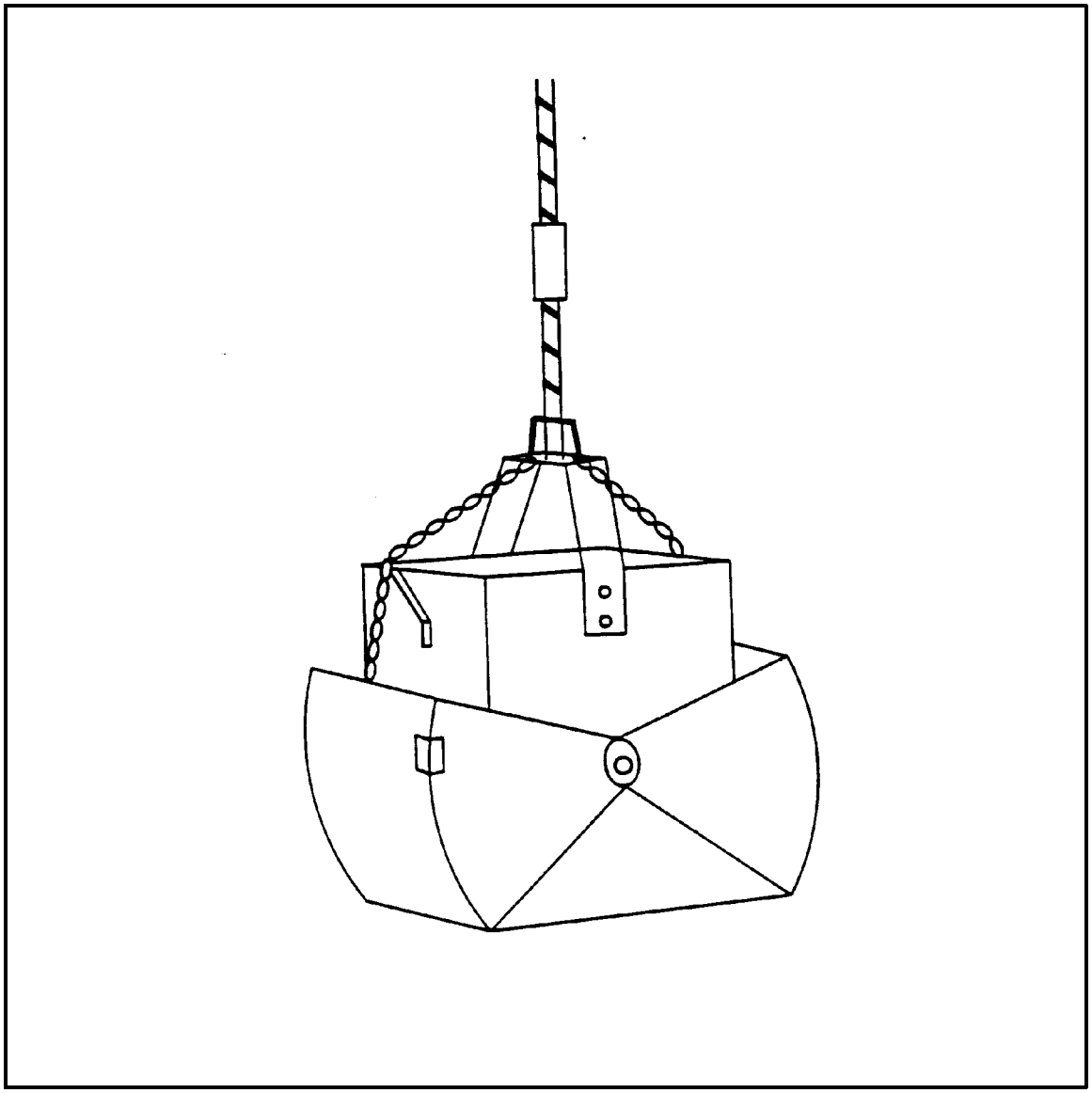
FIGURE 1. Sampling Auger



APPENDIX A (Cont'd)

Figures

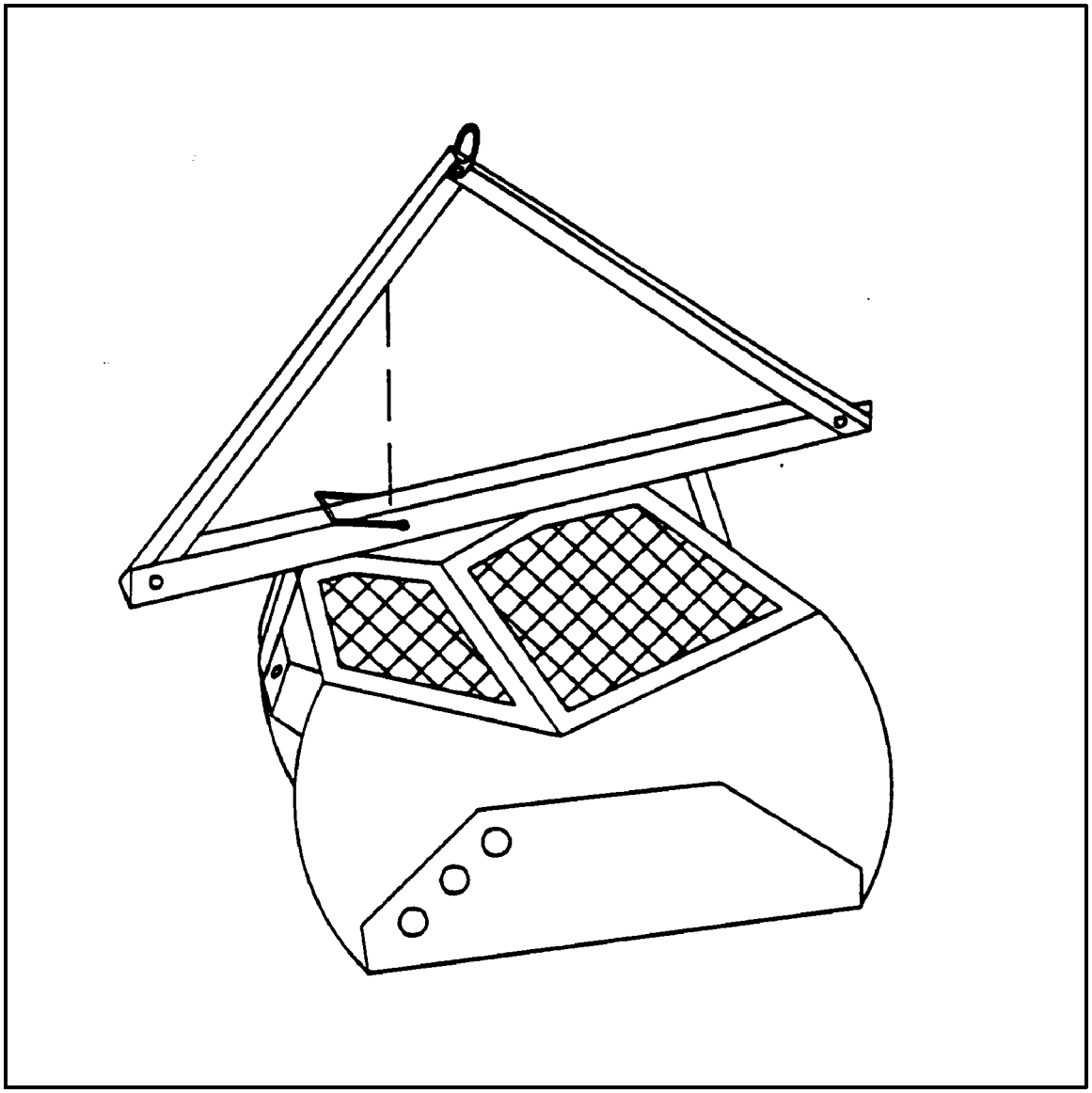
FIGURE 2. Ekman Dredge



APPENDIX A (Cont'd)

Figures

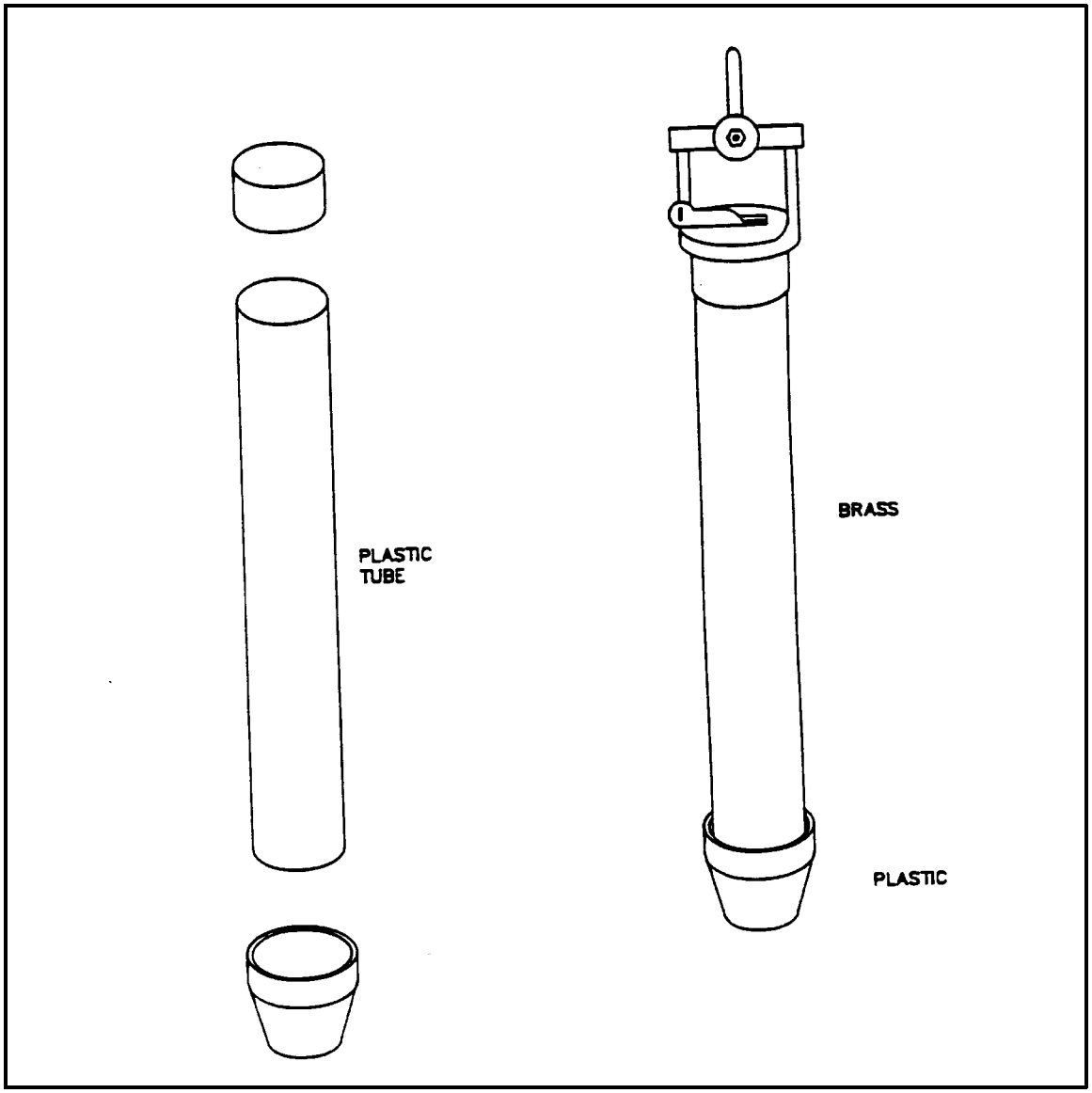
FIGURE 3. Ponar Dredge



APPENDIX A (Cont'd)

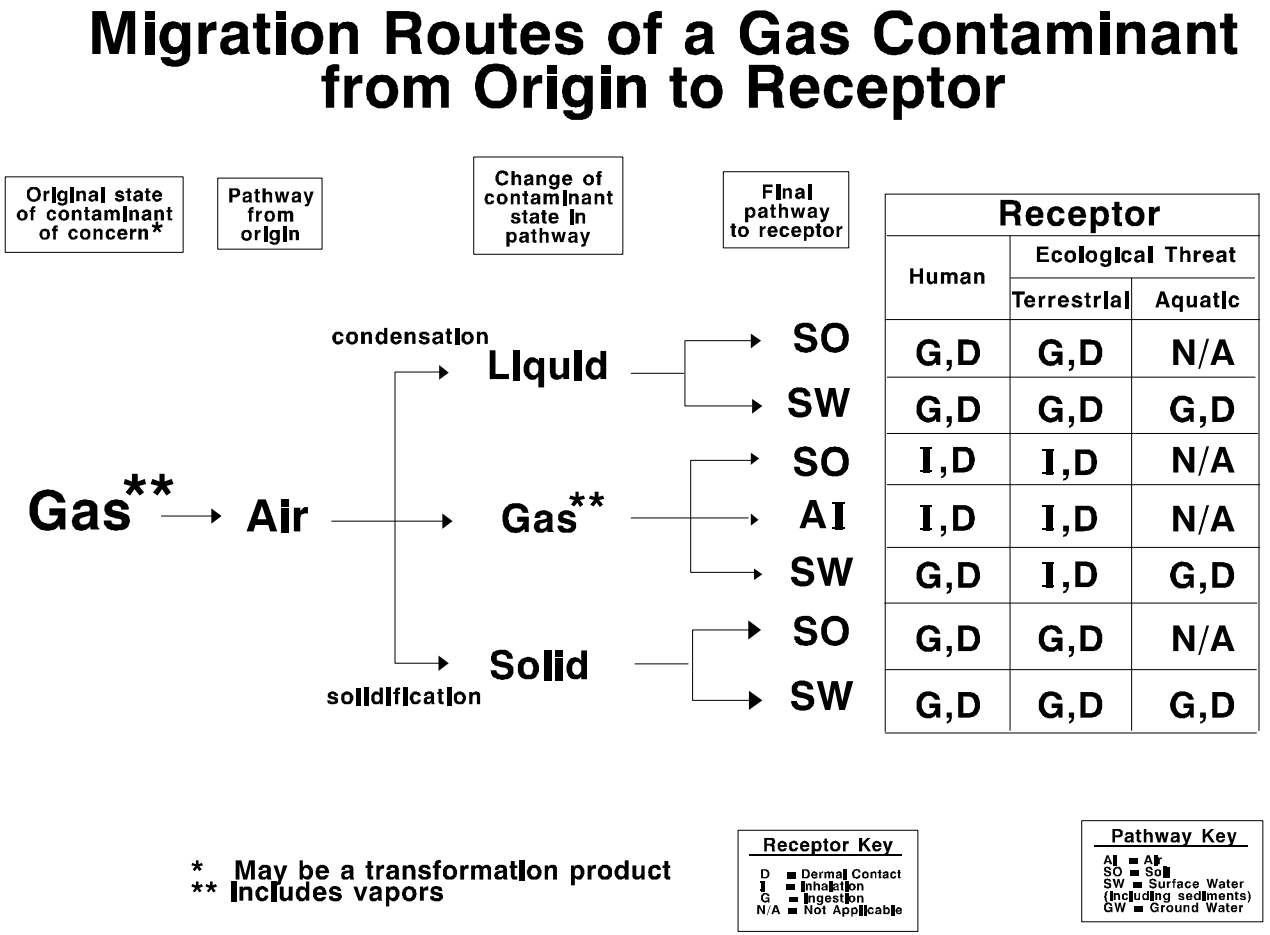
Figures

FIGURE 4. Sample Coring Device



APPENDIX A -- EXAMPLE OF FLOW DIAGRAM FOR CONCEPTUAL SITE MODEL

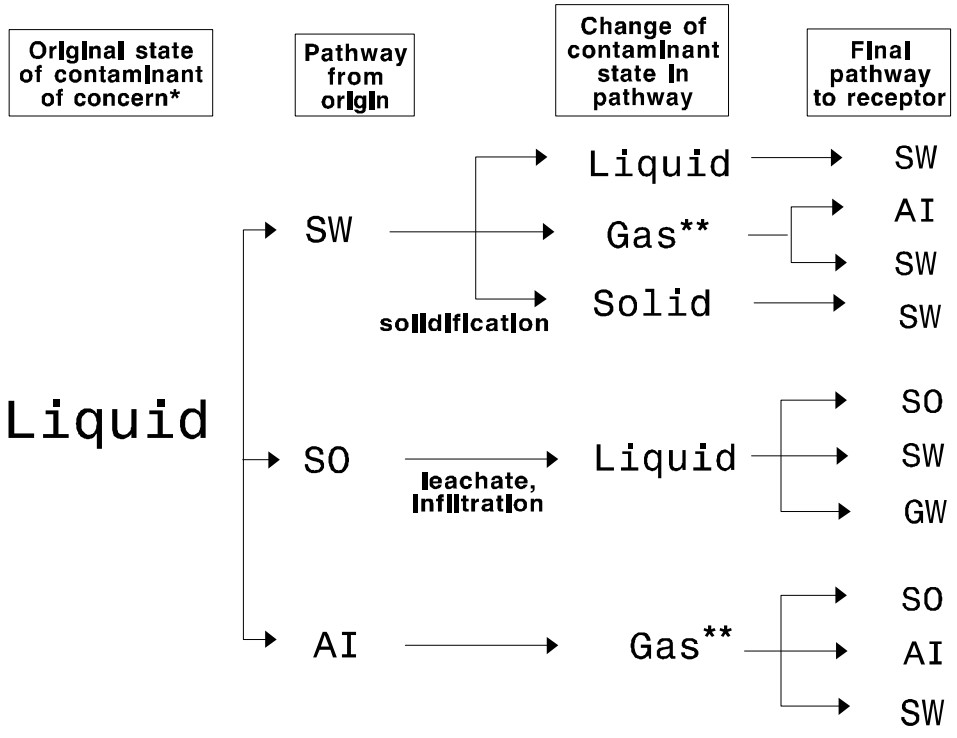
Figure A-1



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
G,D	G,D	N/A
G,D	G,D	G,D
I,D	I,D	N/A
I,D	I,D	N/A
G,D	I,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D

Figure A-2

Migration Routes of a Liquid Contaminant from Origin to Receptor



Original state of contaminant of concern*	Pathway from origin	Change of contaminant state in pathway	Final pathway to receptor	Receptor: Human	Receptor: Ecological Threat: Terrestrial	Receptor: Ecological Threat: Aquatic
Liquid	SW	Liquid	SW	G,D	G,D	G,D
		Gas**	AI	I,D	I,D	N/A
			SW	G,D	G,D	G,A
		Solid (solidification)	SW	G,D	G,D	G,D
	SO	Liquid (leachate, infiltration)	SO	G,D	G,D	N/A
			SW	G,D	G,D	G,D
			GW	G,D	N/A	N/A
	AI	Gas**	SO	G,D	G,D	N/A
			AI	I,D	I,D	N/A
			SW	G,D	G,D	G,D

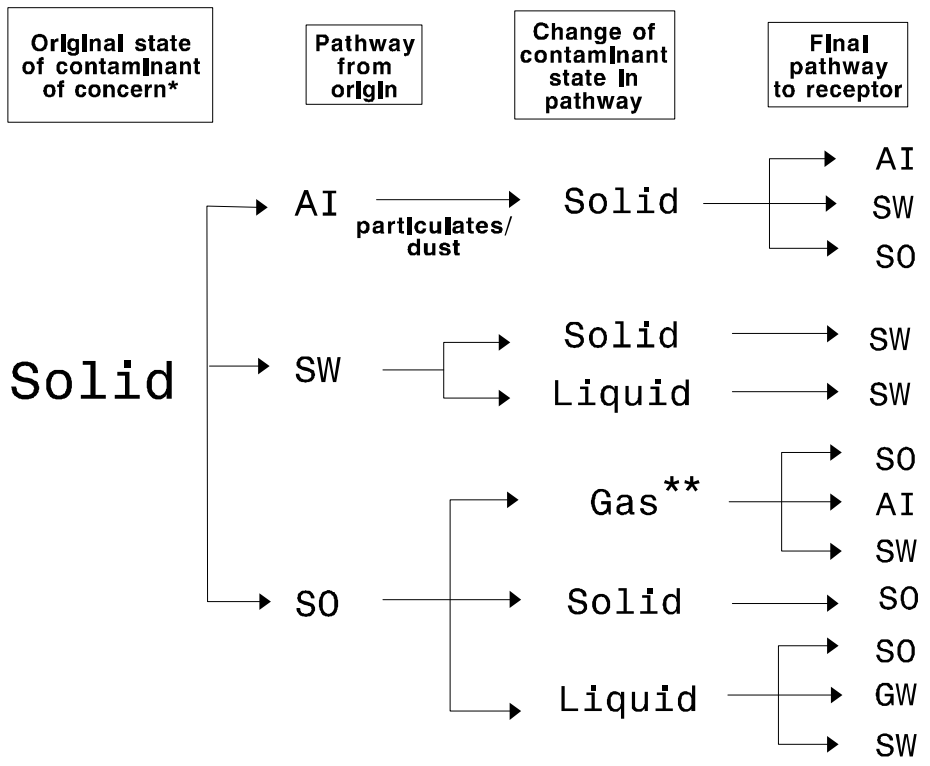
* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

Figure A-3

Migration Routes of a Solid Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D
G,D	G,D	G,D
G,D	G,D	N/A
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	N/A
G,D	N/A	N/A
G,D	G,D	G,D

* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

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Attachment I

ENSR SOP #7115 – Subsurface Soil Sampling by Split Spoon Methods

SOP NUMBER: 7115

Subsurface Soil Sampling by Split Spoon

Date:	3rd Qtr. 1994
Revision Number:	3
Author:	Charles Martin
Discipline:	Geosciencies

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the methods used in obtaining subsurface soil samples for physical and/or chemical analysis. Subsurface soil samples are obtained in conjunction with soil boring programs and provide information as to the physical and/or chemical makeup of the subsurface environment.

The purpose of this SOP is to provide a description of a specific method or procedure to be used in the collection of subsurface soil samples. Subsurface soil is defined as unconsolidated material which may consist of one or a mixture of the following materials: sand, gravel, silt, clay, peat (or other organic soils), and fill material. Subsurface soil sampling, conducted in accordance with this SOP will promote consistency in sampling and provide a basis for sample representativeness.

This SOP covers subsurface soil sampling by split-spoon only, as this is the means most often used for obtained samples of unconsolidated deposits. Other types of equipment are available for use in subsurface soil sampling, including thin-wall tube samplers (Shelby tubes), piston samplers, and continuous core barrel samplers. Information on the use of these other sampling devices may be found in several available drilling handbooks and respective state and/or federal agency technical guidance documents. The American Society for Testing and Materials (ASTM) also provides procedures for use of split-spoon and other sampling devices.

Deviations from this SOP to accommodate other regulatory requirements should be reviewed in advance of the field program, should be explained in the project work plan, and must be documented in the field project notebook when they occur.

1.2 General Principles

Split-spoon subsurface soil sampling generally requires use of a drilling rig and typically the hollow-stem auger or other common drilling method to generate a borehole in which to use the split-spoon sampler. The split-spoon sampler is

inserted through the augers (or other type of drill casing) then is driven into the subsurface soil with a weighted hammer. The sampler is then retrieved and opened to reveal the recovered soil sample. Soil samples may be collected at a continuous interval or at pre-selected vertically spaced intervals within the borehole.

1.3 Quality Assurance Planning Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the QAPP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

1.4 Health and Safety Considerations

Subsurface soil sampling may involve chemical hazards associated with the types of contaminants potentially encountered and will always involve potential physical hazards associated with use of drilling equipment. When sampling is performed in materials which may contain hazardous constituents, or when the quality assurance objectives of the project require the use of hazardous solvents, adequate Health and Safety measures must be taken to protect sampling personnel. These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

2.0 RESPONSIBILITIES

2.1 Drilling Subcontractor

It will be the responsibility of the drilling subcontractor to provide the necessary materials for obtaining subsurface soil samples. This generally includes one or more split-spoon samplers in good operating condition and sample containers used for stratigraphic characterization samples (sample containers for environmental samples should be provided by the designated analytical laboratory). It is the drilling subcontractor's responsibility to provide and maintain their own boring logs if desired. Equipment decontamination materials should also be supplied by the subcontractor and should meet project specifications.

2.2 Project Geologist/Sampling Engineer

It will be the responsibility of the project geologist/sampling engineer to conduct subsurface soil sampling in a manner which is consistent with this SOP. The project geologist/sampling engineer will observe all activities pertaining to subsurface soil sampling to ensure that the SOP is followed, and to record all pertinent data onto a boring log. It is also the project geologist/sampling engineer's responsibility to indicate the specific targeted sampling depth or sampling interval to the drilling subcontractor. The project geologist/sampling engineer is also responsible for the collection of representative environmental or stratigraphic characterization samples once the sampling device has been retrieved and opened. Additional sample collection responsibilities include labeling, handling, and storage of samples until further chain-of-custody procedures are implemented.

3.0 REQUIRED MATERIALS

In addition to those materials provided by the subcontractor, the project geologist/sampling engineer will require:

- Project Sampling Plan, QAPP, and HASP
- Boring logs
- Teaspoon or spatula (stainless steel is recommended)
- Sample kit (bottles, labels, custody records and tape, cooler)
- Sample collection pen
- Folding rule or tape measure
- Equipment decontamination materials
- Health and safety equipment (as required by HASP)
- Field project notebook/pen

4.0 METHOD

4.1 General Method Description

Split-spoon sampling devices are typically constructed of steel and are most commonly available in lengths of 18 and 24 inches and diameters of 1.5 to 3 inches. The split-spoon consists of a tubular body with two halves that split apart lengthwise, a drive head on the upper end with a ball-check valve for venting, and a hardened steel cutting shoe at the bottom. The soil sample enters the split-spoon through the cutting shoe as the device is driven into the ground. A replaceable plastic or metal basket is often inserted into the shoe to assist with retaining samples. Once the

sampler is retrieved, the drive head and cutting shoes are removed and the split-spoon halves are then separated, revealing the sample.

Sample depth intervals are usually defined on a project-specific basis with these requirements specified in the project sampling plan. Sampling intervals typically range from one (1) sample per five (5) feet of drilling to continuous sampling where the entire drilled interval is sampled.

Subsurface soil sampling is usually accomplished as part of a drilling program where a soil boring is advanced with drilling equipment to the designated depth prior to collection of a representative sample. The general procedures outlined briefly in the following section provide requirements for advancing drill casing/augers in preparation for sampling.

4.2 General Procedures - Borehole Preparation

4.2.1 Advancing Casing/Augers

Soil borings that are completed for soil sampling purposes are typically advanced using hollow-stem augers and sometimes drive-and-wash or other casing methods. The casing/augers must be of sufficient diameter to allow for soil sampling at a minimum. The casing/augers will be advanced according to project requirements to the required depth for sampling. If hollow-stem augers are used, a temporary plug shall be used in the lead auger to prevent the auger from becoming filled with drill cuttings while drilling is in progress.

4.2.2 Obstructions

For those borings which encounter obstructions, the casing/augers will be advanced past or through the obstruction if possible. Caution should be exercised when obstructions are encountered and an effort made to identify the obstruction before drilling is continued. If the obstruction is not easily drilled through or removed, the boring should be relocated to an adjacent location.

4.2.3 Use of Added Water

The use of added or recirculated water during drilling is permitted when necessary. Use of extraneous water should be minimized or avoided if possible as it may impact sample quality. Water usage should be documented in the field notebook. Sampling and analysis of added or

recirculated water may be required for quality assurance purposes (refer to QAPP). If a well is installed within the completed borehole, removal of the added water may be required.

4.3 Sampling Procedure

4.3.1 Equipment Decontamination

Each split-spoon must be decontaminated prior to its initial use and following collection of each soil sample. Site-specific requirements for equipment decontamination should be outlined within the Project Sampling Plan. Equipment decontamination procedures are also outlined within SOP 7600 - Decontamination of Equipment.

4.3.2 Standard Penetration Test

The drilling subcontractor will lower the split-spoon into the borehole. Samples are generally obtained using the Standard Penetration Test (SPT) in accordance with ASTM standards (ASTM D 1586-84). Following this method, the sampler will be driven using the 140-pound hammer with a vertical free drop of 30 inches using two turns of the rope on the cathead. The number of hammer blows required for every 6 inches of penetration will be recorded on the boring log. Blowcount information is used as an indicator of soil density for geotechnical as well as stratigraphic logging purposes. Once the split-spoon has been driven to its fullest extent, or to refusal, it will be removed from the borehole.

4.3.3 Sample Recovery

The split-spoon will be immediately opened upon removal from the casing/auger. The open sampler shall then be screened for volatile organics with a photoionization device (PID) if required by the Project Sampling Plan. If the Sampling Plan also requires individual soil sample headspace screening for volatile organic compounds, then a small portion of the split-spoon sample shall be removed and properly contained for that purpose.

Sample recovery will be determined by the project geologist/sampling engineer who will examine the soil core once the sampler is opened. The length of sample shall then be measured with a folding rule or tape measure. Any portion of the split-spoon contents which are not considered part of the true sample (i.e., heaved soils) will be discarded. If the sample recovery is considered inadequate for sample characterization or analytical testing

purposes, another sample should be collected from the next vertical interval if possible before drilling is reinitiated.

Adequate sample recovery for stratigraphic logging purposes and/or headspace organic vapor testing purposes should be approximately 6 inches. Adequate sample recovery for analytical testing purposes should be a minimum of 12 inches and is somewhat dependent on the type of analytical testing required. In some cases, continuous sampling over a short interval, and compositing of the sample, may be required to satisfy analytical testing requirements. Larger diameter samplers may be used if large volumes of soil are required for analytical testing.

4.3.4 Sample Containment - General

Once retrieved, the sample will be removed from the split-spoon with a teaspoon or spatula and placed into the appropriate sample container. The sample will be split if necessary to meet sampling program requirements. Sample splitting may be necessary to provide individual samples for headspace testing, visual characterization, physical testing, analytical testing, or simply for archiving purposes. In general, most sampling programs are structured around environmental characterization needs; therefore, sample portions required for analytical testing should be collected first. The Project Sampling Plan and QAPP provides specific sample container requirements for each type of sample and should be referred to for guidance.

Once filled, the sample containers should be properly capped, cleaned, and labeled, and chain-of-custody and sample preservation procedures initiated. Sampling equipment should then be properly decontaminated.

4.3.5 Sample Containment - Volatile Organic Analyses

Collection of subsurface soil samples for volatile organic analysis (VOA) is slightly more complex than collection of samples for other routine chemical or physical testing primarily because of the concern for the potential loss of volatiles during the sample collection procedure. To limit the potential for loss of volatiles, the soil sample needs to be obtained as quickly and as directly as possible from the split-spoon. This generally means that the VOA sample is to be collected and placed into the appropriate sample container first. The VOA sample should also be obtained from a discrete portion of the entire sample interval and not composited or homogenized. The remainder of the recovered sample can then be composited, homogenized or split to meet the other testing requirements. The boring log and/or sample logbook should be

filled out to indicate actual sample collection depths for both VOA samples and other portions of the sample which may have been composited over a larger vertical interval.

5.0 QUALITY CONTROL

Quality control requirements are dependent on project-specific sampling objectives. The QAPP will provide requirements for sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

6.0 DOCUMENTATION

Various forms are required to ensure that adequate documentation is made of sample collection activities. These forms include:

- Boring logs
- Field log books
- Sample collection records
- Chain-of-custody records
- Shipping labels

Boring logs (Figure 1) will provide visual and descriptive information for each sample collected and are often the most critical form of documentation generated during a sampling program. The field log book is kept as a general log of activities. Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes. Shipping labels are required if sample coolers are to be transported to the laboratory by a third party (courier service). Original copies of these records should be maintained in the appropriate project files.

7.0 REFERENCES

ASTM D 1586-84

Figure 1

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Sheet 1 of __

BORING LOG

Project No. _____ Date – Start _____ Finish _____ Boring _____

Project Name _____ Drilling Co. _____

Location _____ Drilling Method _____

Total Depth _____ Inspector _____ Reviewer _____

Remarks _____

Depth Feet	Sample				Graphic Log	Lithologic Description	Equipment Installed
	Type & No.	Blows per 6 in.	Depth Range	Rec.			

Attachment J

ENSR SOP #7116 – Subsurface Soil Sampling by Geoprobe Methods

SOP NUMBER: 7116

Subsurface Soil Sampling by Geoprobe™ Methods

Date:	August 2006
Revision Number:	0
Author:	Charles Martin
Discipline:	Geoscience

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the methods available for collecting subsurface soil samples using commercially available Geoprobe™ Systems (or other similar vendor) soil probing equipment. Subsurface soil samples may be obtained using this system for purposes of determining subsurface soil conditions and for obtaining soil samples for physical and/or chemical evaluation.

The purpose of this SOP is to provide a description of a specific method or procedure to be used in the collection of subsurface soil samples using the Geoprobe™ system. Subsurface soil is defined as unconsolidated material which may consist of one or a mixture of the following materials: sand, gravel, silt, clay, peat (or other organic soils), and fill material. Subsurface soil sampling, conducted in accordance with this SOP will promote consistency in sampling and provide a basis for sample representativeness.

This SOP covers subsurface soil sampling using Geoprobe™ Systems equipment; specifically, the Macro-Core Soil Sampler, and the Large Bore Sampler. Use of this sampling equipment requires use of the Geoprobe™ hydraulically-powered percussion/probing machine. Geoprobe™ sampling is usually performed by subcontractors, although rental equipment is available for use by trained operators.

The Geoprobe™ sampling methods covered in this SOP are applicable to unconsolidated soil/fill materials and to a maximum recommended depth of approximately 30 feet. Sampling depths are greatly dependent upon soil density as the hydraulically-powered probing unit has power limitations. Sample recovery is also somewhat dependent on grain size as very coarse gravel, cobbles, and boulders will occasionally cause premature refusal of the sampler. It is generally preferable to have some prior knowledge of site soil conditions if sampling activities are proposed where equipment limitations may become a factor.

Other types of equipment and sampling methods are available for use in obtaining samples of unconsolidated materials; and include split-spoons, Shelby tubes, and

continuous core barrel samplers. Information on these and other soil sampling devices may be found in other ENSR SOPs, ASTM procedures, drilling handbooks, and respective state and/or federal agency technical guidance documents.

1.2 General Principles

Soil sampling using the Geoprobe™ System requires use of the hydraulically-powered percussion/probing machine and either the Macro-Core Soil Sampler or the Large Bore Sampler soil sampling devices. The percussion/probing machine is typically mounted onto the bed of a pickup truck or van so that a stable working platform is established. The percussion/probing machine, through its hydraulic operation, pushes and hammers the soil sampling equipment vertically into the ground within the targeted sampling interval. The soil sampler is then extracted from the ground to recover the sample.

The Macro-Core Sampler (Figure 1) consists of a 45-inch long by 1.5-inch diameter open-ended steel sampling tool with liners made of clear plastic (cellulose acetate butyrate), stainless steel, or teflon. The tool is designed for use in a continuous sampling capacity in an open borehole up to depths of approximately 24 feet. The borehole walls are required to stay open in order to collect a sample from the next depth interval. Once the sampling tool is removed from the ground, the inserted liner containing the soil sample is removed from the tool. The soil sample is then cut from or extracted from the liner. This sampling tool is most often used for soil profiling and collection of larger volume soil samples (1,300 ml).

The Large Bore Sampler (Figure 2) consists of a 22-inch long by a slightly over 1-inch diameter steel sampling tool and may be used for sampling to depths of approximately 30 feet. Various liner types are available for use with this sampler, and include: plastic, brass, stainless steel, and teflon. The metal liners are available in segmented 6-inch lengths. The sampler is designed for discrete interval sampling and is not affected significantly by borehole wall collapse. This sampler is similar to a piston sampler where a retractable drive (piston) point is withdrawn when the targeted sampling interval is achieved and the soil sample enters the sampler. Once the sampler is removed from the ground, the inserted liner containing the soil sample is extracted from the sampler and the soil sample is then cut from or extracted from the liner. The segmented liner materials and discrete interval sampling capability gives this device greater suitability for collection of smaller volume soil samples (320 ml).

1.3 Quality Assurance Planning Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the QAPP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

1.4 Health and Safety Considerations

The health and safety considerations for the site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP). All field activities will be conducted in conformance to this HASP. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 Project Geologist/Engineer

It will be the responsibility of the project geologist/sampling engineer to conduct subsurface soil sampling in a manner which is consistent with this SOP. The project geologist/sampling engineer will observe all activities pertaining to subsurface soil sampling to ensure that the SOP is followed, and to record all pertinent data onto a boring log. It is also the project geologist/sampling engineer's responsibility to indicate the specific targeted sampling depth or sampling interval to the drilling subcontractor. The project geologist/sampling engineer is also responsible for the collection of representative environmental or stratigraphic characterization samples once the sampling device has been retrieved and opened. Additional sample collection responsibilities include labeling, handling, and storage of samples until further chain-of-custody procedures are implemented.

2.2 Drilling Subcontractor

It will be the responsibility of the drilling subcontractor to provide the necessary Geoprobe™ equipment for obtaining subsurface soil samples. This generally includes the truck or ATV-mounted percussion/probing machine and one or more Macro-Core and Large Bore samplers in good operating condition, appropriate liners, and other necessary equipment for borehole preparation and sampling. It is the drilling subcontractor's responsibility to provide and maintain their own boring logs if

desired. Equipment decontamination materials should also be provided by the subcontractor and should meet project specifications.

3.0 REQUIRED MATERIALS

In addition to those materials provided by the subcontractor, the project geologist/sampling engineer will require:

- Project Sampling Plan, QAPP, and HASP
- Boring Logs
- Teaspoon or spatula
- Sample kit (bottles, labels, custody records and tape, cooler)
- Sample collection pan
- Folding rule or tape measure
- Utility knife
- Equipment decontamination materials (as required by QAPP)
- Health and safety equipment (as required by HASP)
- Field project notebook/pen

Sampling equipment which comes in direct contact with environmental samples during the sample collection process should be constructed of stainless steel, teflon, or glass, unless specified otherwise in the Project Sampling Plan or QAPP.

4.0 METHOD

4.1 General Method Description

Geoprobe™ soil sampling methods generally involve collection of soil samples by driving the sampling tool directly into the ground using the percussion/probing machine and without the aid of hollow-stem augers or other casing-installed drilling methods. Both the Macro-Core and Large Bore soil samplers consist of metal tubes of seamless construction which can not be split apart like split-spoons. Liner/sleeve inserts are required in order to extract an intact soil core/sample from the sampling device.

Both sampling devices operate by being directly pushed/hammered into the ground by the percussion/probing machine. The borehole is created as the sampling device is advanced downward. The Macro-Core Sampler collects samples continuously and requires that an open borehole be maintained for efficient sample recovery. The Large Bore Sampler contains a piston tip/drive point which allows for advancing the sampler to a designated depth for discrete interval sampling. The piston tip is retracted when the desired sampling interval is reached.

When the soil sampling device is retrieved from the borehole, the drive head, cutting shoe and/or piston assembly is removed, and the liner insert with sample is removed from the sampling device. The project geologist/sampling engineer is then given access to the sample for whatever purpose is required.

Table 1 summarizes the construction characteristics and sampling attributes of each type of sampler. The appropriate type of sampler should be selected based on project-specific sampling requirements.

4.2 Equipment Decontamination

Each sampling device must be decontaminated prior to its initial use and following collection of each soil sample, especially if sampling for analytical testing purposes is conducted. If sampling for soil logging only is conducted, thorough sampler decontamination between samples may not be necessary although sufficient cleansing is necessary for the sampler to operate properly. Site-specific requirements for equipment decontamination should be outlined in the Project Sampling Plan. Equipment decontamination procedures are also outlined within SOP 7600 - Decontamination of Equipment.

4.3 Sampling Procedures - Macro-Core Sampler

(Note: These procedures are excerpted from Geoprobe™ Systems literature. This SOP assumes that the subcontractor will perform sampling; therefore, detailed procedures regarding sample aquisition are not provided.)

4.3.1 Sampler Preparation

- Decontaminate the sampler parts (cutting shoe, sample tube, liners) before assembly.
- Assemble the sampler by first placing the liner over the inside end of the cutting shoe, then inserting the liner/shoe assembly into the sample tube, and then finally threading the cutting shoe into the sample tube. Tighten the cutting shoe with the shoe wrench.
- Thread the sampler onto the drive head.

4.3.2 Sampling

- Using the percussion/probing machine, drive the sampler into the ground until the drive head reaches the ground surface.
- For deeper samples, the borehole walls must remain stable. The cutting shoe is designed with a tapered surface to limit sidewall scraping. Add additional probe rods until the sampler reaches the

targeted sample interval, then drive the sampler through the desired sample interval.

- Use the machine hydraulics to pull the sampler from the borehole.

4.3.3 Sample Recovery

- Once the sampler has been removed from the borehole, the sampler must be unthreaded from the drive head, the cutting shoe unthreaded from the sampler, and the liner/shoe assembly removed from the sample tube.

- Disconnect the cutting shoe from the liner which contains the soil sample. The recovered soil sample may now be viewed, logged, and extracted from the liner for analysis (refer to Section 4.5 for sample containment procedures).

4.4 Sampling Procedures - Large Bore Sampler

(Note: These procedures are excerpted from Geoprobe™ Systems literature. This SOP assumes that the subcontractor will perform sampling; therefore, detailed procedures regarding sample aquisition are not provided. Additional detailed sampling procedures for this specific item of equipment is presented in Geoprobe™ Technical Bulletin No.93-660, appended to this SOP.)

4.4.1 Sampler Preparation

- Decontaminate the sampler parts (cutting shoe, piston rod/tip, sample tube, liners) before assembly.

- Assemble the sampler by first placing the liner on the cutting shoe, then threading the liner/shoe assembly into the sample tube, then connecting the piston tip to the piston rod, and then finally inserting the piston tip/rod assembly into the sample tube. Tighten the cutting shoe with the shoe wrench.

- Thread the sampler onto the drive head. Thread the stop-pin onto the drive head (stop-pin holds the piston tip/rod in place while driving the sampler to the desired sample interval).

4.4.2 Sampling

- Using the percussion/probing machine, drive the sampler into the ground until the upper portion of the targeted sampling interval is achieved.

- Unthread and remove the stop-pin from the drive head using extension rods. This will activate the piston tip/rod.

- Drive the sampler through the targeted sampling interval to collect the sample. The piston tip/rod will retract as the sample enters the sample tube.

- Use the machine hydraulics to pull the sampler from the ground.

4.4.3 Sample Recovery

- Once the sampler has been removed from the ground, the sampler must be unthreaded from the drive head, then the cutting shoe unthreaded from the sample tube, and the liner/shoe assembly removed from the sample tube.

- Disconnect the cutting shoe from the liner which contains the soil sample. The recovered soil sample may now be viewed, logged, and extracted from the liner for analysis (refer to Section 4.5 for sample containment procedures).

4.5 Sample Containment

4.5.1 General

- The soil sample can be removed from the liner following viewing and/or logging. Non-segmented plastic or teflon liners should be cut with a utility knife into approximate 6-inch lengths to facilitate sample extraction or to isolate specific sample zones targeted for analysis. Segmented metal liners can be manually separated.

- Once the liner has been separated, the soil sample may be extracted from the individual liner segments with a spoon or spatula. Except for volatile organic samples (see below), the soil sample should be placed into a sample collection pan and homogenized. Place the sample directly into the required sample container.

- Once filled, the sample container should be properly capped, cleaned and labeled. Sample chain-of-custody and preservation procedures should then be initiated.

- Perform equipment decontamination following containment of the sample.

4.5.2 Volatile Organic Samples

- Use of teflon liners is preferred when sampling for analysis of volatile organic compounds (VOC) because these liners are more inert. In order to limit the potential for loss of volatiles, the soil sample should be removed from the liner as soon as possible after sample recovery. VOC soil samples should be selected from a central point within the liner unless another specific sample zone has been targeted. The liner should be cut with a knife and the sample immediately extracted

and containerized. Clean and label the container and place it into a cooler immediately. Residual sample may then be used to fill other sample or logging requirements.

5.0 QUALITY CONTROL

Quality control requirements are dependent on project-specific sampling objectives. The QAPP will provide requirements for equipment decontamination (frequency and materials), sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

6.0 DOCUMENTATION

Various forms are required to ensure that adequate documentation is made of sample collection activities. These forms include:

- Boring logs
- Field log books
- Sample collection records
- Chain-of-custody records
- Shipping labels

Boring logs (Figure 3) will provide visual and descriptive information for each sample collected and are often the most critical form of documentation generated during a soil sampling program. The field log book is kept as a general log of activities and should not be used in place of the boring log. Occasionally, sample collection records are used to supplement boring logs, especially for environmental samples which have been collected for laboratory analysis. Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes. Shipping labels are required if sample coolers are to be transported to the laboratory by a third party (courier service). Original copies of these records should be maintained in the appropriate project files.

7.0 REFERENCES

Geoprobe™ Systems, August 1993, "1993-94 Equipment and Tools Catalog".

TABLE 1
Geoprobe Systems Soil Sampler Characteristics

Sampler Type	Length (in.)	Diameter (in.)	Volume (ml)	Sleeve Liner Type	Suitability[1]			
					Soil Logging	Physical Testing	Chemical-Inorganics	Chemical-Organics
Macro-Core	45	1.5	1,300	Acetate	A	A	A	B
				Stainless Steel	B	A	B	A
				Teflon	A	A	A	A
Large Bore	22	1.06	320	Acetate	A	A	A	B
				Brass	B	A	B	B
				Stainless Steel	B	A	B	A
				Teflon	A	A	A	A

[1] A - Preferred suitability
B - Acceptable suitability

Figure 1 – Soil Sampling Tools – Macro-Core Sampler - Parts

SOIL SAMPLING TOOLS - Macro-Core Sampler - Parts

Macro-Core Sampler

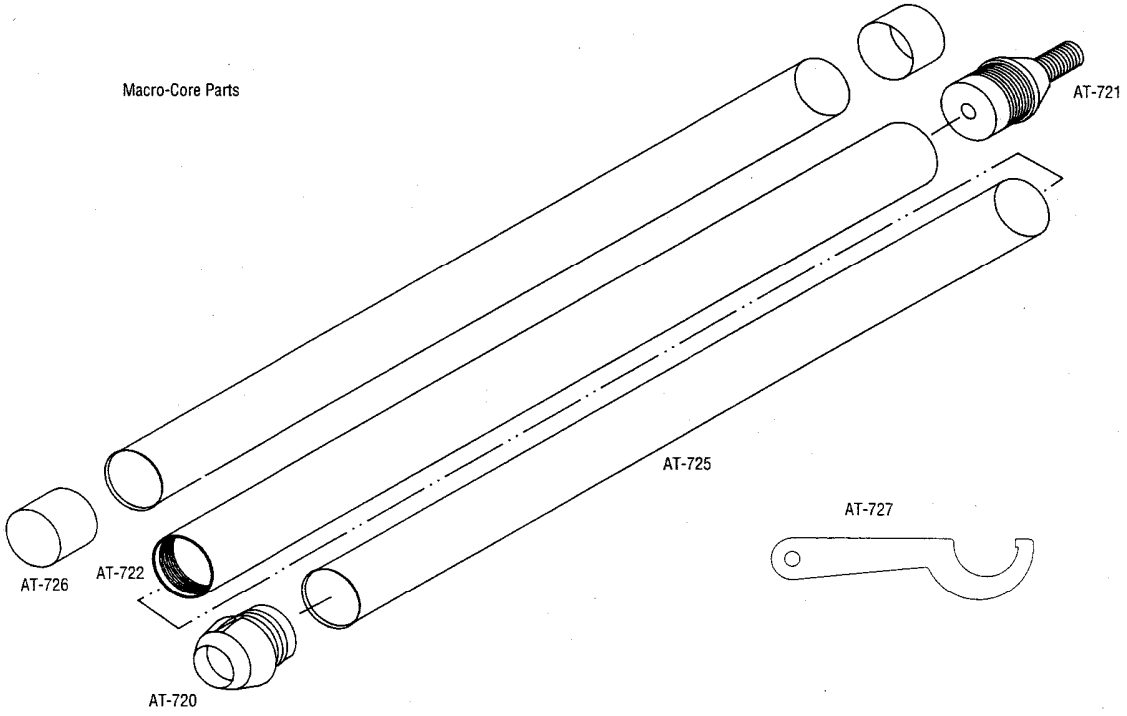
AT-720 Series

The sampler features a nickel-plated sample tube that is 48" long x 2.0" in diameter, a hardened tool steel cutting shoe that has a 1.5" diameter opening, and a tapered drive head that fits standard Geoprobe probe rods. The overall length assembled is 51.25". Sample recovery is 45" long x 1.50" diameter (1302 ml) in a PETG liner.

PARTS	
AT-720	MC Cutting Shoe
AT-721	MC Drive Head
AT-722	MC Sample Tube
AT-725	MC PETG (clear plastic) Liner
AT-726	MC Vinyl End Cap
AT-727	MC Shoe Wrench

KITS
Assembled Macro-Core Sampler*
Part No. AT-720K
Includes the following parts:
(1) **AT-720** MC Cutting Shoe
(1) **AT-721** MC Drive Head
(1) **AT-722** MC Sample Tube
*kit does not include liners and end caps

LINERS	
AT-725K	MC PETG Liners (pre-flared, clear plastic) Box of 66 only
AT-726K	MC Vinyl End Caps (fit AT-725 liners) Box of 66 pairs (66 red/66 black)



The Tools for Site Investigation **4.16**

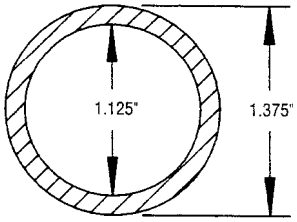
Figure 2 – Soil Sampling Tools – Probe Drive System/Large Bore

SOIL SAMPLING TOOLS - Probe Drive System/Large Bore

Large Bore Sampler

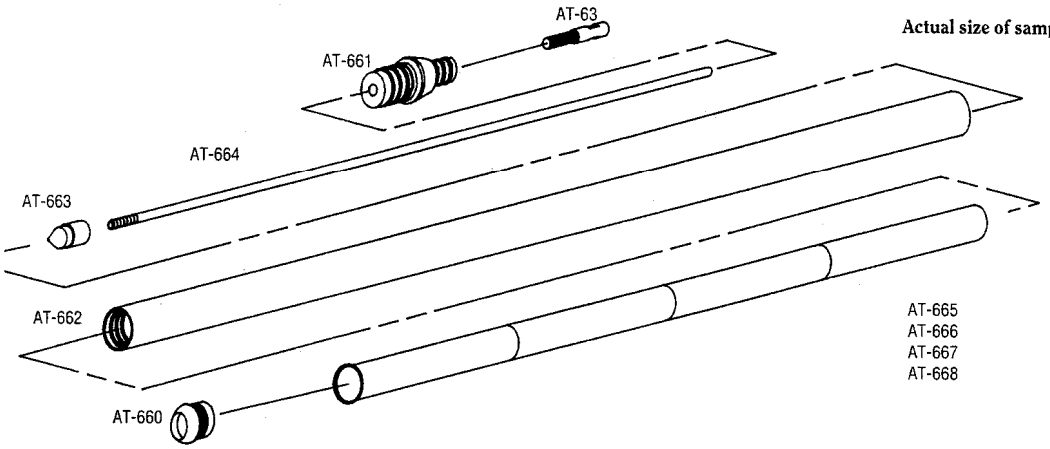
AT-660 Series

Features nickel plated sample tube, replaceable hardened tool steel cutting shoe and removable liner. Recovers core approximately 22" long x 1-1/16" diameter (320 ml). Uses STD Piston Stop-Pin. Recommended for sampling depths up to 30 feet. Liners available in brass, stainless steel, PTFE (Teflon), or PETG (clear plastic).



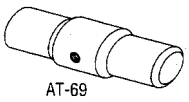
Actual size of sample tube

Large Bore Sampler Parts:



PARTS

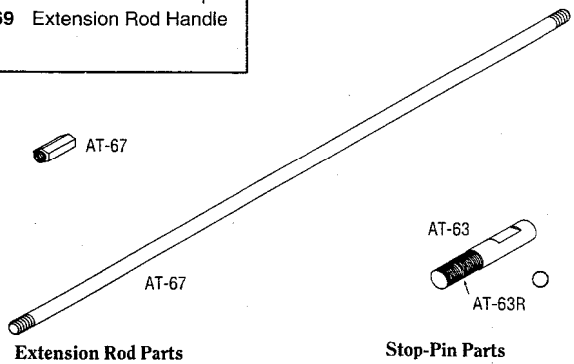
Large Bore Sampler Parts		Liners		Also required:	
AT-660	LB Cutting Shoe	**AT-665**	LB PETG Liner	**AT-63**	STD Stop-Pin
AT-661	LB Drive Head	**AT-666**	LB Brass Liner	**AT-63R**	Stop-Pin O-rings
AT-662	LB Sample Tube	**AT-667**	LB Stainless Liner	**AT-67**	Extension Rod
AT-663	LB Piston Tip	**AT-668**	LB PTFE Liner	**AT-68**	Extension Rod Coupler
AT-664	LB Piston Rod			**AT-69**	Extension Rod Handle
AT-669	LB Shoe Wrench				



PARTS

Parts used with all Probe-Drive Samplers

AT-63	STD Stop-Pin
AT-63R	Stop-Pin O-rings
AT-67	Extension Rod
AT-68	Extension Rod Coupler
AT-69	Extension Rod Handle



Extension Rod Parts

Stop-Pin Parts

Geoprobe Systems

Figure 3 – Boring Log

ENSR

BORING LOG

Sheet 1 of

Project No. _____ Date - Start _____ Finish _____ Boring _____

Project Name _____ Drilling Co. _____

Location _____ Drilling Method _____

Total Depth _____ Inspector _____ Reviewer _____

Remarks _____

Depth Feet	Sample				Graphic Log	Lithologic Description	Equipment Installed
	Type & No.	Blows per 6 In.	Depth Range	Rec.			

Attachment K

EPA Region 2 Low Flow Sampling SOP

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION II

GROUND WATER SAMPLING PROCEDURE
LOW STRESS (Low Flow) PURGING AND SAMPLING

I. SCOPE & APPLICATION

This Low Stress (or Low-Flow) Purging and Sampling Procedure is the EPA Region II standard method for collecting low stress (low flow) ground water samples from monitoring wells. Low stress Purging and Sampling results in collection of ground water samples from monitoring wells that are representative of ground water conditions in the geological formation. This is accomplished by minimizing stress on the geological formation and minimizing disturbance of sediment that has collected in the well. The procedure applies to monitoring wells that have an inner casing with a diameter of 2.0 inches or greater, and maximum screened intervals of ten feet unless multiple intervals are sampled. The procedure is appropriate for collection of ground water samples that will be analyzed for volatile and semi-volatile organic compounds (VOCs and SVOCs), pesticides, polychlorinated biphenyls (PCBs), metals, and microbiological and other contaminants in association with all EPA programs.

This procedure does not address the collection of light or dense non-aqueous phase liquids (LNAPL or DNAPL) samples, and should be used for aqueous samples only. For sampling NAPLs, the reader is referred to the following EPA publications: DNAPL Site Evaluation (Cohen & Mercer, 1993) and the RCRA Ground-Water Monitoring: Draft Technical Guidance (EPA/530-R-93-001), and references therein.

II. METHOD SUMMARY

The purpose of the low stress purging and sampling procedure is to collect ground water samples from monitoring wells that are representative of ground water conditions in the geological formation. This is accomplished by setting the intake velocity of the sampling pump to a flow rate that limits drawdown inside the well casing.

Sampling at the prescribed (low) flow rate has three primary benefits. First, it minimizes disturbance of sediment in the bottom of the well, thereby producing a sample with low turbidity (i.e., low concentration

of suspended particles). Typically, this saves time and analytical costs by eliminating the need for collecting and analyzing an additional filtered sample from the same well. Second, this procedure minimizes aeration of the ground water during sample collection, which improves the sample quality for VOC analysis. Third, in most cases the procedure significantly reduces the volume of ground water purged from a well and the costs associated with its proper treatment and disposal.

III. ADDRESSING POTENTIAL PROBLEMS

Problems that may be encountered using this technique include a) difficulty in sampling wells with insufficient yield; b) failure of one or more key indicator parameters to stabilize; c) cascading of water and/or formation of air bubbles in the tubing; and d) cross-contamination between wells.

Insufficient Yield

Wells with insufficient yield (i.e., low recharge rate of the well) may dewater during purging. Care should be taken to avoid loss of pressure in the tubing line due to dewatering of the well below the level of the pump's intake. Purging should be interrupted before the water level in the well drops below the top of the pump, as this may induce cascading of the sand pack. Pumping the well dry should therefore be avoided to the extent possible in all cases. Sampling should commence as soon as the volume in the well has recovered sufficiently to allow collection of samples. Alternatively, ground water samples may be obtained with techniques designed for the unsaturated zone, such as lysimeters.

Failure to Stabilize Key Indicator Parameters

If one or more key indicator parameters fails to stabilize after 4 hours, one of four options should be considered: a) continue purging in an attempt to achieve stabilization; b) discontinue purging, do not collect samples, and document attempts to reach stabilization in the log book; c) discontinue purging, collect samples, and document attempts to reach stabilization in the log book; or d) Secure the well, purge and collect samples the next day (preferred). The key indicator parameter for samples to be analyzed for VOCs is dissolved oxygen. The key indicator parameter for all other samples is turbidity.

Cascading

To prevent cascading and/or air bubble formation in the tubing, care should be taken to ensure that the flow rate is sufficient to maintain pump suction. Minimize the length and diameter of tubing (i.e., 1/4 or 3/8 inch ID) to ensure that the tubing remains filled with ground water during sampling.

Cross-Contamination

To prevent cross-contamination between wells, it is strongly recommended that dedicated, in-place pumps be used. As an alternative, the potential for cross-contamination can be reduced by performing the more thorough "daily" decontamination procedures between sampling of each well in addition to the start of each sampling day (see Section VII, below).

Equipment Failure

Adequate equipment should be on-hand so that equipment failures do not adversely impact sampling activities.

IV. PLANNING DOCUMENTATION AND EQUIPMENT

- Approved site-specific Field Sampling Plan/Quality Assurance Project Plan (QAPP). This plan must specify the type of pump and other equipment to be used. The QAPP must also specify the depth to which the pump intake should be lowered in each well. Generally, the target depth will correspond to the mid-point of the most permeable zone in the screened interval. Borehole geologic and geophysical logs can be used to help select the most permeable zone. However, in some cases, other criteria may be used to select the target depth for the pump intake. In all cases, the target depth must be approved by the EPA hydrogeologist or EPA project scientist.

- Well construction data, location map, field data from last sampling event.

- Polyethylene sheeting.

- Flame Ionization Detector (FID) and Photo Ionization Detector (PID).

- Adjustable rate, positive displacement ground water sampling pump (e.g., centrifugal or bladder pumps constructed of stainless steel or Teflon). A peristaltic pump may only be used for inorganic sample collection.

- Interface probe or equivalent device for determining the presence or absence of NAPL.

- Teflon or Teflon-lined polyethylene tubing to collect samples for organic analysis. Teflon or Teflon-lined polyethylene, PVC, Tygon or polyethylene tubing to collect samples for inorganic analysis. Sufficient tubing of the appropriate material must be available so that each well has dedicated tubing.

- Water level measuring device, minimum 0.01 foot accuracy, (electronic preferred for tracking water level drawdown during all pumping operations).

- Flow measurement supplies (e.g., graduated cylinder and stop watch or in-line flow meter).

- Power source (generator, nitrogen tank, etc.).
- Monitoring instruments for indicator parameters. Eh and dissolved oxygen must be monitored in-line using an instrument with a continuous readout display. Specific conductance, pH, and temperature may be monitored either in-line or using separate probes. A nephalometer is used to measure turbidity.

- Decontamination supplies (see Section VII, below).

- Logbook (see Section VIII, below).

- Sample bottles.

- Sample preservation supplies (as required by the analytical methods).

- Sample tags or labels, chain of custody.

V. SAMPLING PROCEDURES

Pre-Sampling Activities

1. Start at the well known or believed to have the least contaminated ground water and proceed systematically to the well with the most contaminated ground water. Check the well, the lock, and the locking cap for damage or evidence of tampering. Record observations.

2. Lay out sheet of polyethylene for placement of monitoring and sampling equipment.

3. Measure VOCs at the rim of the unopened well with a PID and FID instrument and record the reading in the field log book.

4. Remove well cap.

5. Measure VOCs at the rim of the opened well with a PID and an FID instrument and record the reading in the field log book.

6. If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Note that the reference point should be surveyed for correction of ground water elevations to the mean geodesic datum (MSL).

7. Measure and record the depth to water (to 0.01 ft) in all wells to be sampled prior to purging. Care should be taken to minimize disturbance in the water column and dislodging of any particulate matter attached to the sides or settled at the bottom of the well.

8. If desired, measure and record the depth of any NAPLs using an interface probe. Care should be taken to minimize disturbance of any sediment that has accumulated at the bottom of the well. Record the observations in the log book. If LNAPLs and/or DNAPLs are detected, install the pump at this time, as described in step 9, below. Allow the well to sit for several days between the measurement or sampling of any DNAPLs and the low-stress purging and sampling of the ground water.

Sampling Procedures

9. Install Pump: Slowly lower the pump, safety cable, tubing and electrical lines into the well to the depth specified for that well in the EPA-approved QAPP or a depth otherwise approved by the EPA hydrogeologist or EPA project scientist. The pump intake must be kept at least two (2) feet above the bottom of the well

to prevent disturbance and resuspension of any sediment or NAPL present in the bottom of the well. Record the depth to which the pump is lowered.

10. Measure Water Level: Before starting the pump, measure the water level again with the pump in the well. Leave the water level measuring device in the well.

11. Purge Well: Start pumping the well at 200 to 500 milliliters per minute (ml/min). The water level should be monitored approximately every five minutes. Ideally, a steady flow rate should be maintained that results in a stabilized water level (drawdown of 0.3 ft or less). Pumping rates should, if needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. As noted above, care should be taken to maintain pump suction and to avoid entrainment of air in the tubing. Record each adjustment made to the pumping rate and the water level measured immediately after each adjustment.

12. Monitor Indicator Parameters: During purging of the well, monitor and record the field indicator parameters (turbidity, temperature, specific conductance, pH, Eh, and DO) approximately every five minutes. The well is considered stabilized and ready for sample collection when the indicator parameters have stabilized for three consecutive readings as follows (Puls and Barcelona, 1996):

    $\pm$0.1 for pH
    $\pm$3% for specific conductance (conductivity)
    $\pm$10 mv for redox potential
    $\pm$10% for DO and turbidity

    Dissolved oxygen and turbidity usually require the longest time to achieve stabilization. The pump must not be removed from the well between purging and sampling.

13. Collect Samples: Collect samples at a flow rate between 100 and 250 ml/min and such that drawdown of the water level within the well does not exceed the maximum allowable drawdown of 0.3 ft. VOC samples must be collected first and directly into sample containers. All sample containers should be filled with minimal turbulence by allowing the ground water to flow from the tubing gently down the inside of the container.

Ground water samples to be analyzed for volatile organic compounds (VOCs) require pH adjustment. The appropriate EPA Program Guidance should be consulted to determine whether pH adjustment is necessary. If pH adjustment is necessary for VOC sample preservation, the amount of acid to be added to each sample vial prior to sampling should be determined, drop by drop, on a separate and equal volume of water (e.g., 40 ml). Ground water purged from the well prior to sampling can be used for this purpose.

14. Remove Pump and Tubing: After collection of the samples, the tubing, unless permanently installed, must be properly discarded or dedicated to the well for resampling by hanging the tubing inside the well.

15. Measure and record well depth.

16. Close and lock the well.

VI. FIELD QUALITY CONTROL SAMPLES

Quality control samples must be collected to determine if sample collection and handling procedures have adversely affected the quality of the ground water samples. The appropriate EPA Program Guidance should be consulted in preparing the field QC sample requirements of the site-specific QAPP.

All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples should be collected during the sampling event:

< Field duplicates
< Trip blanks for VOCs only
< Equipment blank (not necessary if equipment is dedicated to the well)

As noted above, ground water samples should be collected systematically from wells with the lowest level of contamination through to wells with highest level of contamination. The equipment blank should be collected after sampling from the most contaminated well.

VII. DECONTAMINATION

Non-disposable sampling equipment, including the pump and support cable and electrical wires which contact the sample, must be decontaminated thoroughly each day before use ("daily decon") and after each well is sampled ("between-well decon"). Dedicated, in-place pumps and tubing must be thoroughly decontaminated using "daily decon" procedures (see #17, below) prior to their initial use. For centrifugal pumps, it is strongly recommended that non-disposable sampling equipment, including the pump and support cable and electrical wires in contact with the sample, be decontaminated thoroughly each day before use ("daily decon").

EPA's field experience indicates that the life of centrifugal pumps may be extended by removing entrained grit. This also permits inspection and replacement of the cooling water in centrifugal pumps. All non-dedicated sampling equipment (pumps, tubing, etc.) must be decontaminated after each well is sampled ("between-well decon," see #18 below).

17. **Daily Decon**

A) Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.

B) Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Alconox, for 5 minutes and flush other equipment with fresh detergent solution for 5 minutes. Use the detergent sparingly.

C) Rinse: Operate pump in a deep basin of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.

D) Disassemble pump.

E) Wash pump parts: Place the disassembled parts of the pump into a deep basin containing 8 to 10 gallons of non-phosphate detergent solution. Scrub all pump parts with a test tube brush.

F) Rinse pump parts with potable water.

G) Rinse the following pump parts with distilled/ deionized water: inlet screen, the shaft, the suction interconnector, the motor lead assembly, and the stator housing.

H) Place impeller assembly in a large glass beaker and rinse with 1% nitric acid (HNO_3).

I) Rinse impeller assembly with potable water.

J) Place impeller assembly in a large glass bleaker and rinse with isopropanol.

K) Rinse impeller assembly with distilled/deionized water.

18. **Between-Well Decon**

A) Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.

B) Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Alconox, for 5 minutes and flush other equipment with fresh detergent solution for 5 minutes. Use the detergent sparingly.

C) Rinse: Operate pump in a deep basin of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.

D) Final Rinse: Operate pump in a deep basin of distilled/deionized water to pump out 1 to 2 gallons of this final rinse water.

VIII. FIELD LOG BOOK

A field log book must be kept each time ground water monitoring activities are conducted in the field. The field log book should document the following:

- Well identification number and physical condition.
- Well depth, and measurement technique.
- Static water level depth, date, time, and measurement technique.
- Presence and thickness of immiscible liquid layers and detection method.

- Collection method for immiscible liquid layers.
- Pumping rate, drawdown, indicator parameters values, and clock time, at three to five minute intervals; calculate or measure total volume pumped.
- Well sampling sequence and time of sample collection.
- Types of sample bottles used and sample identification numbers.
- Preservatives used.
- Parameters requested for analysis.
- Field observations of sampling event.
- Name of sample collector(s).
- Weather conditions.
- QA/QC data for field instruments.

IX. REFERENCES

Cohen, R.M. and J.W. Mercer, 1993, DNAPL Site Evaluation, C.K. Smoley Press, Boca Raton, Florida.

Puls, R.W. and M.J. Barcelona, 1996, Low-Flow (Minimal Drawdown) Ground-water Sampling Procedures, EPA/540/S-95/504.

U.S. EPA, 1993, RCRA Ground-Water Monitoring: Draft Technical Guidance, EPA/530-R-93-001.

U.S. EPA Region II, 1989, CERCLA Quality Assurance Manual.

Attachment L

ENSR SOP #7220 – Monitoring Well Construction and Installation

SOP NUMBER: 7220

Monitoring Well Construction and Installation

Date:	August 2006
Revision Number:	4
Author:	Charles Martin
Discipline:	Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This SOP provides guidance for installing groundwater monitoring wells. Monitoring wells are installed to monitor the depth to groundwater, to measure aquifer properties, and to obtain samples of groundwater for chemical analysis.

This SOP is applicable to installation of single monitoring wells within a borehole. The construction and installation of nested, multilevel or other special well designs is not covered within this SOP as these type of wells are not frequently constructed. This SOP applies to both overburden and bedrock monitoring wells.

Some states and EPA Regions have promulgated comprehensive guidelines for monitoring well construction and for subsurface investigation procedures. Deviations from this SOP to accommodate other regulatory requirements should be reviewed in advance of the field program, should be explained in the project work plan, and must be documented in the field project notebook when they occur.

1.2 General Principles

Monitoring well construction and installation generally involves drilling a borehole using conventional drilling equipment, installing commercially available well construction and filter/sealing materials, and development of the well prior to sampling. This SOP covers well construction and installation methods only. Borehole drilling and well development methods are covered under SOP-7115 (Subsurface Soil Sampling) and SOP-7221 (Monitoring Well Development), respectively.

1.3 Quality Assurance Planning Considerations

Field personnel should follow specific quality assurance guidelines as outlined in the site-specific QAPP.

The following aspects of monitoring well design and installation procedures depend on project-specific objectives which should be addressed in the QAPP and in the project work plan:

- Borehole drilling method and diameter,
- Type of construction materials for well screen, riser, filter pack and seals,
- Diameter of well materials,
- Length of well screen,
- Location, thickness, and composition of annular seals, and
- Well completion and surface protection requirements.

1.4 Health and Safety Considerations

Monitoring well installation may involve chemical hazards associated with materials in the soil or groundwater being investigated; and always involves physical hazards associated with drilling equipment and well construction methods. When wells are to be installed in locations where the aquifer and/or overlying materials may contain chemical hazards, a Health and Safety Plan (HASP) must be prepared and approved by the Health and Safety Officer before field work commences. This plan must be distributed to all field personnel and must be adhered to as field activities are performed.

2.0 RESPONSIBILITIES

2.1 Drilling Subcontractor

It is the responsibility of the drilling subcontractor to provide the necessary equipment for well construction and installation. Well construction materials should be consistent with project requirements.

2.2 Surveying Subcontractor

It is the responsibility of the surveying subcontractor to provide one or more of the following well measurements as specified in the project work plan: ground surface elevation, horizontal well coordinates, top of well casing elevation (i.e., top-of-casing, or measuring point elevation), and/or top of protective casing elevation.

2.3 Project Geologist/Engineer

It is the responsibility of the Project Geologist/Engineer to directly oversee the construction and installation of the monitoring well by the drilling subcontractor to ensure that the well-installation specifications defined in the project work plan are adhered to, and that all pertinent data are recorded on the appropriate forms.

2.4 Project Manager

It is the responsibility of the Project Manager to ensure that each project involving monitoring well installation is properly planned and executed.

3.0 REQUIRED MATERIAL

3.1 Well Construction Materials

Well construction materials are usually provided by the drilling subcontractor and most often consist of commercially available flush-threaded well screen and riser pipe constructed of PVC or stainless steel with a minimum 2-inch inside diameter. The length of the screen and the size of the screen slots should be specified in the project work plan.

3.2 Well Completion Materials

Well completion materials include silica sand, bentonite, cement, protective casings and locks. Completion materials are generally provided by the drilling subcontractor.

3.3 Other required materials include the following:

- Potable water supply
- Fiberglass or steel measuring tape
- Water level indicator
- Well construction diagrams (Figure 1)
- Waterproof marker or paint (to label wells)
- Health and Safety supplies

- Equipment decontamination materials
- Field project notebook/pen

4.0 METHOD

4.1 General Preparation

4.1.1 Borehole Preparation

Standard drilling methods should be used to achieve the desired drilling/well installation depths specified in the project work plan. Soil sampling, if conducted, should be conducted in accordance with ENSR SOP-7115 (Subsurface Soil Sampling).

The diameter of the borehole must be a minimum of 2 inches greater than the outside diameter of the well screen or riser pipe used to construct the well. This is necessary so that sufficient annular space is available to install filter packs, bentonite seals, and grout seals. Bedrock wells may require reaming after coring in order to provide a large enough borehole diameter for well installation.

Rotary drilling methods requiring bentonite-based drilling fluids, if selected, should be used with caution to drill boreholes that will be used for monitoring well installation. The bentonite mud builds up on the borehole walls as a filter cake and permeates the adjacent formation, potentially reducing the permeability of the material adjacent to the well screen.

If water or other drilling fluids have been introduced into the boring during drilling or well installation, samples of these fluids should be obtained and analyzed for chemical constituents that may be of interest at the site. In addition, an attempt should be made to recover the quantity of fluid or water that was introduced, either by flushing the borehole prior to well installation and/or by overpumping the well during development.

4.1.2 Well Material Decontamination

Although new well materials (well screen and riser pipe) generally arrive at the site boxed and sealed within plastic bags, it is sometimes necessary to decontaminate the materials prior to their use. Well materials should be inspected by the project geologist/engineer upon delivery to check

cleanliness. If the well materials appear dirty, or if local or regional regulatory guidance requires decontamination, then well material decontamination should be performed by the drilling subcontractor in accordance with ENSR SOP-7600 (Decontamination of Equipment).

4.2 Well Construction Procedure

4.2.1 Depth Measurement

Once the target drilling depth has been reached, the drilling subcontractor will measure the total open depth of the borehole with a weighted, calibrated tape measure. Adjustments of borehole depth can be made at this time by drilling further or installing a small amount of sand filter material to achieve the desired depth. If drilling fluids were used during the drilling process, the borehole should be flushed at this time using potable water. The water table depth may also be checked with a water level indicator if this measurement cannot be obtained with the calibrated tape.

4.2.2 Centralizers

In order to install a well which is centered within the borehole, it is recommended that centralizers be used. Centralizers are especially helpful for deep well installations where it may be difficult to position the well by hand. Centralizers may not be necessary on shallow water table well installations where the well completion depth is within 25 feet of the ground surface.

4.2.3 Well Construction

The well screen and riser pipe generally are assembled by hand as they are lowered into the borehole. Before the well screen is inserted into the borehole, the full length of the slotted portion of the well screen as well as the unslotted portion of the bottom of the screen should be measured with the measuring tape. These measurements should be recorded on the well construction diagram.

After the above measurement has been taken, the drilling subcontractor may begin assembling the well. As the assembled well is lowered, care should be taken to ensure that it is centered in the hole if centralizers are not used. The well should be temporarily capped before filter sand and other annular materials are installed.

4.2.4 Filter Sand Installation

The drilling subcontractor should fill the annular space surrounding the screened section of the monitoring well to at least 1 foot above the top of the screen with an appropriately graded, clean sand or fine gravel. In general, the filter pack should not extend more than 3 feet above the top of the screen to limit the thickness of the monitoring zone. If coarse filter materials are used, an additional 1-foot thick layer of fine sand should be placed immediately above the filter pack to prevent the infiltration of sealing components (bentonite or grout) into the filter pack. As the filter pack is placed, a weighted tape should be lowered in the annular space to verify the depth to the top of the layer. Depending upon depth, some time may be required for these materials to settle. If necessary, to eliminate possible bridging or creation of voids, placement of the sand pack may require the use of a tremie pipe. Tremie pipe sandpack installations are generally suggested for deep water table wells and for wells which are screened some distance beneath the water table.

4.2.5 Bentonite Seal Installation

A minimum 2-foot thick layer of bentonite pellets or slurry seal will be installed by the drilling subcontractor immediately above the well screen filter pack in all monitoring wells. The purpose of the seal is to provide a barrier to vertical flow of water in the annular space between the borehole and the well casing. Bentonite is used because it swells significantly upon contact with water. Pellets generally can be installed in shallow boreholes by pouring them very slowly from the surface. If they are poured too quickly, they may bridge at some shallow, undesired depth. As an option, powdered bentonite may be mixed with water into a very thick slurry and a tremie pipe used to inject the seal to the desired depth.

4.2.6 Annular Grout Seal Installation

This grout seal should consist of a bentonite/cement mix with a ratio of bentonite to cement of between 1:5 and 1:20. The grout ratio should be chosen based on site conditions with a higher percentage of bentonite generally used for formations with higher porosity. A mud balance should be used if a specific mud density is required at a particular site. Grout slurry should be pumped into the annular space using a side-discharging tremie pipe located about 2 feet above the sand pack. Side discharge will help preserve the integrity of the sand pack.

In situations where the monitoring well screen straddles the water table, the seal will be in the unsaturated zone and pure bentonites (pellets or powder) will not work effectively as seals without hydration. Dry bentonite may be used if sufficient time to hydrate the seal is allowed. Seal hydration requires the periodic addition of clean water. Optionally, seals in this situation may be a cement/bentonite mixture containing up to 10 percent bentonite by weight. This type of mixture shall be tremied to the desired depth in the borehole.

The borehole annulus will be grouted with seal materials to within 3 feet of the ground surface. Drill cuttings, even those known not to be contaminated, will not be used as backfill material.

4.2.7 Well Completion

The drilling subcontractor will cut the top of the well to the desired height and install a vented (if possible), locking cap. The upper portion of the well casing can optionally be drilled to allow venting. Well casings are usually cut to be a certain height above ground surface (typically 2.5 to 3 feet) or are cut to be flush with the ground surface.

4.2.8 Protective Casing/Concrete Pad Installation

The drilling subcontractor will install a steel guard pipe on the well as a protective casing. The borehole around the guard pipe will be dug out to an approximate 2 to 3-foot radius to a minimum depth of 1 foot at the center and 6 inches at the edges. After installing the protective casing, the excavation will be filled with a concrete/sand mix. The surface of the concrete pad will be sloped so that drainage occurs away from the well. Flush-mount protective casings may not require an extensive concrete pad and should be completed such that they are slightly mounded above the surrounding surface to prevent surface water from running over or ponding on top of the casing. It should be noted, however, that in areas subject to snowfall, flush-mount casings may have to be installed so that they are entirely flush with the ground surface as they may be damaged by snow plows.

Above-ground protective casings should also be vented or should have non-air tight caps. Road box installations should not be vented. Installation of additional guard pipes may be necessary around above-ground well completions in traffic areas. Protective casings should be lockable to prevent unauthorized access.

4.2.9 Well Numbering

The project geologist/engineer will number each well casing with an indelible marker or paint to identify the well. This is particularly important with nested or paired wells to distinguish between shallow and deep wells. The well should be labeled on both the outside of the protective casing and inside beneath the protective casing lid.

4.2.10 Measuring Point Identification

The project geologist/engineer will mark the measuring point from which water level measurements will be made at a specific location along the upper edge of the well casing. PVC wells can easily be notched with a pocket knife or saw. Stainless steel wells (or PVC wells) can be marked with a waterproof marker on the outside of the well casing with an arrow pointing to the measuring point location. The measuring point is the point which will require surveying during the well elevation survey task.

4.2.11 Well Measurements

Upon completion, the following well measurements should be taken by the project geologist/engineer and recorded on the well construction diagram (Figure 1):

- Depth to static water level if water level has stabilized,
- Total length of well measured from top-of-well casing,
- Height of well casing above ground surface,
- Height of protective casing above ground surface,
- Depth of bottom of protective casing below ground surface (may be estimated).

Well screen filter pack, bentonite seal and annular seal thicknesses and depths should also be recorded on the well construction diagram.

4.2.12 Disposal of Drilling Wastes

Drill cuttings and other investigation-derived wastes such as drilling mud or well development/purge water must be properly contained and disposed of. Site-specific requirements for collection and removal of these waste materials should be outlined within the project work plan. Containment of these materials should be performed by the drilling subcontractor.

4.2.13 Well Development

At some point after installation of a well and prior to use of the well for water-level measurements or collection of water quality samples, development of the well shall be undertaken in accordance with ENSR SOP-7221 (Monitoring Well Development). Well development may be performed by the drilling subcontractor if contracted to do so, or by the project geologist/engineer or other project staff.

4.2.14 Well Elevation Survey

At the completion of the well installation program, all monitoring wells are usually surveyed to provide, at a minimum, the top-of-casing measuring point elevation for water level monitoring purposes. Other surveyed points which may be required by the project work plan include: ground surface elevation, top of protective casing elevation, and well coordinate position. Well elevation surveys are usually conducted by a surveying subcontractor.

5.0 QUALITY CONTROL

Certain quality control measures should be taken to ensure proper well completion.

5.1 The borehole will be checked for total open depth, and extended by further drilling or shortened by backfilling, if necessary, before any well construction materials are placed.

5.2 Water level and non-aqueous phase liquid (NAPL) presence will be checked during well installation to ensure that the positions of well screen, sand pack, and seal, relative to water level, conform to project requirements.

5.3 The depth to the top of each layer of packing (i.e., sand, bentonite, grout, etc.) will be verified and adjusted if necessary to conform to project requirements before the next layer is placed.

5.4 If water or other drilling fluids have been introduced into the boring during drilling or well installation, samples of these fluids may be required for analysis of chemical constituents of interest at the site.

6.0 DOCUMENTATION

All well construction data will be recorded on the Monitoring Well Construction Detail form (Figure 1). All wells will be referenced onto the appropriate site map. A field notebook and/or boring log will be used as additional means of recording data. In no case will the notebook or boring log take the place of the well construction diagram.

7.0 TRAINING/QUALIFICATIONS

Well construction and installation requires a moderate degree of training and experience as numerous drilling situations may occur which will require field decisions to be made. It is recommended that inexperienced personnel be supervised for several well installations before working on their own. Experienced drillers are also of great assistance with problem resolution in the field. Field personnel should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous waste materials are considered to be present.

8.0 REFERENCES

1. Standard References for Monitoring Wells, Massachusetts Department of Environmental Protection, WSC-310-91, 1991.

APPENDIX: DEFINITIONS

Annulus: The measured width between the borehole wall and the outside of the well screen or riser pipe.

Bentonite Seal: A granular, chip, or pellet-size bentonite material that is often used to provide an annular seal above the well screen filter pack. This seal is typically installed dry followed by in-place hydration with or without the addition of water. Hydrated bentonite is sometimes used as a grout seal.

Bottom Cap/Plug: Threaded or slip-on cap placed at the bottom of the well prior to installation. Often serves as a sump for accumulation of silt which settles within the well. The measured length from the lowermost well screen slot to the bottom of the bottom cap is known as the sump or tail pipe portion of the well.

Centralizers: Stainless steel expansion clamps which, when fitted to well screens or riser pipe, expand to contact the borehole walls positioning the well centrally within the open borehole. Centralizers assist with even positioning and distribution of filter pack and sealant materials and assist with maintaining well plumbness.

Expansion Cap/Well Cap: Cap used to cover the opening at the top of the well riser pipe. Expansion caps are equipped with a rubber gasket and threaded wing nut which, when turned, provides a watertight seal. Expansion caps may also be locked, and generally are recommended for use with flush-constructed wells where road box protective casings are also used. Other well caps may include slip-on or threaded caps made of the same material as the well casing.

Filter Pack: A well-graded, clean sand or gravel placed around the well screen to act as a filter in preventing the entry of very fine soil particles into the well.

Grout Seal: A cement/bentonite mixture used to seal a borehole that has been drilled to a depth greater than the final well installation depth or to seal the remaining borehole annulus once the well has been installed. Occasionally, pure cement or pure bentonite is used as a grout seal.

Measuring Point: A selected point at the top of the well casing (riser pipe) used for obtaining periodic water-level measurements. The measuring point should consist of either a notch or indelibly marked point on the upper surface of the casing. Typically, the highest point on the casing (if not level) is used as the measuring point. The measuring point is also the point that is surveyed when well elevation data is obtained.

Protective Casing: A locking metal casing, placed around that portion of the well riser pipe that extends above the ground surface. The protective casing is generally cemented in place when the concrete pad is constructed around the well.

Riser Pipe: The section of unperforated well casing material used to connect the well screen with the ground surface. Frequently, it is made of the same material and has the same diameter as the well screen. Riser pipe is typically available pre-cleaned and pre-threaded for immediate use.

Road Box: A protective casing that is flush-mounted with the ground around a well installation. Road boxes are used in areas where the monitoring well cannot extend above the ground surface for traffic or security reasons. Road boxes usually require a special key to open.

Tremie Pipe: A small diameter pipe which fits in the open borehole annulus and is used to inject filter sands or hydrated seal materials under pressure.

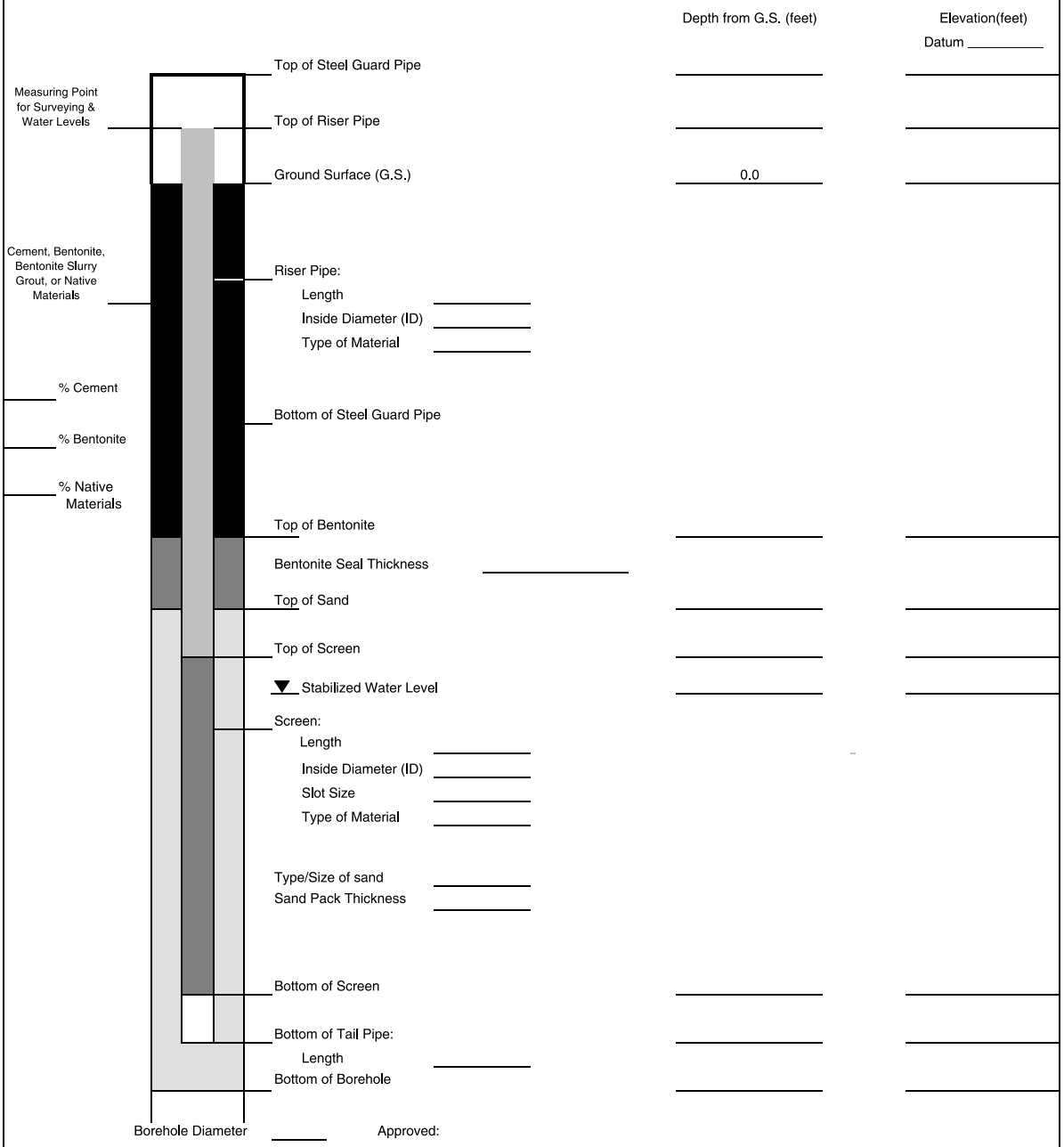
Well Screen: That portion of the well casing material that is perforated in some manner so as to provide a hydraulic connection to the aquifer. Typically a well screen is purchased pre-slotted, pre-cleaned, and pre-threaded for immediate use.

Vent Hole: Small diameter hole drilled in the upper portion of the well riser pipe which provides atmospheric venting of the well. Allows for constant equilibration of the water level with changing atmospheric conditions. In flood-prone areas, or with flush-mount wells, vent holes should not be used.

Figure 1 Monitoring Well Construction Detail

ENSR	Client:	WELL ID:
	Project Number:	
	Site Location:	Date installed:
	Well Location: Coords:	Inspector:
	Method:	Contractor:

MONITORING WELL CONSTRUCTION DETAIL



Attachment M

ENSR SOP #7221 – Monitoring Well Development

SOP NUMBER: 7221

Monitoring Well Development

Date: August 2006
Revision Number: 2
Author: Charles Martin
Discipline: Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This SOP describes the methods used for developing newly installed monitoring wells and/or existing wells which may require redevelopment/rehabilitation. This SOP is applicable to monitoring wells and/or small diameter recovery wells and piezometers.

Monitoring well development and/or redevelopment is necessary for several reasons:

- To improve/restore hydraulic conductivity of the surrounding formations as they have likely been disturbed during the drilling process, or may have become partially plugged with silt,
- To remove drilling fluids (water, mud), when used, from the borehole and surrounding formations, and
- To remove residual fines from well filter materials and reduce turbidity of groundwater, therefore, reducing the chance of chemical alteration of groundwater samples caused by suspended sediments.

Respective state or federal agency (regional offices) regulations may require specific types of equipment for use or variations in the indicated method of well development. Deviations from this SOP to accommodate other regulatory requirements should be reviewed in advance of the field program, should be explained in the project work plan, and must be documented in the field project notebook when they occur.

1.2 General Principles

Well development generally involves withdrawal of an un-specified volume of water from a well using a pump, surge block or other suitable method such that, when completed effectively, the well is in good or restored hydraulic connection with the surrounding water bearing unit and is suitable for obtaining representative groundwater samples or for other testing purposes.

1.3 Quality Assurance Planning Considerations

Field project personnel should follow specific quality assurance guidelines as outlined in the site-specific Quality Assurance Project Plan (QAPP) and/or Sampling Plan. The plan should indicate the preferred method of well development at a particular site based on project objectives, aquifer conditions, and agency requirements. Specific well performance criteria such as low turbidity values to be achieved following well development should also be specified as well as any requirements for collection/containerization and disposal of well development water.

1.4 Health and Safety Considerations

Monitoring well development may involve chemical hazards associated with materials in the soil or aquifer being characterized and may involve physical hazards associated with use of well development equipment. When wells are to be installed and developed on hazardous waste investigation sites, a Health and Safety Plan must be prepared and approved by the Health and Safety Officer before field work commences. This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all field project personnel, and must be adhered to as field activities are performed.

2.0 RESPONSIBILITIES

2.1 Project Geologist/Engineer

Development or oversight of development of new monitoring wells is the responsibility of the project geologist/engineer involved in the original installation of the well. Records of well development methods and results will be retained in the project file.

2.2 Project Manager

The project manager is responsible for ensuring that the appropriate method of well development has been chosen which best meets project objectives, site hydrogeologic conditions, and/or relevant regulatory requirements.

3.0 REQUIRED MATERIALS

Well development can be performed using a variety of methods and equipment. The specific method chosen for development of any given well is governed by the purpose of the

well, well diameter and materials, depth, accessibility, geologic conditions, static water level in the well, and type of contaminants present, if any.

The following list of equipment, each with their own particular application, may be used to develop and/or purge monitoring wells.

3.1 Bailer Purging

A bailer is used to purge silt-laden water from wells after using other devices such as a surge block. In some situations, the bailer can be used to develop a well by bailing and surging, often accompanied with pumping. A bailer should be used for purging in situations where the depth to static water is greater than 25 feet and/or where insufficient hydraulic head is available for use of other development methods.

3.2 Surge Block Development

Surge blocks are commercially available for use with Waterra™-type pumping systems or may be manufactured using a rubber or teflon "plunger" attached to a rod or pipe of sufficient length to reach the bottom of the well. Well drillers usually can provide surge blocks if requested. A recommended design is shown in Figure 1.

3.3 Pump Development

A pump is often necessary to remove large quantities of silt-laden ground water from a well after using the surge block. In some situations, the pump alone can be used to develop the well and remove the fines by overpumping. Since the purpose of well development is to remove suspended solids from a well and surrounding filter pack, the pump must be capable of moving some solids without damage. The preferred pump is a submersible pump which can be used in both shallow and deep ground water situations. A centrifugal pump may be used in shallow wells but will work only where the depth to static ground water is less than approximately 25 feet. Pumping may not be successful in low-yielding aquifer materials or in wells with insufficient hydraulic head.

3.4 Compressed Gas Development

Compressed gas, generally nitrogen from a tank or compressed air through a compressor, can be used to both surge and develop a monitoring well. The method works by injection of compressed gas at the bottom of the water column, driving sediment-laden water to the surface. Compressed gas can also be used for "jetting" - a process by which the gas is directed at the slots in the well screen to cause

turbulence (thereby disturbing fine materials in the adjacent filter pack). Compressed gas is not limited by any depth range.

Since the compressed gas will be used to "lift" water from the monitoring well, provisions must be made for controlling the discharge from contaminated wells. This is generally accomplished by attaching a "tee" discharge to the top of the casing and providing drums to contain the discharged water. Gas-lifting should never be done in contaminated wells without providing a means to control discharge.

3.5 Other Required Materials:

- Well development records (Figure 2)
- Health and Safety equipment
- Equipment decontamination materials
- Water quality instrumentation: nephelometer, pH, temperature, specific conductance meters, as required
- Field project notebook/pen

4.0 METHOD

4.1 General Preparation

4.1.1 Well Records Review: Well completion diagrams should be reviewed to determine well construction characteristics. Formation characteristics should also be determined from review of available boring logs.

4.1.2 Site Preparation: Well development, similar to groundwater sampling, should be conducted in as clean an environment as possible. This usually requires, at a minimum, placing sheet plastic on the ground to provide a clean working area for development equipment.

4.1.3 IDW Containment: Provisions should be in place for collection and management of investigation-derived wastes (IDW), specifically well development water and miscellaneous expendable materials generated during the development process. The collection of IDW in drums or tanks may be required depending on project-specific requirements. The QAPP should specify the requirements for IDW containment.

4.1.4 Water Level/Well Depth Measurement: The water level and well depth should be measured with a water level indicator and written on the well development record. This information is used to calculate the volume of standing water (i.e., the well volume) within the well.

4.1.5 Equipment Decontamination: All down-well equipment should be decontaminated prior to use in accordance with ENSR SOP-7600 (Decontamination of Equipment).

4.1.6 Removal of Drilling Fluids: Drilling fluids such as mud or water, if used during the drilling and well installation process, should be removed during the well development procedure. It is recommended that a minimum of 1.5 times the volume of added fluid be removed from the well during development. Drilling muds should initially have been flushed from the drilling casing during the well installation procedure with water added during the flushing process. If the quantity of added fluid is not known or could not be reasonably estimated, removal of a minimum of 10 well volumes of water is recommended during the development procedure.

4.2 Development Procedures

4.2.1 Development Method Selection

The construction details of each well shall be used to define the most suitable method of well development. Some consideration should be given to the potential degree of contamination in each well as this will impact IDW containment requirements.

The criteria for selecting a well development method include well diameter, total well depth, static water depth, screen length, the likelihood and level of contamination, and characteristics of the geologic formation adjacent to the screened interval.

The limitations, if any, of a specific procedure are discussed within each of the following procedures.

4.2.2 General Water Quality Measurements

Measure and record water temperature, pH, specific conductance, and turbidity periodically during development using the available water quality instruments. These measurements will aid in determining whether well

development is proceeding efficiently, will assist in identifying when well development is complete, will determine whether the development process is effective or not with any given well and, potentially, may identify well construction irregularities (i.e., grout in well, poor well screen slot-size selection). Water quality parameters should be checked a minimum of 3 to 5 times during the development process.

4.2.3 Bailer Procedure

- As stated previously, bailers shall preferably not be used for well development but may be used in combination with a surge block to remove silt-laden water from the well.
- When using a bailer to purge well water; select the appropriate bailer, then tie a length of bailer cord onto the end of it.
- Lower the bailer into the screened interval of the monitoring well. Silt, if present, will generally accumulate within the lower portions of the well screen.
- The bailer may be raised and lowered repeatedly in the screened interval to further simulate the action of a surge block and pull silt through the well screen.
- Remove the bailer from the well and empty it into the appropriate storage container.
- Continue surging/bailing the well until sediment-free water is obtained. If moderate to heavy siltation is still present, the surge block procedure should be repeated and followed again with bailing.
- Check water quality parameters periodically.

4.2.4 Surge Block Procedure

- A surge block effectively develops most monitoring wells. This device first forces water within the well through the well screen and out into the formation, and then pulls water back through the screen into the well along with fine soil particles. Surge blocks may be manufactured to meet the design criteria shown in the example (Figure 1) or may be

purchased as an adaptor to fit commercially available well purging systems such as the Waterra system.

- Insert the surge block into the well and lower it slowly to the level of static water. Start the surge action slowly and gently above the well screen using the water column to transmit the surge action to the screened interval. A slow initial surging, using plunger strokes of approximately 3 feet, will allow material which is blocking the screen to separate and become suspended.

- After 5 to 10 plunger strokes, remove the surge block and purge the well using a pump or bailer. The returned water should be heavily laden with suspended silt and clay particles. Discharge the purged water into the appropriate storage container.

- Repeat the process. As development continues, slowly increase the depth of surging to the bottom of the well screen. For monitoring wells with long screens (greater than 10 feet) surging should be undertaken along the entire screen length in short intervals (2 to 3 feet) at a time. Continue this cycle of surging and purging until the water yielded by the well is free of visible suspended material.

- Check water quality parameters periodically.

4.2.5 Pump Procedure

- Well development using only a pump is most effective in monitoring wells that will yield water continuously. Theoretically, pumping will increase the hydraulic gradient and velocity of groundwater near the well by drawing the water level down. The increased velocity will move residual fine soil particles into the well and clear the well screen of this material. Effective development cannot be accomplished if the pump has to be shut off to allow the well to recharge.

- When using a submersible pump or surface pump, set the intake of the pump or intake line in the center of the screened interval of the monitoring well.

- Pump a minimum of three well volumes of water from the well and raise and lower the pump line through the screened interval to remove any silt/laden water.

- Continue pumping water from the well until sediment-free water is obtained. This method may be combined with the manual surge block method if well yield is not rapid enough to extract silt from the surrounding formations.

- Check water quality parameters periodically.

4.2.6 Compressed Gas Procedure

- Although the equipment used to develop a well using this method is more difficult to obtain and use, well development using compressed gas is considered to be a very effective method. This method is also not limited by well depth, well diameter, or depth to static water. Caution must be exercised, however, in highly permeable formations not to inject gas into the formation. Drilling subcontractors will often provide the necessary materials as well as perform this method, if requested. When using a compressor, an oil-less compressor should be used, or an oil trap/filter should be placed on the air discharge line which enters the well.

- Lower the gas line into the well, setting it near the bottom of the screened interval. Install the discharge control equipment (i.e., tee fitting) at the well head.

- Set the gas flow rate to allow continuous discharge of water from the well.

- At intervals during gas-lifting, especially when the discharge begins to contain less suspended material , shut off the air flow and allow the water in the well to backflush through the screened interval to disturb any bridging that may have occurred. Re-establish the gas flow when the water level in the well has returned to the pre-development level.

- Continue gas-lifting and/or jetting until the discharged water is free from suspended material.

- Check water quality parameters periodically.

5.0 QUALITY CONTROL

A well has been successfully developed when one or more of the following criteria are met:

- The sediment load in the well has been eliminated or greatly reduced. Regulatory requirements may be in place which state that water turbidity values ranging from 5 to 50 NTU must be achieved at the end of the development procedure. Use of a nephelometer is required during the well development procedure to measure water turbidity if meeting a specific turbidity value is required by the regulations. Attaining low turbidity values in fine-grained formations may be difficult to achieve.

- Permeability tests conducted in accordance with ENSR SOP-7720 (Hydraulic Conductivity Testing) yield repeatable hydraulic conductivity values.

6.0 DOCUMENTATION

The Monitoring Well Development Record (Figure 2) will be completed by the geologist or hydrogeologist conducting the development. In addition, a field project notebook should be maintained detailing any problems or unusual conditions which may have occurred during the development process.

7.0 TRAINING/QUALIFICATIONS

Well development procedures vary in complexity. It is recommended that initial development attempts be supervised by more experienced personnel. Field personnel should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous waste materials are considered to be present.

8.0 REFERENCES

Standard References for Monitoring Wells, Massachusetts Department of Environmental Protection, WSC-310-91, 1991.

APPENDIX: DEFINITIONS

Bridging: A condition within the filter pack outside the well screen whereby the smaller particles are wedged together in a manner that causes blockage of pore spaces.

Hydraulic Conductivity: a characteristic property of aquifer materials which describes the permeability of the material with respect to flow of water.

Hydraulic Connection: A properly installed and developed monitoring well should have good hydraulic connection with the aquifer. The well screen and filter material should not provide any restriction to the flow of water from the aquifer into the well.

Permeability Test: Used to determine the hydraulic conductivity of the aquifer formation near a well screen. Generally conducted by displacing the water level in a well and monitoring the rate of recovery of the water level as it returns to equilibrium. Various methods of analysis are available to calculate the hydraulic conductivity from these data.

Static Water Level: The water level in a well that represents an equilibrium or stabilized condition, usually with respect to atmospheric conditions in the case of monitoring wells.

Well Surging: That process of moving water in and out of a well screen to remove fine sand, silt and clay size particles from the adjacent formation.

Well Purging: The process of removing standing water from a well to allow surrounding formation water to enter the well.

Well Screen: That portion of the well casing material that is perforated in some manner so as to provide a hydraulic connection to the aquifer. The perforated, or slotted, portion of a well is also known as the screened interval.

Figure 1
Recommended Surge Block Design

SURGE BLOCK DESIGN
(Not to Scale)

Steel washers should be 1/2" to 3/4" smaller in diameter than the well ID. Gasket can be rubber or leather and should be the same diameter or 1/8" smaller than the well ID to compensate for swelling of the leather/ Rod can be steel, fiberglass, or plastic but must be strong and lightweight.

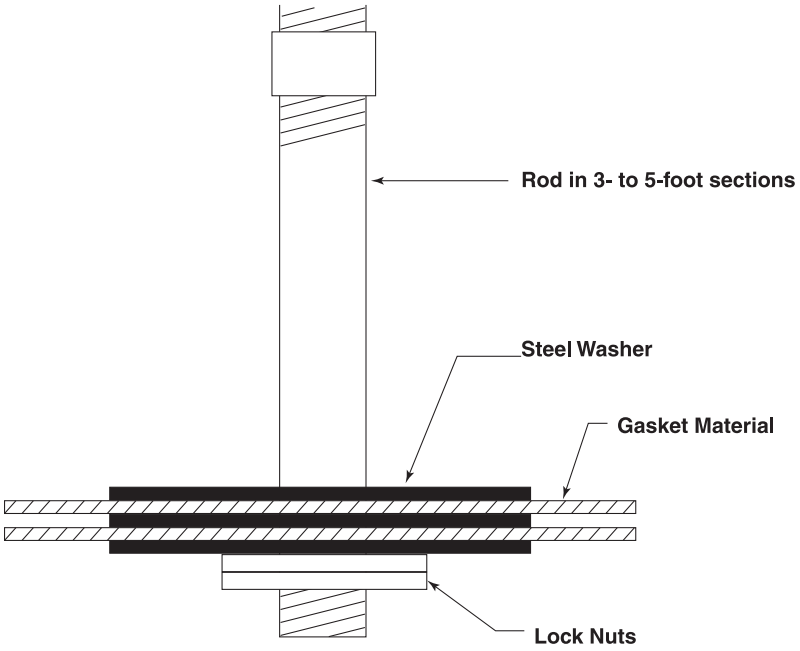


Figure 2
Well Development Record

ENSR

MONITORING WELL DEVELOPMENT RECORD

DATE: _____ WELL I.D.: _____

PROJECT NAME: _____ LOCATION: _____

PROJECT NUMBER: _____ DEVELOPER: _____

☐ ORIGINAL DEVELOPMENT ☐ REDEVELOPMENT ORIGINAL DEVELOPMENT DATE: _____

WELL DATA

Well Diameter: _____

Total Well Depth: _____

Depth to Top of Screen: _____

Depth to Bottom of Screen: _____

Depth to Static Water Level: _____

Geology at Screened Interval:

Likely Contaminants:

Purge Water & Sediment Disposal Method:

DEVELOPMENT METHOD	PURGE METHOD	PERMEABILITY TEST RESULTS

ACCEPTANCE CRITERIA

Signature: _____ Date: _____

Attachment N

ENSR SOP #7230 – Test Pit/Trench Subsurface Exploration

SOP NUMBER: 7230

Test Pits/Trench Subsurface Exploration

Date:	October 2002
Revision Number:	2
Author:	Scott Olson
Discipline:	Geosciences

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the methods for excavating and logging test pits or trenches. Test pits/trenches are generally excavated to visually determine subsurface soil and rock conditions and for environmental sampling. Test pits/trenches are generally excavated by a qualified subcontractor under the direction of the project geologist.

The purpose of this SOP is to provide a specific method and/or procedure to be used for test pit excavation, soil sample collection, and test pit logging. If followed properly, use of this SOP will promote consistency in each of the above areas.

This SOP is applicable to test pit/trench excavations which are usually completed with a backhoe. Test pits/trenches are generally completed to shallow depths (up to 10 feet approximately), and usually within unconsolidated materials, including but not limited to, native materials (sand, gravel, silt, clay), fill materials, and weathered bedrock.

1.2 General Principles

Test pit/trench subsurface explorations generally involve use of backhoes to perform excavations for the purpose of visually assessing subsurface soil/fill conditions and to allow for collection of representative soil samples. The excavation subcontractor is directed by the project geologist/engineer to complete a test pit/trench at a designated location. The lateral extent and depth of the test pit/trench is dependent upon project objectives. Once excavated, the test pit/trench is logged and sampled, if required. Following this, the test pit/trench is backfilled with the excavated material or with clean fill.

1.3 Quality Assurance Planning Considerations

Project personnel should follow specific quality assurance guidelines for sampling as outlined in the site-specific Quality Assurance Project Plan (QAPP) and/or Sampling Plan. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance

requirements typically suggest the collection of a sufficient quantity of field duplicate, field blank, and equipment blank samples.

1.4 Health and Safety Considerations

All utilities (electric, water, sewer, etc.) or property owners who may have equipment or transmission lines buried in the vicinity of proposed test pits should be notified. Many regions have organizations that represent all utilities for these notification purposes. Sufficient time should be allowed after notification (typically 3 working days) for the utilities to respond and mark locations of any equipment that may be buried on site. It should be noted, however, that these organizations may not be responsible for locating utilities on private property. This is often the responsibility of the property owner. The estimated location of utility installations, such as sewer, telephone, electric, water lines and other underground installations that may reasonably be expected to be encountered during excavation work, shall be verified by the site owner prior to opening an excavation. The subcontractors will be made aware of the potential of encountering underground utilities at each test pit location.

To avoid the hazards associated with the cave-in or collapse of an excavation or trench, ENSR employees will not enter an excavation or trench to collect the required samples. Samples will be collected remotely, using long-handled sampling tools, or directly from the bucket of the backhoe. If entry becomes necessary and the excavation is greater than 5 feet in depth, the contractor will be required to slope or shore the walls of the excavation. Specific requirements will depend on soil type and site constraints and will be addressed in the site-specific health and safety plan (HASP). All sloping or shoring must be conducted in compliance with OSHA's rules for trenching and excavation (29 CFR 1926.650-652.)

For safety reasons in case of sidewall collapse, all personnel and materials will be kept at least 2 feet from the edge of any open excavation. Open excavations can be viewed by the geologist from test pit endwalls which are more stable than test pit sidewalls.

If excavations are to be left open temporarily, the perimeter of the excavation must be marked with "Caution-Open Trench" tape. Other site-specific restrictions on leaving test pits open temporarily may be required by the property owner. Those requirements should be determined prior to startup of the excavation program.

Ambient air quality conditions should be periodically monitored both within and surrounding the excavation for potentially toxic and/or explosive atmospheric conditions. The project HASP should be reviewed for specific information regarding ambient air quality monitoring.

2.0 RESPONSIBILITIES

2.1 Project Manager

The project manager will be responsible for ensuring that the project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the work in accordance with this SOP and the project plan.

2.2 Project Geologist/Engineer

It will be the responsibility of the geologist or engineer to determine the location, total depth and overall size of each test pit/trench. It will also be his or her responsibility to collect representative samples from the test pit/trench and to log the test pit/trench according to the procedures described in this SOP.

2.3 Subcontractor

It will be the responsibility of subcontractors to construct test pits/trenches according to ENSR project-specific requirements and in accordance with OSHA safety requirements for trench construction.

3.0 REQUIRED MATERIALS

- Stakes
- Fluorescent flagging tape/caution tape
- Sample kit (i.e., bottles/labels, custody records, cooler, ice, etc.)
- Measuring tape
- Compass (optional)
- Camera
- Sheet plastic
- Sampling equipment: spoons, trowels, scoops, shovels
- Field records: test pit log, test pit profile log
- Field logbook/pen
- Project plans (HASP, QAPP, Sampling Plan)
- Decontamination materials and solutions

4.0 METHOD

4.1 General Preparation

General locations for test pits or trenches should be marked with a stake and/or flagging tape prior to start of the excavation program. Final post-excavation locations should be

documented by using topographic maps and/or other site plans. Final locations should also be measured from a fixed feature or surveyed if necessary.

Excavation equipment should be properly decontaminated prior to initial use, between test pit/trench excavations, and following completion of the last excavation. It should be noted that excavation equipment may need to be brushed clean or fully decontaminated at the completed test pit location if the potential exists for spreading contaminated soils by transport of the excavation equipment.

4.2 Excavation

Test pits/trenches will be excavated to the depth specified in the project-specific plan. Test pit completion depths should be indicated to the subcontractor by the project geologist or engineer. The test pits or trenches will be excavated in compliance with applicable safety regulations. Walls should initially be cut as near vertical as possible to facilitate stratigraphic mapping. Proper sidewall sloping will, however, be required for test pits that extend beyond 5 feet in depth if sampling or logging personnel require access to the open excavation.

As the test pit/trench is excavated, the excavated soils should be placed to one side of the excavation and no closer than 2 to 3 feet from the excavation's edge. Depending on the project requirements, sheet plastic may be required to cover the ground surface before placing excavation soils on the ground.

Excavation should proceed slowly and with caution. The project geologist/engineer should view the excavation (from the far end wall) after each removed bucket of soil for the presence of unusual features such as waste accumulations, free liquids (water or free product), and buried utilities. The excavation subcontractor should continue the excavation only after receiving approval to proceed from the project geologist/engineer.

4.3 Logging

A test pit log will be prepared in the field by the geologist or engineer. The test pit log, which is similar to a boring log, will include notations on soil types and depth of stratigraphic changes, depth to water table, identification of waste materials, and the depth/location of any environmental samples that were collected. The dimensions and orientation of each test pit/trench will also be recorded on the test pit log.

A supplemental sketch is often necessary to depict the physical orientation of the strata encountered. These observations should be recorded on the test pit profile log.
The test pit profile log allows for sketching a view of the test pit sidewall (i.e., a test pit cross section) and for listing of sample collection information.

The project geologist/engineer will measure the depth to the groundwater table in test pits, if encountered, only after sufficient time is allowed for stabilization of the groundwater table. If there is insufficient time to achieve stabilization, the depth to where groundwater is entering the test pit should be indicated on the logs.

If photographs are necessary, they can be taken at this time.

4.4 Sample Collection

Requirements for soil sampling will be determined by the project geologist/engineer in accordance with the project sampling plan.

Soil samples may be collected for several reasons including stratigraphic logging, field headspace organic vapor testing, and laboratory environmental testing. Various types of sampling equipment are commonly available for use in sample collection. SOP 7110 (Surface Soil Sampling) provides instruction in the use of scoops, trowels, shovels, and other types of soil sampling equipment. Guidance on decontamination of field equipment is provided in SOP 7600.

Soil samples may be collected from test pits/trenches from several locations: the test pit/trench sidewalls or base, the excavated soil pile, or directly from the backhoe bucket. Additional information regarding each sampling method are presented in the following subsections.

4.4.1 Test Pit/Trench Sidewall or Base Sampling

Test pit/trench sidewall or base sampling is generally the preferred method by regulatory agencies because it allows for in-situ sampling of soils. In-situ sampling limits the potential for sample contamination which can occur during the excavation procedures. This method, valid for any type of proposed analysis, is especially preferred for samples which will be analyzed for volatile organic compounds (VOC).

Sidewall or base sampling is considered to be somewhat more dangerous than sampling from the soil pile or backhoe bucket because it may require entry of sampling personnel into the excavation. A recommended option in place of entry into the excavation is to use long-handled sampling equipment. The use of long-handled sampling equipment allows for collection of samples without entry into the excavation and often from the excavation ends where it is generally considered safe. Long-handled sampling equipment can be fabricated using standard surface soil sampling equipment (trowels, scoops, etc.) attached to long wooden or aluminum extension handles with duct tape or clamps. When using duct tape, or any kind of tape, caution should be exercised during sampling not letting the sample come into contact with the tape or handle.

Regardless of whether entry into the excavation is required, sampling should be conducted in the following manner:

- Select the sampling location and "dress" the excavation surface by scraping to remove any loose surface soil or smearing residues.
- Replace the dressing tool with a clean sampling tool.
- Collect the soil sample with the sampling tool in accordance with the methods outlined within SOP 7110 (Surface Soil Sampling).
- Complete the test pit log and test pit profile log to provide description and location information for each sample collected.

4.4.2 Excavated Soil Pile Sampling

This method is considered favorable for soil logging and headspace VOC testing in the field. It is, however, generally considered unsuitable for collection of samples for laboratory analytical testing for the simple reason that it is difficult to determine the exact position in the test pit/trench from which the sample was obtained.

Sampling from the soil pile is recommended if single or composite soil samples are required for general soil quality testing or when larger quantities of soil are needed for testing. Soil pile sampling is accomplished following the same methods specified in SOP 7110.

4.4.3 Backhoe Bucket Sampling

Sampling from the backhoe bucket is an improvement on soil pile sampling in that the geologist or engineer is reasonably sure of the position where the soil was obtained. Backhoe bucket sampling is considered suitable for soil logging and headspace VOC testing; however, it is generally considered to be unsuitable for analytical testing. Sampling from the backhoe bucket may be considered suitable for analytical testing if, for instance, the base of the test pit is covered with water and use of standard sampling equipment has been unsuccessful in retrieving an unacceptable sample.

Backhoe bucket sampling is accomplished following the same methods specified in SOP 7110. If analytical testing is the objective, however, certain precautions must be taken. The bucket must be free of rust, grease, and paint. Some care is required to obtain a sample which has been minimally disturbed. For example, if a cohesive block of soil is present within the bucket, the soil sample should be retrieved from within the block of soil as much as possible. Only soil that has not been in contact with the backhoe bucket should be taken for analytical testing.

4.4.4 General Sampling Procedures

Representative samples shall be collected for laboratory analysis by the project geologist/engineer using the appropriate equipment.

Sample bottling, handling and transport shall be conducted in accordance with the requirements of the project specific QAPP.

Specific procedures pertaining to the handling and shipment of samples can be found in ENSR SOP 7510 (Packaging and Shipment of Samples).

4.5 Backfilling

Prior to backfilling, all collected information will be reviewed to ensure that all the appropriate and/or required logs, photographs, measurements and samples have been collected.

After review of the records, backfilling and compaction of test pit/trenches will be accomplished according to contract specifications. If excavation sidewalls have been undermined, the excavation may require temporary expansion to backfill properly.

All test pits/trenches will be backfilled to original grade unless otherwise specified.

It should be noted that project-specific requirements may include the use of clean backfill material. The requirements for clean backfill and the potential requirements for disposal of excavated soils should be defined within the project-specific plan.

5.0 QUALITY CONTROL

Quality control requirements for sample collection are dependent on project-specific sampling objectives. The QAPP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

6.0 DOCUMENTATION

Test pit locations shall be referenced on the site map. Sample locations shall be referenced on a plan view/vertical section of each test pit/trench.

Photographs of specific geologic features may be required for documentation purposes. A scale or an item providing a size perspective shall be placed in each photograph. Frame number/picture location shall also be documented in the field log book.

The following records will be maintained:

- Test Pit Log (Figure 1) and/or Test Pit Profile Log (Figure 2)
- Sample collection records
- Field notebook
- Chain-of-custody forms
- Shipping receipts

All documentation will be placed in the project files and retained following completion of the project.

7.0 TRAINING/QUALIFICATIONS

Test pit/trench subsurface explorations require a moderate degree of training and experience as numerous situations may occur which will require field decisions to be made. It is recommended that inexperienced personnel be supervised for several test pit/trench explorations before working on their own. Experienced excavation subcontractors are also of great assistance with problem resolution in the field. Field personnel should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous materials may be present.

8.0 REFERENCES

United States Environmental Protection Agency. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM). USEPA, Region 4, Enforcement and Investigations Branch, Athens, GA. November 2001.

29 CFR Part 1926.650-652.

SOP NUMBER: 7230

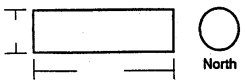
TEST PIT LOG

Sheet 9 of 1.

Project Number:	Date:	Test Pit:
Project Name:	Contractor:	Total Depth:
Location:		Ground Elevation:
Equipment Used:		Logged By:
Remarks:		

Elev. Feet	Depth Feet	Sample Type & No.	Sample Depth Range	Soil & Rock Description and Comments

Test Pit Plan



Groundwater

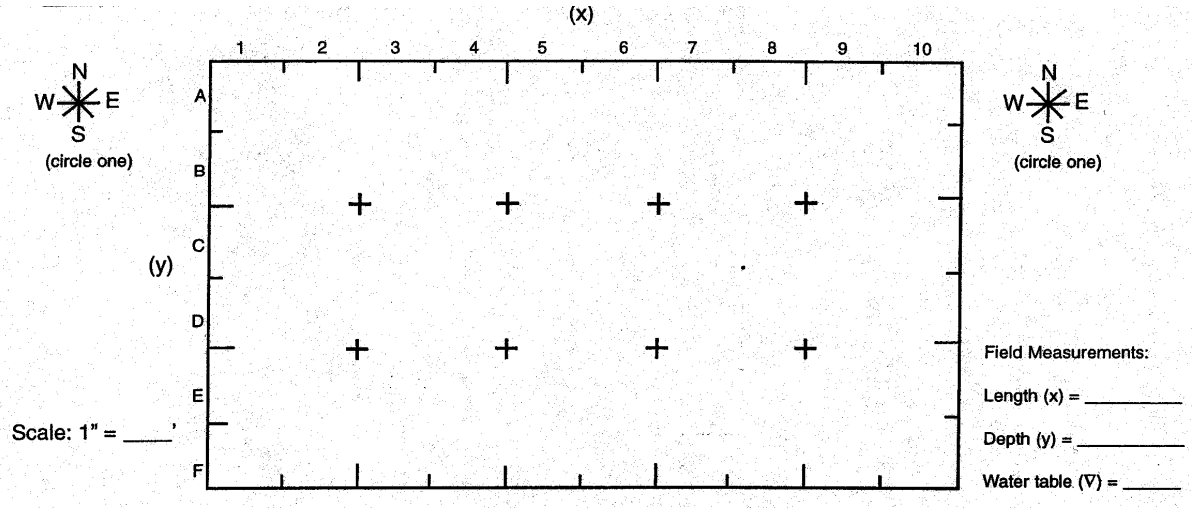
Date	Time (Hrs. after Completion)	Depth (Feet)

SOP NUMBER: 7230

TEST PIT PROFILE LOG

Project Number:	Test Pit No.:
Project Name:	Date:
Project Location:	Inspector:

Field Sketch:



Sample Summary:

Sample	Sample I.D.	Time	Depth(ft)	Hor. Loc.	Matrix	Misc. Information	PID	
B								
C								
D								
E								
F								
G								

Attachment O

ENSR SOP #7510 – Packaging and Shipment of Samples

SOP NUMBER: 7510

Packaging and Shipment of Environmental Samples

Date:	4th Qtr. 1999
Revision Number:	4
Author:	Charles Martin
Discipline:	Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedures associated with the packaging and shipment of environmental samples. Two general categories of samples exist: environmental samples consisting of water and soil submitted for routine environmental testing, and waste material samples which include non-hazardous solid wastes and/or hazardous wastes as defined by 40 CFR Part 261 submitted for environmental testing or bench/pilot-scale treatability testing. Packaging and shipping procedures will differ for the two sample categories.

This SOP is applicable to packaging and shipment of environmental samples submitted for routine environmental testing. Environmental samples are not considered a hazardous waste by definition; therefore, more stringent Department of Transportation (DOT) regulations regarding sample transportation do not apply. Environmental samples do, however, require fairly stringent packaging and shipping measures to ensure sample integrity as well as safety for those individuals handling and transporting the samples.

This SOP is designed to provide a high degree of certainty that environmental samples will arrive at their destination intact. This SOP assumes that samples will often require shipping overnight by a commercial carrier service, therefore, the procedures are more stringent than may be necessary if a laboratory courier is used or if samples are transported directly to their destination by a sampling team member. Should the latter occur, the procedures may be modified to reflect a lesser degree of packaging requirements.

Respective state or federal agency (regional offices) protocols may require or recommend specific types of equipment for use in sample packaging or a specific method of shipment that may vary from the indicated procedures. Deviations from this SOP to accommodate other regulatory requirements should be reviewed in advance of the field program, should be explained in the project work plan, and must be documented in the field project notebook when they occur.

1.2 General Principles

Sample packaging and shipment generally involves the placement of individual sample containers into a cooler or other similar shipping container and placement of packing materials and coolant in such a manner as to isolate the samples, maintain the required temperature, and to limit the potential for damage to sample containers when the cooler is transported.

1.3 Quality Assurance Planning Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific work plan or Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will specify sample packaging and shipment requirements if variations to the indicated procedures are necessary on a particular project.

1.4 Health and Safety Considerations

Sampling personnel should be aware that packaging and shipment of samples involves potential physical hazards primarily associated with handling of occasional broken sample containers and lifting of heavy objects. Adequate health and safety measures must be taken to protect sampling personnel from these potential hazards. The project Health and Safety Plan (HASP) generally addresses physical and other potential hazards. This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed. In the absence of a HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 Sampling Technician

It is the responsibility of the sampling technician to be familiar with the procedures outlined within this SOP and with specific sampling, quality assurance, and health and safety requirements outlined within the project-specific plans. The sampling technician is responsible for proper packaging and shipment of environmental samples and for proper documentation of sampling activities for the duration of the sampling program.

2.2 Sampling Coordinator

Large sampling programs may require additional support personnel such as a sampling coordinator. The sampling coordinator is responsible for providing management support such as maintaining an orderly sampling process, providing instructions to sampling technicians regarding sampling locations, and fulfilling sample documentation requirements, thereby allowing sampling technicians to collect samples in an efficient manner.

2.3 Project Manager

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the activities in accordance with the project plan and this SOP. The project manager is also responsible for ensuring that proper arrangements have been made with the designated analytical laboratory. These arrangements include, but are not necessarily limited to, subcontractor agreements, analytical scheduling, and bottle/cooler orders. The project manager may delegate some of these responsibilities to other project staff.

3.0 REQUIRED MATERIALS

- Sample coolers
- Sample containers
- Shipping labels
- Chain-of-custody records, custody seals
- Bubble wrap
- Vermiculite (granular), or styrofoam pellets
- "Blue Ice" refreezable ice packs, or ice cubes
- Transparent tape, or rubber bands
- Fiber tape
- Duct tape
- Zipper-lock plastic bags

- Trash bags
- Health and Safety supplies
- Equipment decontamination materials
- Field project notebook/pen

4.0 METHOD

4.1 General Information

4.1.1 Regulatory Information

The extent and nature of sample containerization will be governed by the type of sample, and the most reasonable projection of the sample's hazardous nature and constituents. The EPA regulations (40 CFR Section 261.4(d)) specify that samples of solid waste, water, soil or air, collected for the sole purpose of testing, are exempt from regulation under the Resource Conservation and Recovery Act (RCRA) when any of the following conditions are applicable:

- Samples are being transported to a laboratory for analysis;
- Samples are being transported to the collector from the laboratory after analysis;
- Samples are being stored (1) by the collector prior to shipment for analyses, (2) by the analytical laboratory prior to analyses, (3) by the analytical laboratory after testing but prior to return of sample to the collector or pending the conclusion of a court case.

4.1.2 Sample Information:

The following information must accompany each shipment of samples on a chain-of-custody form (Figure 1) where each sample has an individual entry:

- Sample collector's name, mailing address and telephone number,
- Analytical laboratory's name, mailing address and telephone number,
- A unique identification of each sample,

- Sample description (matrix),
- Number and type of sample containers,
- Container size,
- Preservative,
- Type and method of analysis requested, and
- Date and time that the samples were collected and prepared for shipping,
- Special handling instructions, including notation of suspected high concentration samples.

4.1.3 Laboratory Notifications:

Prior to sample collection, the Project Manager, or designated alternative must notify the laboratory manager of the number, type and approximate collection and shipment dates for the samples. If the number, type or date of sample shipment changes due to program changes which may occur in the field, the Project Manager or alternate must notify the laboratory of the changes. Additional notification from the field is often necessary when shipments are scheduled for weekend delivery.

4.2 General Site Preparation

4.2.1 Small Projects

Small projects of one or two days duration may require packaging and shipment of samples using the field vehicle as the sample preparation area. If sample coolers will be sent via third party commercial carrier service, adequate sample packaging materials should be sent to the project location in advance of sampling or purchased from stores located near the site.

4.2.2 Large Projects

Multi-day or week sampling programs usually require rental of an office trailer or use of existing office/storage facilities for storage of equipment as well as for sample preparation. If possible, a designated area should be selected for storage of unused sample containers/coolers and another area for sample handling, packaging, and shipment. Handling of environmental samples should preferably be conducted in a clean area and away from unused

sample containers to minimize the potential for cross contamination. Large quantities of packaging materials may require advance special ordering. Shipping forms/labels may be preprinted to facilitate shipping.

4.2.3 Cooler Inspection and Decontamination

Laboratories will often re-use coolers. Every cooler received at a project location should be inspected for condition and cleanliness. Any coolers that have cracked interior or exterior linings/panels or hinges should be discarded as their insulating properties are now compromised. Any coolers missing one or both handles should also be discarded if replacement handles (i.e., knotted rope handles) can not be fashioned in the field. Replacement coolers may be purchased in the field if necessary.

The interior and exterior of each cooler should be inspected for cleanliness before using it. Excess strapping tape and old shipping labels should be removed. If the cooler interior exhibits visible contamination or odors it should be decontaminated in accordance with ENSR SOP-7600 (Decontamination of Equipment) prior to use. Drain plugs should be sealed on the inside with duct tape.

4.2.4 Other Considerations

VOC Samples - Sample containers used for VOC analysis may be grouped into a single cooler, with separate chain-of-custody record, to limit the number of trip blanks required for transportation and analysis. Individual VOC samples may also be placed into Zipper-lock bags to further protect the samples.

Contaminated Samples - Sample containers with presumed high contaminant concentrations should be isolated within their own cooler with each sample container placed into a Zipper-lock bag.

4.3 Sample Packaging Method

Sample packaging should be conducted in the following manner:

4.3.1 Place plastic bubble wrap matting over the base of each cooler or shipping container as needed. A 2- to 3-inch thickness layer of vermiculite may be used as a substitute base material.

4.3.2 Insert a clean trash bag into the cooler to serve as a liner.

4.3.3 Check that each sample container is sealed, labelled legibly, and is externally clean. Re-label and/or wipe bottles clean if necessary. Clear tape should be placed over the labels to protect them. Wrap each sample bottle individually with bubble wrap secured with tape or rubber bands. Place bottles into the cooler in an upright single layer with approximately one inch of space between each bottle. Do not stack bottles or place them in the cooler lying on their side. If plastic and glass sample containers are used, alternate the placement of each type of container within the cooler so that glass bottles are not placed side by side.

4.3.4 Insert cooler temperature blanks if required.

4.3.5 Place additional vermiculite, bubble wrap, and/or styrofoam pellet packing material throughout the voids between sample containers within each cooler to a level which meets the approximate top of the sample containers. Packing material may require tamping by hand to reduce the potential for settling.

4.3.6 Place cubed ice or cold packs in heavy duty Zip-lock type plastic bags, close the bags, and distribute the packages in a layer over the top of the samples. Cubed ice should be double-bagged to prevent leakage. Loose ice should never be used. Cold packs should be used only if the samples are chilled before being placed in the cooler.

4.3.7 Add additional bubble wrap/styrofoam pellets or other packing materials to fill the balance of the cooler or container.

4.3.8 Obtain two pieces of chain of custody tape as shown in Figure 2 and enter the custody tape numbers in the appropriate place on the chain-of-custody form. Sign and date the chain-of-custody tape.

4.3.9 Complete the chain-of-custody form. If shipping the samples involves use of a third party commercial carrier service, sign the chain-of-custody record thereby relinquishing custody of the samples. Shippers should not be asked to sign chain of custody records. If a laboratory courier is used, or if samples are transported to the laboratory, the receiving party should accept custody and sign the chain-of-custody records. Remove the last copy from the form and retain it with other field notes. Place the original (with remaining copies) in a Zipper-lock type plastic bag and tape the bag to the inside lid of the cooler or shipping container.

4.3.10 Close the top or lid of the cooler or shipping container.

4.3.11 Place the chain of custody tape at two different locations (i.e., one tape on each side) on the cooler or container lid and overlap with transparent packaging tape.

4.3.12 Packaging tape should be placed entirely around the sample shipment containers. A minimum of two full wraps of packaging tape will be placed at least two places on the cooler.

4.3.13 Repeat the above steps for each cooler or shipping container.

4.4 Sample Shipping Method

Packaged sample coolers should be shipped using one of the following options:

4.4.1 Hand Delivery

When a project member is transporting samples by automobile to the laboratory, the cooler should only be sealed with tape. In these cases, chain-of-custody will be maintained by the person transporting the sample and chain-of-custody tape need not be used. Chain-of-custody records should be relinquished upon delivery and a copy of the record retained in the project file.

4.4.2 Laboratory Courier

Laboratory couriers are usually employees of the analytical laboratory receiving the samples. As such, they will accept custody of the samples and must be asked to sign the chain-of-custody records. Chain-of-custody records do not need to be sealed in the cooler although it is recommended that the coolers be sealed with tape. All other packaging requirements generally apply unless otherwise specified in the QAPP.

If the laboratory courier is not authorized to accept custody of the samples, or if the requirements of the project plan preclude transfer to the laboratory courier, samples will be handled as described below in Section 4.4.3.

4.4.3 Third Party Courier

If overnight shipment is required, a third party package delivery service should be used. Transport the cooler to the package delivery service office

or arrange for package pick-up at the site. Fill out the appropriate shipping form or airbill and affix it to the cooler. Some courier services may use multi-package shipping forms where only one form needs to be filled out for all packages going to the same destination. If not, a separate shipping form should be used for each cooler. Keep the receipt for package tracking purposes should a package become lost. Please note that each cooler also requires a shipping label which indicates point of origin and destination. This will aid in recovery of a lost cooler if a shipping form gets misplaced. Never leave coolers unattended while waiting for package pick-up. Airbills or waybills will be maintained as part of the custody documentation.

4.5 Sample Receipt

Upon receipt of the samples, the analytical laboratory will open the cooler or shipping container and will sign "received by laboratory" on each chain-of-custody form. The laboratory will verify that the chain-of-custody tape has not been broken previously and that the tape number corresponds with the number on the chain-of-custody record. The laboratory will note the condition of the samples upon receipt and will identify any discrepancies between the contents of the cooler and chain-of-custody. The analytical laboratory will then forward the back copy of the chain-of-custody record to the project manager to indicate that sample transmittal is complete.

5.0 QUALITY CONTROL

The potential for samples to break during transport increases greatly if individual containers are not snugly packed into the cooler. Completed coolers may be lightly shake-tested to check for any loose bottles. The cooler should be repacked if loose bottles are detected.

Environmental samples are generally shipped so that the samples are maintained at a temperature of approximately 4°C. Temperature blanks may be required for some projects as a quality assurance check on shipping temperature conditions. These blanks usually are supplied by the laboratory and consist of a 40-ml vial or plastic bottle filled with tap water. Temperature blanks should be placed near the center of the cooler.

6.0 DOCUMENTATION

Documentation supporting sample packaging and shipment generally consists of chain-of-custody records and shipping records. In addition, a description of sample packaging procedures will be written in the field project notebook. All documentation will be retained in the project files following project completion.

7.0 TRAINING/QUALIFICATIONS

Sample packaging and shipment is a relatively simple procedure requiring minimal training and a minimal amount of equipment. It is, however, recommended that initial attempts be supervised by more experienced personnel. Sampling technicians should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous waste materials are considered to be present.

Attachment P

ENSR SOP #7600 – Decontamination of Equipment

SOP NUMBER: 7600

Decontamination of Field Equipment

Date:	4th Qtr. 1994
Revision Number:	4
Author:	Charles Martin
Discipline:	Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This SOP describes the methods to be used for the decontamination of field equipment used in the collection of environmental samples. The list of field equipment may include a variety of items used in the collection of soil and/or water samples, such as split-spoon samplers, trowels, scoops, spoons, bailers and pumps. Heavy equipment such as drill rigs and backhoes also require decontamination, usually in a specially constructed temporary decontamination area.

Decontamination is performed as a quality assurance measure and a safety precaution. Improperly decontaminated sampling equipment can lead to misinterpretation of environmental data due to interference caused by cross-contamination. Decontamination protects field personnel from potential exposure to hazardous materials. Decontamination also protects the community by preventing transportation of contaminants from a site.

This SOP emphasizes decontamination procedures to be used for decontamination of reusable field equipment. Occasionally, dedicated field equipment such as well construction materials (well screen and riser pipe) or disposable field equipment (bailers or other general sampling implements) may also require decontamination prior to use. The project-specific work plan should indicate the specific decontamination requirements for a particular project.

Respective state or federal agency (regional offices) regulations may require specific types of equipment or procedures for use in decontamination of field equipment. The project manager should review the applicable regulatory requirements, if any, prior to the start of the field investigation program.

1.2 General Principles

Decontamination is accomplished by manually scrubbing, washing, or spraying equipment with detergent solutions, tap water, distilled/deionized water, steam and/or high pressure water, or solvents. The decontamination method and agents

are generally determined on a project-specific basis and must be stated in the Quality Assurance Project Plan (QAPP).

Generally, decontamination of equipment is accomplished at each sampling site between collection points. Waste decontamination materials such as spent liquids and solids will be collected and managed as investigation-derived waste for later disposal. All decontamination materials, including wastes, should be stored in a central location so as to maintain control over the quantity of materials used or produced throughout the investigation program.

1.3 Quality Assurance Planning Considerations

1.3.1 General Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific QAPP. The QAPP guidelines typically require collection of equipment blank samples in order to determine the effectiveness of the decontamination procedure.

The decontamination method, solvent, frequency, location on site and the method of containment and disposal of decontamination wash solids and solutions are dependent on site logistics, site-specific chemistry, and nature of the contaminated media to be studied and the objectives of the study. Each topic must be considered and addressed during development of a decontamination strategy and should be outlined in the Quality Assurance Project Plan (QAPP).

1.3.2 Solvent Selection

There are several factors which need to be considered when deciding upon a decontamination solvent. The solvent should not be an analyte of interest. The sampling equipment must be resistant to the solvent. The solvent must be evaporative or water soluble or preferably both. The applicable regulatory agency may have specific requirements regarding decontamination solvents. The QAPP should specify the type of solvent to be used for a particular project.

The analytical objectives of the study must also be considered when deciding upon a decontamination solvent. Pesticide-grade methanol is the solvent of choice for general organic analyses. It is relatively safe and effective. Hexane, acetone, and isopropanol are sometimes used as well. A 10% nitric acid in deionized water solution is the solvent of choice for general metals

analyses. Nitric acid can be used only on Teflon, plastics and glass. If used on metal equipment, nitric acid will eventually corrode the metal and lead to the introduction of metals to the collected samples. Dilute hydrochloric acid is usually preferred over nitric acid when cleaning metal sampling equipment.

Equipment decontamination should be performed a safe distance away from the sampling area so as not to interfere with sampling activities but close enough to the sampling area to maintain an efficient working environment. If heavy equipment such as drill rigs or backhoes are to be decontaminated, then a central decontamination station should be constructed with access to a power source and water supply.

1.4 Health and Safety Considerations

Decontamination procedures may involve chemical exposure hazards associated with the type of contaminants encountered or solvents employed and may involve physical hazards associated with decontamination equipment. When decontamination is performed on equipment which has been in contact with hazardous materials or when the quality assurance objectives of the project require decontamination with chemical solvents, the measures necessary to protect personnel must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all personnel performing equipment decontamination, and must be adhered to as field activities are performed.

2.0 RESPONSIBILITIES

2.1 Sampling Technician

It is the responsibility of the sampling technician to be familiar with the decontamination procedures outlined within this SOP and with specific quality assurance, and health and safety requirements outlined within project-specific work plans (HASP, QAPP). The sampling technician is responsible for decontamination of field equipment and for proper documentation of decontamination activities. The sampling technician is also responsible for ensuring that decontamination procedures are followed by subcontractors when heavy equipment requires decontamination.

2.2 Field Project Manager

The field project manager is responsible for ensuring that the required decontamination procedures are followed at all times. The project manager is also responsible for ensuring that subcontractors construct and operate their decontamination facilities according to project specifications. The project manager is responsible for collection and control of IDW in accordance with project specifications.

3.0 REQUIRED MATERIALS

- Decontamination agents (per work plan requirements):
  - LIQUI-NOX, ALCONOX, or other phosphate-free biodegradable detergent,
  - Tap water,
  - Distilled/deionized water,
  - Nitric acid and/or hydrochloric acid,
  - Methanol and/or hexane, acetone, isopropanol.
- Health and Safety equipment
- Chemical-free paper towels
- Waste storage containers: drums, 5-gallon pails w/covers, plastic bags
- Cleaning containers: plastic buckets or tubs, galvanized steel pans, pump cleaning cylinder
- Cleaning brushes
- Pressure sprayers
- Squeeze bottles
- Plastic sheeting
- Aluminum foil
- Field project notebook/pen

4.0 METHODS

4.1 General Preparation

4.1.1 It should be assumed that all sampling equipment, even new items, are contaminated until the proper decontamination procedures have been performed on them or unless a certificate of analysis is available which demonstrates the items cleanliness.

Field equipment that is not frequently used should be wrapped in aluminum foil, shiny side out, and stored in a designated "clean" area. Small field equipment can also be stored in plastic bags to eliminate the potential for contamination. Field equipment should be inspected and decontaminated prior to use if the equipment appears contaminated and/or has been stored for long periods of time. Unless customized procedures are stated in the QAPP for decontamination of equipment, the standard procedures specified in this SOP shall be followed.

4.1.2 Establish the decontamination station within an area that is convenient to the sampling location. If single samples will be collected from multiple locations, then a centralized decontamination station, or a portable decontamination station should be established.

4.1.3 An investigation-derived waste (IDW) containment station should be established at this time also. The project-specific work plan should specify the requirements for IDW containment. In general, decontamination solutions are discarded as IDW between sampling locations. Solid waste is disposed of as it is generated.

4.2 Decontamination for Organic Analyses

4.2.1 This procedure applies to soil sampling and groundwater sampling equipment used in the collection of environmental samples submitted for organic constituents analysis. Examples of relevant items of equipment include split-spoons, trowels, scoops/spoons, bailers, and other small items. Submersible pump decontamination procedures are outlined in Section 4.4.

4.2.2 Decontamination is to be performed before sampling events and between sampling points.

4.2.3 After a sample has been collected, remove all gross contamination from the equipment or material by brushing and then rinsing with available tap water.

This initial step may be completed using a 5-gallon pail filled with tap water. Steam or a high-pressure water rinse may also be conducted to remove solids and/or other contamination.

4.2.4 Wash the equipment with a phosphate-free detergent and tap water solution. This solution should be kept in a 5-gallon pail with its own brush.

4.2.5 Rinse with tap water or distilled/deionized water until all detergent and other residue is washed away. This step can be performed over an empty bucket using a squeeze bottle or pressure sprayer.

4.2.6 Rinse with methanol or other appropriate solvent using a squeeze bottle or pressure sprayer. Rinsate should be collected in a waste bucket.

4.2.7 Rerinse with deionized water to remove any residual solvent. Rinsate should be collected in the solvent waste bucket.

4.2.8 Allow the equipment to air-dry in a clean area or blot with chemical-free paper towels before reuse. Wrap the equipment in tin foil and/or seal it in a plastic bag if it will not be reused for a while.

4.2.9 Dispose of soiled materials and spent solutions in the designated IDW disposal containers.

4.3 Decontamination for Inorganic (Metals) Analyses

4.3.1 This procedure applies to soil sampling equipment used primarily in the collection of environmental samples submitted for inorganic constituents analysis. Examples of relevant items of equipment include split-spoons, trowels, scoops/spoons, bailers, and other small items.

4.3.2 For plastic and glass sampling equipment, follow the steps outlined in 4.2 above, however, use a 10% nitric acid solution (acid in water) in place of the solvent rinse in Section 4.2.6.

4.3.3 For metal sampling equipment, follow the steps outlined in 4.2 above, however, use a 10% hydrochloric acid solution (acid in water) in place of the solvent rinse in Section 4.2.6.

4.4 Decontamination of Submersible Pumps

4.4.1 This procedure will be used to decontaminate submersible pumps before and between ground-water sample collection points. This procedure applies to both electric submersible and bladder pumps. This procedure also applies to discharge tubing if it will be reused between sampling points.

4.4.2 Prepare the decontamination area if pump decontamination will be conducted next to the sampling point. If decontamination will occur at another location, the pump and tubing may be removed from the well and placed into a clean trash bag for transport to the decontamination area. Pump decontamination is easier with the use of 3-foot tall pump cleaning cylinders (i.e., Nalgene cylinder) for the various cleaning solutions, although the standard bucket rinse equipment may be used.

4.4.3 Once the decontamination station is established, the pump should be removed from the well and the discharge tubing and power cord coiled by hand as the equipment is removed. If any of the equipment needs to be put down temporarily, place it on a plastic sheet (around well) or in a clean trash bag. If a disposable discharge line is used it should be removed and discarded at this time.

4.4.4 As a first step in the decontamination procedure, use a pressure sprayer with tap water to rinse the exterior of the pump, discharge line, and power cord as necessary. Collect the rinsate and handle as IDW.

4.4.5 Place the pump into a pump cleaning cylinder or bucket containing a detergent solution (detergent in tap water). Holding the tubing/power cord, pump solution through the pump system. A minimum of one gallon of detergent solution should be pumped through the system. Collect the rinsate and handle as IDW.

4.4.6 Place the pump into another cylinder/bucket containing a 10% solution of solvent (methanol, or other designated solvent) in distilled/deionized water. Pump until the detergent solution is removed. Collect the rinsate and handle as IDW.

4.4.7 Place the pump into another cylinder/bucket containing distilled/deionized water. Pump a minimum of 3 to 5 pump system volumes (pump and tubing) of water through the system. Collect the rinsate and handle as IDW.

4.4.8 Remove the pump from the cylinder/bucket and if the pump is reversible, place the pump in the reverse mode to discharge all removable water from the system. If the pump is not reversible the pump and discharge line should be drained by hand as much as possible. Collect the rinsate and handle as IDW.

4.4.9 Using a pressure sprayer with distilled/deionized water, rinse the exterior of the pump, discharge line, and power cord thoroughly, shake all excess water, then place the pump system into a clean trash bag for storage. If the pump system will not be used again right away, the pump itself should also be wrapped with aluminum foil before placing it into the bag.

4.5 Decontamination of Large Equipment

4.5.1 Consult the QAPP for instruction on the location of the decontamination station and the method of containment of the wash solutions. On large projects usually a temporary decontamination facility (decontamination pad) is required which may include a membrane-lined and bermed area large enough to drive heavy equipment (drill rig, backhoe) onto with enough space to spread other equipment and to contain overspray. Usually a small sump with pump is necessary to collect and contain rinsate. A water supply and power source is also necessary to run steam cleaning and/or pressure washing equipment.

4.5.2 Upon arrival and prior to leaving a sampling site, all heavy equipment such as drill rigs, trucks, and backhoes should be thoroughly cleaned and then the parts of the equipment which come in contact or in close proximity to sampling activity should be decontaminated. This can be accomplished in two ways, steam cleaning or high pressure water wash and manual scrubbing. Following this initial cleaning, only those parts of the equipment which come in close proximity to the sampling activities (i.e., auger stems, rods, backhoe bucket) must be decontaminated in between sampling events.

Occasionally, well construction materials such as well screen and riser pipe may require decontamination before the well materials are used. These materials may be washed in the decontamination pad, preferably on a raised surface above the pad (i.e., on sawhorses), with clean plastic draped over the work surfaces. Well materials usually do not require a multistep cleaning process as they generally arrive clean from the manufacturer. Usually, a thorough steam-cleaning of the interior/exterior of the well materials will be sufficient. The QAPP should provide specific guidance regarding decontamination of well materials.

5.0 QUALITY CONTROL

5.1 Field Blank Sample Collection

General guidelines for quality control check of field equipment decontamination usually require the collection of one field blank from the decontaminated equipment per day. The QAPP should specify the type and frequency of collection of each type of quality assurance sample.

Field blanks are generally made by pouring laboratory-supplied deionized water into, over, or through the freshly decontaminated sampling equipment and then transferring this water into a sample container. Field blanks should then be labeled as a sample and submitted to the laboratory to be analyzed for the same parameters as the associated sample. Field blank sample numbers, as well as collection method, time and location should be recorded in the field notebook.

6.0 DOCUMENTATION

Specific information regarding decontamination procedures should be documented in the project-specific field notebook. Documentation within the notebook should thoroughly describe the construction of each decontamination facility and the decontamination steps implemented in order to show compliance with the project work plan. Decontamination events should be logged when they occur with the following information documented:

- Date, time and location of each decontamination event
- Equipment decontaminated
- Method
- Solvents
- Noteable circumstances
- Identification of field blanks and decontamination rinsates
- Method of blank and rinsate collection
- Date, time and location of blank and rinsate collection
- Disposition of IDW

Repetitive decontamination of small items of equipment does not need to be logged each time the item is cleaned.

7.0 TRAINING/QUALIFICATIONS

All sampling technicians performing decontamination must be properly trained in the decontamination procedures employed, the project data quality objectives, health and safety

procedures and the project QA procedures. Specific training or orientation will be provided for each project to ensure that personnel understand the special circumstances and requirements of that project. Field personnel should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous materials may be present.

8.0 REFERENCES

Not applicable.

Attachment Q

Example ENSR Field Data Sheets

ENSR | AECOM

Soil Boring Log

Client:	*Project:*		**BORING ID:**
Project Number:			
Site Location:			
Coordinates:	*Elevation:*		*Sheet: of*
Drilling Method:			*Monitoring Well Installed:* **Y N**
Sample Type(s):	*Boring Diameter: _____in.*		*Screened Interval:*
Weather:	*Logged By:*	*Date/Time Started:*	*Depth of Boring:*
Drilling Contractor:	*Ground Elevation:*	*Date/Time Finished:*	*Water Level:*

Depth (feet)	PID (PPM)	Sample Depth (ft)	Blow Count (per 6-inches)	Recovery (in. or ft.)	(Headspace (ppmv)	U.S.C.S	**MATERIALS: Color, size, range, MAIN COMPONENT, minor component(s), moisture content, structure, angularity, maximum grain size, odor, and Geologic Unit (If Known)**	Lab Sample ID	Lab Sample Depth
0									
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									

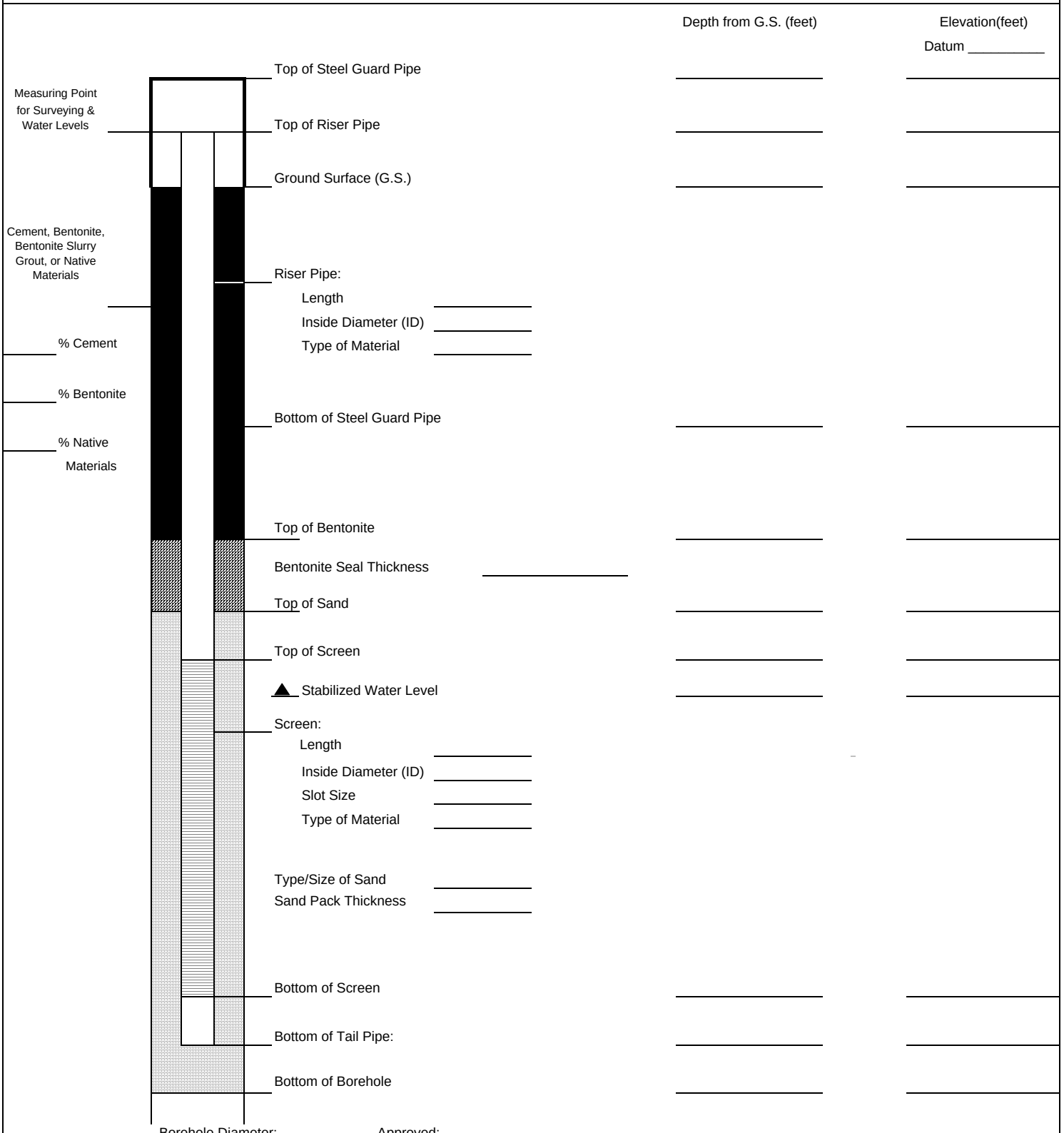
NOTES:

Checked by _____ Date:_____

Date	Time	Depth to groundwater while drilling

ENSR | AECOM	*Client:*	***WELL ID:***
	Project Number:	
	Site Location:	*Date Installed:* #
	Well Location: *Coords:*	*Inspector:*
	Method:	*Contractor:*

MONITORING WELL CONSTRUCTION DETAIL



ENSR | AECOM

Test Pit Log

Client:	*Project:*	**TEST PIT ID:**
Project Number:		
Site Location:		
Coordinates:	*Elevation:*	*Sheet: of*
Equipment Used:		
Total Depth:		
Weather:	*Logged By:*	*Date/Time Started:*
Drilling Contractor:	*Ground Elevation:*	*Date/Time Finished:*

Depth (feet)	Geologic sample ID	Sample Depth (ft)	(Headspace (ppmv)	Soil and Rock Description and Comments	Lab Sample ID	Lab Sample Depth
0						
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

NOTES:

Date	Time	Depth to groundwater

Checked by _____ Date:_____

ENSR | AECOM

Well/Piezometer Development Record

Well/Piez. ID:

Client: _____ Site Location: _____

Project #: _____ Date: _____ Developer: _____

WELL/PIEZOMETER DATA

Well ☐ Piezometer ☐ Diameter _____ Material _____

Measuring Point Description _____

Depth to Top of Screen (ft.) _____

Depth to Bottom of Screen (ft.) _____

Total Well Depth (ft.) _____

Depth to Static Water Level (ft.) _____

Geology at Screen Interval (if known) _____

Time of Water Level Measurement _____

Calculate Purge Volume (gal.) _____

Disposal Method _____

Wellhead PID/FID _____

Original Well Development ☐ Redevelopment ☐ Date of Original Development _____

DEVELOPMENT METHOD _____ **PURGE METHOD** _____

Field Testing Equipment Used: Make Model Serial Number

Field Testing Calibration Documentation Found in Field Notebook # _____ Page # _____

Time	Volume Removed (gal)	T° (C/F)	pH	Spec. Cond (umhos)	Turbidity (NTUs)	DO	Color	Odor	Other

ACCEPTANCE CRITERIA (from workplan)

Min. Purge Volume (____ well volumes) _____gallons

Maximum Turbidity Allowed _____ NTUs

Stabilization of parameters _____%

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

If no or N/A explain below:

_____

_____

_____

Signature _____ Date: _____

ENSR | AECOM

Well ID:

Low Flow Ground Water Sample Collection Record

Client: _____ Date: _____ Time: Start _____ am/pm
Project No: _____ Finish _____ am/pm
Site Location: _____
Weather Conds: _____ Collector(s): _____

1. WATER LEVEL DATA: (measured from Top of Casing)

a. Total Well Length _____ c. Length of Water Column 0 (a-b)

Casing Diameter/Material _____

b. Water Table Depth _____ d. Calculated System Volume (see back) _____

2. WELL PURGE DATA

a. Purge Method: _____

b. Acceptance Criteria defined (see workplan)

- Temperature	3%	-D.O.	10%
- pH	± 1.0 unit	- ORP	± 10mV
- Sp. Cond.	3%	- Drawdown	< 0.3'

c. Field Testing Equipment used: Make Model Serial Number

_____
_____

Time (24hr)	Volume Removed (Liters)	Temp. (°C/F)	pH (SU)	Spec. Cond. (mS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (feet)	Color/Odor

d. Acceptance criteria pass/fail

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

(continued on back)

If no or N/A - Explain below.

_____

3. SAMPLE COLLECTION: Method: _____

Sample ID	Container Type	No. of Containers	Preservation	Analysis Req.	Time

Comments _____

Signature _____ Date _____

Attachment R

Example Sample Label

STL Buffalo (716) 691-2600

EVENT DESCRIPTION
VIOC Monthly

SAMPLE LOCATION
PORT 1

ANALYSIS
HEM

PRESERVATIVE
H2SO4 TO pH < 2, COOL <=6°C

BOTTLE	FILTER	SHIP TO
32ozGN	No	RECNY

AC73175001

DATE
TIME
BY

STL Buffalo (716) 691-2600

EVENT DESCRIPTION
VIOC Monthly

SAMPLE LOCATION
PORT 1

ANALYSIS
PH

PRESERVATIVE
COOL <=6°C

BOTTLE	FILTER	SHIP TO
4ozP	No	RECNY

AC73175002

DATE
TIME
BY

Attachment S

Example Laboratory Chain of Custody Record and Custody Seal

Chain of Custody Record

SEVERN TRENT STL

Severn Trent Laboratories, Inc.

STL-4124 (0901)

Client	Project Manager	Date	Chain of Custody Number 298898
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact	Lab Contact	Analysis (Attach list if more space is needed)
Project Name and Location (State)	Carrier/Waybill Number		
Contract/Purchase Order/Quote No.			

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Matrix: Air	Aqueous	Sed.	Soil		Containers & Preservatives: Unpres	H2SO4	HNO3	HCl	NaOH	ZnAc/ NaOH		Special Instructions/ Conditions of Receipt

Possible Hazard Identification

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal

☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months

(A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required

☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

	Date	Time		Date	Time
1. Relinquished By			1. Received By		
2. Relinquished By			2. Received By		
3. Relinquished By			3. Received By		

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

SEVERN TRENT LABORATORIES, INC.

SAMPLE L № 184783

Attachment T

Laboratory Quality Manual



Laboratory Quality Manual
for
STL Knoxville

5815 Middlebrook Pike
Knoxville, Tennessee 37921
(865) 291-3000

Approved by:

J. Thomas Yoder — 5/25/06
Laboratory Director — Date

Robyn M. Wagner — 5/25/06
Robyn M. Wagner — Date
Technical Director

Snell Austin Mills III — 5/26/2006
Snell A. Mills — Date
Technical Director

Christopher W. Rigell — 05/25/06
Christopher W. Rigell — Date
Quality Assurance Manager

Revision 4
May 25, 2006
Controlled Copy Number: UNCONTROLLED COPY

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Purpose and Scope

The purpose of this Laboratory Quality Manual (LQM) is to describe the implementation of the Severn Trent Laboratories (STL) Quality System at the STL Knoxville laboratory. The LQM is written within the guidelines of the STL Quality Management Plan (QMP), which applies to all STL laboratories. The organization of this LQM is based on the "EPA Requirements for Quality Management Plans" (EPA QA/R-2, March 2001). This LQM outlines specific policies, organization, responsibilities, and activities required to assure high quality laboratory services. The LQM also fulfills the requirements of our clients, government agencies, and NELAC to document the laboratory Quality System.

This LQM contains references to other essential STL quality documents. The company-wide QMP, STL Knoxville LQM, and referenced policies and SOPs are interrelated. Together they provide an integrated quality foundation that meets the objectives of the STL Quality Assurance Policy, as stated in Section 1.2.

The requirements set forth in this document are applicable to all employees at the STL Knoxville laboratory. The policies and practices described here are presented as minimum guidelines only. Based on good scientific judgment, more rigorous requirements may be applied by laboratory employees. Specific requirements delineated in project plans may supersede general quality requirements described in this manual.

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1.0 Management Commitment and Organization

1.1 STL Mission Statement

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

1.2 STL Quality Assurance Policy

It is STL's policy to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

The STL Quality Assurance Policy also defines the objectives of the quality system (see section 2.1).

1.3 STL Management Statement of Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a quality system that is clear, effective, well communicated, and supported at all levels in the company. The laboratory management is also committed to compliance with the NELAC standard.

1.4 Ethics, Waste, Fraud and Abuse

Establishing and maintaining a high ethical standard is an important element of a quality system. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times, STL has established an Ethics Policy, including an Employee Ethics Statement (see Figure 1.4-1). Ethics is also a major component of the STL QA training program (see Section 4 for more discussion). A

central tenant is that management must consistently convey the message to analysts that financial pressures can never be allowed to compromise the quality of work.

See the following documents related to ethics in the laboratory:
P-L-006, Ethics Policy
LQM Section 6 – Computer Hardware and Software
KNOX-QA-0013 – Instrument Records and Logbooks
S-Q-004 – Acceptable Manual Integration Practices
P-T-001 – Selection of Calibration Points

1.5 Organizational Structure and Relationships

STL Knoxville is a local operating unit of Severn Trent Laboratories, Inc., a Delaware Corporation. The date of incorporation was August 27, 1997. Severn Trent Laboratories is wholly owned by Severn Trent (Del.), Inc., a Delaware Corporation.

The organizational structure, responsibilities and authorities of the corporate staff of Severn Trent Laboratories, Inc. are presented in the STL QMP.

STL Knoxville has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.). The STL Knoxville laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for STL Knoxville is presented in Figure 1.5-1. A list of key STL Knoxville personnel qualifications is provided in Figure 1.5-2. The lab also maintains job descriptions that contain general job responsibilities for all laboratory employees. The following section outlines responsibilities and authorities for all employees of the STL Knoxville laboratory, as they relate to quality management.

The STL Knoxville QA Manager (QAM) is <u>independent</u> from day-to-day laboratory operations, has no direct analytical testing responsibilities, and is free from financial and other undue pressures which might adversely affect the quality of work. The QAM, a key member of the laboratory's management team, has direct access to the Corporate Quality Assurance Director on all matters involving quality. The QAM is available to any lab employee to resolve quality or ethical issues. The QAM, if required, has the authority to cease operations adversely affecting the validity or integrity of the analytical data.

Figure 1.4-1 STL Employee Ethics Statement

SEVERN TRENT STL

Severn Trent Laboratories, Inc.
EMPLOYEE ETHICS STATEMENT

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees;
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and will report this action immediately to a member of senior management, up to and including the President of STL; and
- I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the Severn Trent family of companies.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____ Date _____

Supervisor/Trainer: _____ Date _____

Reference: STL Ethics Policy, P-L-006, Rev. 4.2.

Figure 1.5-1 STL Knoxville Organizational Structure

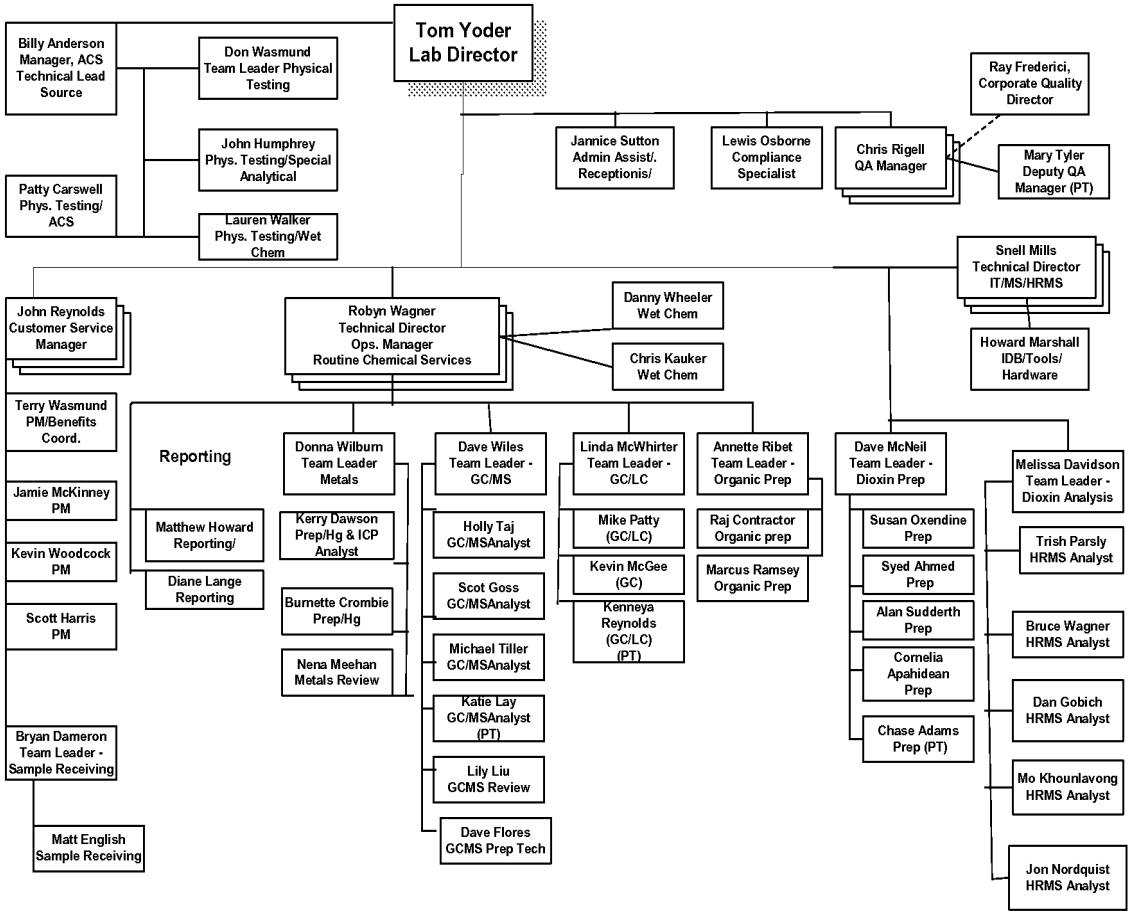


Figure 1.5-2 STL Knoxville Key Personnel

Position	Name	Degree(s), Institution(s), Year	Years Environmental Lab Experience	Years with Company
Laboratory Director	J. Thomas Yoder	B.S., Business Administration, Shippensburg State College, Shippensburg, Pennsylvania; 1982	18	19
Operations Manager, Technical Director	Robyn M. Wagner	B.S., Biology, Rutgers University, New Brunswick, New Jersey; 1981	26	24
IT Supervisor, Technical Director	Snell A. Mills, III	B.A., Chemistry, Maryville College, Maryville, Tennessee, 1979.	27	26
Compliance Specialist, E H &S Coordinator	B. Lewis Osborne	BS, Industrial Statistics-University of Tennessee, Knoxville, Tennessee, 1987	17	17
Quality Assurance Manager	Christopher W. Rigell	Ph.D., Biochemistry, University of Tennessee, Knoxville, Tennessee; 1986; M.S., Biochemistry, University of Tennessee, Knoxville, Tennessee; 1985, B.A., Chemistry and Mathematics, Maryville College, Maryville, Tennessee; 1979	20	18
Deputy Quality Assurance Manager	Mary E. Tyler	M.S., Environmental Engineering, University of Notre Dame, Notre Dame, Indiana; 1984; B.A., Biology, Indiana University, Bloomington, Indiana; 1980	26	21
Customer Service Manager	John D. Reynolds	B.S., Biology, Troy State University, Troy, Alabama; 1988	16	14
Specialty Organics Analysis Team Leader	Melissa Davidson	B.S., Biochemistry, North Carolina State University, Raleigh, North Carolina; 1987.	20	17
Inorganics Team Leader	Donna Wilburn	Certified Laboratory Technician, Knoxville Area Adult Vocational School; 1972	21	21
GC/HPLC Team Leader	Linda McWhirter	B.S., General Chemistry, University of North Alabama, Florence, Alabama; 1991	14	14
GCMS Team Leader	David Wiles	Appalachian State University, B.S. in Mathematics, Minor Physics; 1990; Wilkes Community College, Associate in Science, 1987	14	14
Organic Prep Team Leader	Annette Ribet	Associate Degree in Science, Western Piedmont Community College; 1985	16	14
Specialty Organics Extractions Team Leader	David McNeil	B.S., Natural Science, Bryan College, Dayton, Tennessee, 1985	19	17

1.6 Quality Organization

All personnel are responsible for quality, which includes complying with all QA/QC requirements that pertain to their organizational/technical function. This section outlines the primary QA responsibilities for each position.

1.6.1 Quality Assurance Manager

- Reports directly to the Laboratory Director and, for all QA matters, to the Corporate QA Director to maintain independence of QA oversight
- Responsible for implementing and communicating the QMP
- Maintains, approves, and implements the LQM
- Has joint signature authority, with the Laboratory Director and Technical Director for approval of quality documents
- Directs controlled distribution of laboratory quality documents
- Provides Quality System training to all new personnel
- Reviews and approves documentation of analyst training records
- Serves as a focal point for QA and QC issues, reviews corrective actions and recommends resolution for recurring nonconformances within the laboratory
- Assists in maintaining regulatory analytical compliance, including maintaining certifications
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs systems, data, and surveillance audits
- Hosts external audits conducted by outside agencies
- Responsible for approving quality control reference data changes in the LIMS
- Oversees the selection, review, and approval of analytical subcontractors
- Prepares monthly QA Reports to management describing significant quality events
- Has the final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data
- Responsible for continuous improvement

1.6.2 Laboratory Director

- Reports directly to the Regional General Manager
- Responsible for implementation and adherence by lab staff to the STL QMP, STL Knoxville LQM and all policies and procedures within the laboratory
- Has signature authority for LQM, policies, SOPs, and contracts (as detailed in STL policy)
- Annually assesses the effectiveness of the QMP and LQM within the operation
- Maintains adequate trained staffing documented on organization charts

- Responsible for implementing internal/external audit finding corrective actions

1.6.3 Operations Manager

- Reports directly to the Laboratory Director
- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs
- Supervises the daily activities of the Report Production Group
- Works with the Project Managers and Group/Team Leaders to assure the requirements of projects are met in a timely manner

1.6.4 Laboratory Technical Director

- Reports to Laboratory Director
- Responsible for the technical operation of the laboratory
- Responsible for coordinating the development and implementation of SOPs
- Has joint signature authority for LQM, SOPs, and training records
- Performs technical training in area(s) of expertise
- Interfaces with management on technical needs and solving day-to-day technical issues
- Determines qualifications required for technical positions and evaluates job candidates against those requirements
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work
- Certifies technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible
- Performs other tasks as required by NELAC

The Technical Director meets the requirements specified in the Section 4.1.1.1 of the NELAC standards.

1.6.5 Customer Service Manager (CSM)

- Reports directly to the Laboratory Director
- Supervises the daily activities of the Project Management and Sample Control groups
- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner
- Has signature authority for laboratory reports

- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs
- Brings together resources necessary to ensure customer satisfaction

1.6.6 Project Manager

- Reports directly to the Customer Service Manager
- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff
- Prepares Quality Assurance Summary (QAS) or equivalent summary form and communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Reviews project data packages for completeness and compliance to client needs
- Has signature authority for final reports
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Prepares reissue requests for project data

1.6.7 Group (Area) Leader, Team Leader or Supervisor

- Reports directly to the Operations Manager
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for generation of SOPs for their section

1.6.8 Analyst

- Performs analytical methods and data recording in accordance with documented procedures
- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures

- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures

1.6.9 Sample Custodian

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports nonconformances associated with condition-upon-receipt of samples
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal

1.6.10 Report Production Staff

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Produces deliverables that meet the NELAC requirements, as required.

2.0 Quality System and Description

2.1 Objectives of the STL Quality System

The Quality System is a set of management principles, objectives, policies, responsibilities, and implementation plans at the organizational and project-specific levels. The goal of the STL Quality System is to ensure that business operations are conducted with the highest level of professionalism in the industry. To achieve this goal, it is necessary to provide STL clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that STL provides the highest quality service available in the industry. A well-structured and well-communicated Quality System is essential in meeting this goal. STL's Quality System is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

2.2 Structure of the STL Quality System

At the highest level, the STL Quality Management Plan (QMP) is the basis for STL's Quality System. The QMP provides the guidance under which all STL facilities conduct their operations. This Laboratory Quality Manual (LQM) describes the implementation of the Quality System at the STL Knoxville laboratory. This LQM and the series of associated quality documents described in Section 2.4 define the organization, project-specific principles, goals, controls, and tools of the Quality System as it is applied at this laboratory. The Quality System as described in this LQM demonstrates the commitment to accepted laboratory practices by STL Knoxville.

2.3 Quality Assurance and Quality Controls

Quality Assurance (QA) is defined as the system of activities which ensures the quality of a process, product, or service. Quality controls (QC) are the tools used to monitor and regulate the desired type and quality of product. The QA activities and QC employed in STL Knoxville are defined in the following quality documents.

2.4 Quality Documents

The STL Quality System is developed from the reference documents shown in Table 2-1. The review and control of the STL Knoxville documents described in the following subsections are described in Section 3 of this LQM. A cross-reference of the LQM to the NELAC requirements for quality manuals is presented in Table 2-2.

2.4.1 STL Quality Management Plan (QMP)

The requirements set forth in the QMP are applicable to all STL facilities. The policies and practices outlined in the QMP are minimum guidelines only.

Requirements that are more rigorous may be applied for specific client or regulatory programs.

2.4.2 STL Company-Wide Policies

Severn Trent Laboratories, Inc. has certain policies that apply company-wide. These policies are consistent with the QMP, and set forth requirements that all STL facilities are to follow.

2.4.3 Laboratory Quality Manual (LQM)

This STL Knoxville LQM along with the associated policies and SOPs, provides the criteria and specifications for the generation of environmental analytical data. The LQM with the SOPs provides QC criteria for standard procedures, facility-specific instrumentation, and reporting.

2.4.4 Laboratory Policies and Standard Operating Procedures

Laboratory policies provide further detail to the requirements of the LQM. Standard Operating Procedures (SOPs) describe step-by-step instructions for performing a method or activity. In addition, there are SOPs that relate to other support services performed in the laboratory. Details of SOP format and document control are described in SOP KNOX-QA-0018. SOPs that are actively used in this laboratory are listed in Table 8-2. SOPs are living documents and may supersede some requirements in this document until the LQM is updated.

2.4.5 Quality Assurance Project or Program Plans (QAPPs)

Regulations and contracts may contain QA requirements that are different from those described in this LQM. To address unique project requirements, Quality Assurance Project Plans (QAPPs) may be prepared and implemented. The requirements documented in a QAPP, as agreed to by STL Knoxville, take precedence over the LQM for that project. Typical specifications contained in a QAPP or similar documentation include:

- New or modified testing methods
- Unique QC logic
- Special requirements for equipment use and maintenance
- Special handling due to safety considerations
- Project-specific detection and reporting limits
- Project-specific accuracy and precision limits or the statistical treatment of data.
- Additional or unique documentation or records management requirements.

2.4.5.1 Quality Assurance Summary

Quality Assurance Summaries (QAS) or the equivalent (e.g., Client Requirement Checklist in LIMS) are used to distill client-specific requirements, typically documented in project QA plans, into a concise format, highlighting the requirements that are different than the laboratory standard practice. The summary describes for each project the required quality control samples, batching schemes, flagging conventions, deliverables, or other special client requests that may differ from routine laboratory operations. The QAS or equivalent is disseminated to laboratory operations by the Project Manager or Quality Assurance Manager to document client or program specific requirements. The QAS may be used alone or in conjunction with the project-specific QA plans.

2.4.6 Other Documents

Other documents that can affect the quality program may include the STL Corporate Safety Manual, memos, guidance documents, work instructions, and periodic management assessment reports. These documents may further define or guide the implementation of quality standards at STL, but shall not conflict with the LQM or diminish the effectiveness of the Quality System.

3.0 Document Control and Records Management

3.1 Objectives for Control of Quality Documents and Vital Records

Quality Documents - The quality documents discussed in Section 2 define the framework of the STL Quality System. Control and security of these documents are necessary to ensure that all staff have access to current policies and procedures at all times, to ensure that all changes to the policies and procedures are properly reviewed, to ensure that the history of use of documents can be reconstructed, and to ensure that confidential information is not improperly distributed. The system described in this section is designed to accomplish these objectives.

Vital Records – Vital records are the documents that provide objective evidence of the performance of a process or observations of an item. Records management ensures that results produced by the laboratory are scientifically and legally defensible, and ensures that project events can be reconstructed. Confidentiality of the records and records retention requirements are discussed in this section.

3.2 Document Control Procedures

Unambiguous identification of a controlled document is maintained by identification of the following items in the document header:

- unique document number/name,
- revision number,
- revision date,
- effective or implementation date (first page only),
- number of pages.

Controlled documents are marked as such, and the QA department keeps records of document distribution. Controlled distribution may be achieved by either electronic or hardcopy means. The effective date is the date when controlled copies are distributed. Controlled documents must be available in the immediate areas where the related work is performed. Details of the numbering system, required format, and restrictions for uncontrolled distribution of documents are in SOP KNOX-QA-0018 and the STL-wide Policy # S-Q-001.

3.3 Document Review, Approval and Revision

Controlled quality documents are authorized by the Laboratory Director, the Technical Director, and the QA Manager. They indicate their authorization by signing the cover page of the document. STL Knoxville quality documents, the individuals responsible for reviewing the documents, and the required frequency of review are listed in Table

2-3 and Table 2-4. In addition to periodic review and revision, quality documents must be revised when a procedure or activity changes in a significant manner.

Amendments to documents must be reviewed and approved by the same parties approving the original document, distributed in a controlled manner, and clearly indicated in the document. Obsolete versions of documents are removed from service when new revisions are issued. The QA Department maintains a record of history of use of all documents based on the effective date. For further details see SOP KNOX-QA-0018, "Preparation and Management of Standard Operating Procedures".

3.4 Records Management

Records may be either hardcopy or electronic copies. It is not required to maintain both if they are properly secured and are complete and true copies. The record keeping system allows for reconstruction of all laboratory activities that produced the analytical results. The history of the sample is readily understood through the documentation. This includes

- chain-of-custody records, including intra-laboratory and inter-laboratory transfers of samples;
- records identifying the personnel involved in sampling, preparation, calibration, and testing;
- observations, calculations, and derived data;
- information relating to laboratory facilities, equipment, analytical test methods, and related laboratory activities (e.g., sample preparation, standards preparation, and data verification);
- original records clearly identifying all subcontracted test data; and
- a copy of the final test report.

Requirements for data recording are described in STL Knoxville SOP KNOX-QA-0013, "Instrument Records and Logbooks". Details concerning control of electronic records are given in Section 6.

3.5 Document and Record Storage, Retention and Disposal

It is the policy of STL Knoxville that company records will be available to meet business needs and comply with all applicable legal records retention and disposition requirements. STL Knoxville retains copies of records in a manner that allows prompt retrieval of documents and records for inspection purposes. In accordance with NELAC, all quality documents and records are stored for at least five years.

Specific projects and regulatory programs have longer record retention requirements than the standard STL record retention time. Refer to the QMP Table 6 for a listing of examples of special program requirements.

When records, as contained in files, are transferred to a records storage area or off-site storage area, they shall be placed in suitable containers and include an inventory sheet (hard copy or electronic) prepared by the person submitting the records. The contents of each container shall be compared to the inventory sheet and the container labeled. If there are any discrepancies, the container and inventory sheet shall be reconciled. Archives are indexed such that records are accessible on a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Backup copies of electronic media are stored in archive facilities and are protected against deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented. Further details of the laboratory's document and records archiving process are described in SOP KNOX-AD-0001, current revision.

If the laboratory transfers ownership, vital records will be transferred to the new owner. If the laboratory goes out of business, vital records will be transferred to another operating STL laboratory or to our clients.

3.6 Data Confidentiality

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client.

In some cases the client may identify projects requiring confidentiality due to national security. Information concerning these projects will be limited only to those STL Knoxville associates with a need to know.

The audit reports supplied by federal, state, and local regulatory agencies are public information and can be released without written consent of those agencies. However, specific client audits are confidential and must be approved by the client before releasing them to a third party.

Internal evaluation reports shall be maintained as confidential documents and shall not be released for use outside the laboratory. External auditors may view internal audit reports as part of their on-site audit.

4.0 Staff Qualification, Orientation and Training

All activities performed by STL Knoxville shall be accomplished by qualified personnel. Each staff member must have the combination of experience and education needed to demonstrate the required knowledge for his or her position. Each must also have an appropriate general knowledge of laboratory operations, test methods, quality assurance and quality control procedures, and records management. Minimum training requirements are shown in Figure 4-1 at the end of this section. SOP KNOX-QA-0009 describes the details and documentation of the training process. The STL Corporate Safety Manual describes details for health and safety training.

4.1 Qualifications

STL Knoxville maintains job descriptions for all positions. These job descriptions specify the minimum qualifications for education and experience, knowledge and skills, which are necessary to perform at a satisfactory level. Qualifications of professional staff are documented by resumes that include academic credentials, employment history, experience, and professional registrations. A copy of each person's resume is maintained in an electronic file, and is readily available for inspection.

4.2 Orientation and Technical Training

Each new staff member shall receive orientation in quality, health and safety and technical training to the appropriate SOPs. Each new staff member shall be supervised in their assigned duties by their supervisor or a knowledgeable individual designated by the supervisor. The ability and authorization to perform independently shall be documented in the training files, as described below.

4.2.1 Quality Assurance (QA) Orientation

Each new staff member will receive a QA orientation. The QA Manager or designee will conduct this orientation within two weeks of the new employee's first day on the job. The orientation will, at a minimum, include the following topics:

- STL Quality System and hierarchy of quality documents (QMP, LQM, policies, and SOPs);
- key elements of the LQM and the Quality Control Policy (QA-003);
- introduction to the nonconformance memo (NCM) system and corrective action procedures;
- proper data recording practices;
- STL ethics agreement (within 1-2 days of first day on the job), including the potential consequences of unethical behavior; and

- A confidentiality agreement.

The QA orientation will be documented on a checklist, which is signed by the trainee. The documentation will be placed in the employee's training file.

4.2.2 Quality Training

Continued training in the mission and goals of the QMP and LQM shall be provided at least annually. This may be done in a single session or divided into separate sessions conducted at different times throughout the year. Formal training sessions are conducted and documented by the QA Manager or designee. In addition, each lab staff member shall read and document their awareness of the quality documents related to his or her position.

4.2.3 Ethics and Data Integrity Training

Training on ethics is provided to each new employee within 30 days of hire. Each new employee must sign an Ethics Statement, documenting their commitment to data integrity as part of the employee orientation. Annual ethics training is provided to all employees at STL Knoxville and annual data integrity training is required for all technical employees and project management staff. All employees must sign the Ethics Statement annually. Further details regarding ethics and data integrity training is found in the Ethics Policy, P-L-006, current revision.

4.2.4 Health and Safety, Orientation and Training

Each new employee, contract worker, or working visitor is required to go through health and safety orientation and training as described in the STL Corporate Safety Manual. The Health and Safety Coordinator must conduct the orientation as soon as possible after the individual reports to work and before chemicals are handled. More comprehensive health and safety training, both initial and on-going, must be completed at the frequency given in the STL Corporate Safety Manual. Additional training is required for radiation health and safety, as described in the Radiation Safety documents.

4.3 Training Files

Each active STL Knoxville staff member has an individual training file maintained by the QA Manager or designee. This file can be documented on paper forms or in a database. The following sections shall be included in the training files at a minimum:

- Resume –a hardcopy or an electronic file may be used.
- Quality Assurance - containing documentation of QA/QC orientation and training completed.
- Health and Safety - orientation and training documents.

- Technical Proficiency - initial and on-going demonstrations of proficiency, training courses or workshops on specific equipment or analytical methods are documented in this file. Note that proficiency with technical SOPs is documented on demonstration of capability forms (see next section).

Other types of records to be included in the training file include work place regulatory compliance training and professional development courses. The exact contents will vary depending upon a person's job function and tenure with the company. Details of requirements for training records and the approval process are given in SOP KNOX-QA-0009.

4.4 Technical Proficiency Demonstration

All new personnel are required to demonstrate competency in performing a particular method by successfully completing a Demonstration of Capability (DOC) before conducting analysis independently on client samples. On-going proficiency must be demonstrated annually.

DOCs are most commonly performed by analysis of four replicate QC check samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. As required by the referenced method, the accuracy and precision, measured as average recovery and standard deviation (using n-1 population), of the four replicates are calculated and compared to the method limits or against current laboratory limits if multi-laboratory method acceptance limits are not specified. The DOC Certification documentation must be signed by the Technical Director and the Quality Assurance Manager and filed in the employee's training file (see example in KNOX-QA-0009). The DOC Certification documentation must include a statement that the individual has read, understood, and agreed to perform the most recent version of the test procedure. Use of single-blind proficiency samples and other acceptable techniques to demonstrate on-going proficiency are described in SOP KNOX-QA-0009.

Figure 4-1 Employee Minimum Training Requirements

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Month 1	All
Ethics – New Hires	1-2 days of hire	All
Ethics – Comprehensive Data Inegrity Quality Assurance	30 days of hire (all training)	All Technical and PMs All
Ethics Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

**From date of initial employment unless otherwise indicated.*

5.0 Procurement of Supplies and Services

Controlling the quality of supplies and services is necessary to ensure that STL Knoxville provides high quality analytical services to our clients. The STL procurement program requires:

- assurance that purchased items and services meet requirements set by STL Knoxville and perform as expected;
- definition of the levels of documentation required for applicable technical and administrative procurement functions; and
- maintenance of records of all suppliers from whom we obtain services or supplies required for our analytical testing.

5.1 Selection of Vendors

Materials and supplies are purchased from approved vendors. Prospective vendors are selected based upon criteria appropriate to the materials or supplies provided. STL Corporate Policy P-PU-006 "Procurement and Contracts" details the process used. For national vendors and contracts, the vendor is selected by the STL Procurement Director through a competitive bidding process, strategic business alliance or negotiated vendor partnership. Potential vendors are required to complete a vendor acceptance application and are evaluated on the following criteria, as appropriate:

- the vendor's history of providing identical or similar products that perform satisfactorily in actual use;
- the vendor's service record and ability to provide a complete product line and commensurate service;
- the vendor's ability to administer inventory at the STL Knoxville facility through an inventory management system that will ensure correct stocking levels as well as shelf-life tracking;
- objective evaluation of the vendor's current quality records, supported by documentation;
- results of audits by STL of the vendor's technical and quality capabilities.

Vendors that provide measuring equipment, solvents, chemical standards, instrument service contracts, or subcontracted laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items.

5.2 Controlling Quality of Purchased Items

The quality of equipment, reagents, solvents, chemical standards, gases, and laboratory containers used in analyses must be of known quality so that their effect upon analytical results can be defined. These quality specifications are derived from analytical method requirements, project-specific requirements, and defined national standards for analytical testing. Quality specifications of materials are described in analytical SOPs. These quality specifications shall be included or referenced in the purchasing documents for the items being purchased. This includes specifications for the purity of standards, reagents,

or chemicals, and technical specifications for accuracy and precision (e.g., Class A volumetric glassware). Reference to a catalogue number, model, lot number, or chemical grade is sufficient.

The Laboratory Director has the responsibility for approving purchase orders. The section supervisors or designees are responsible for ensuring that the requested quality of materials ordered matches those received, for verifying that material storage is properly maintained and for removing materials from use when shelf life has expired.

5.2.1 Evaluation of Off-the-Shelf Items

For items that are used regularly by STL Knoxville where no unique requirements or specifications exist, the items may be purchased off-the-shelf. These items are ordered from the supplier on the basis of specifications set forth in the supplier's published product description. These include items such as glassware, filter paper, pipettes, and chromatography columns. The items are evaluated as a function of the standard analytical process.

5.2.2 Evaluation of Instruments

Evaluation of instruments purchased shall be conducted according to an acceptance testing plan. The acceptance testing plan may be defined by the vendor or the method demonstration requirements specified in the laboratory analytical SOPs. Acceptance criteria may include instrument reliability, sensitivity, stability, selectivity, accuracy, precision, and ability to interface with existing computer systems.

5.2.3 Evaluation of Critical Solvents and Acids

STL Knoxville is part of a group of STL laboratories that conducts additional evaluations for certain solvents and chemical reagents where our criteria for purity are more stringent than the vendor's. These chemicals are listed in Table 5-1. These chemicals are subject to analysis on a lot-by-lot basis before they are put into use. They are tested at one of the STL laboratories, and the chemical test results are evaluated by a designated quality representative. If the solvents or reagents meet the specifications given in SOP S-T-001, an approval memorandum is issued to all participating laboratories. The laboratories use the approved lots for analysis.

5.2.4 Evaluation of Chemical Standards and Reference Materials

Where available, chemical standards will be traceable to the National Institute of Standards Technology (NIST) or an equivalent source. This is largely limited to physical and inorganic chemical standards. If NIST traceability is not commercially available, commercially certified materials shall be used, which are

then tested for accuracy before reporting data. Details of the testing procedures and documentation are described in the laboratory SOPs. Standards must be received with a certification report from the vendor with information such as purity/concentration, traceability, lot number, expiration date, preparation date, unique identification number, formula weight, density, mass and/or volume of standards, and suggested storage requirements. Further details about labeling and handling of standards is described in Section 8 of this LQM.

5.2.5 Corrective Action for Failure to Meet Required Specifications

Corrective actions for failure of an item to meet required specifications are as follows:

- review of current supplies to eliminate the problem item
- notification to the STL Procurement Director to avoid additional problems at other STL labs
- return of the problem item to the vendor
- evaluation of the impact on product or process.

The QA Manager shall be notified of any significant or systematic quality problems. The STL Procurement Director and the STL Quality Assurance Director shall be notified of any quality problems with national vendors.

5.3 Procurement of Subcontract Laboratory Services

Whether external to STL or not, all subcontracting from the STL Knoxville laboratory to another laboratory is arranged with the consent and documented notification of the client, in a timely response that shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Documentation of required certifications from the subcontract facility are maintained in STL project records. Where applicable, specific QC guidelines, QAPPs, and similar project documents are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves an assessment of compliance with the required test method, QC requirements, documentation, as well as any special client requirements.

Project reports received from external laboratories are not altered and are included in original form in the final report provided by STL Knoxville. Intracompany subcontracting may also occur between STL facilities. The originating laboratory is responsible for communicating QA/QC, reporting, and other project requirements.

The final report from STL Knoxville clearly identifies what testing was performed by other laboratories. For projects requiring NELAC approval, the subcontractor must also be NELAC approved.

6.0 Computer Hardware and Software

The primary purpose of quality assurance systems for computer hardware and software is to protect the integrity of computer-resident data. Procedures are in place at STL Knoxville to assure that computer-resident data are accurate, traceable to a known source, protected against loss, and secure.

STL's computer and hardware controls are based on the guidance in EPA's "Good Automated Laboratory Practices" (GALP), August, 1995. This includes both corporate level Information Technology (IT) functions and STL Knoxville IT functions. Some GALP requirements, such as management responsibilities and the training program, are addressed in other sections of the LQM. Some corporate level IT functions, such as the system change management procedures, are described in more detail in corporate IT documents. Table 6-1 provides a cross reference of practices outlined in Section 8 of the GALP manual to corresponding sections of STL's QA and IT documents.

6.1 Computer Hardware

Computer hardware used in the generation, measurement, or assessment of client data shall be of appropriate design and adequate capacity to function according to specifications. Computer equipment must be installed in accordance with the manufacturer's recommendations and must undergo documented acceptance testing.

6.1.1 Wide-Area Systems

STL Knoxville's LIMS and the Office Network run on a wide-area network (WAN) serving multiple laboratories. The central node for the WAN is located at the Denver facility with multiple servers and routers. The central processor for QuantIMS is a mainframe computer. Records for the system architecture, testing and maintenance are documented in the System Log in Denver. Records for installation of the network hardware are maintained by the central System Administrator.

6.1.2 Local Systems

The local systems consist of computer equipment for analytical instruments, data evaluation, and upload to the LIMS. A local-area network (LAN) supports the local office software. Testing, maintenance, and repair of the local computer hardware is the responsibility of the STL Knoxville LAN Analyst. Documentation for the local systems is maintained by the LAN Analyst.

6.2 Facilities and Security

6.2.1 Central Computer Facilities

The environmental conditions of the facility housing the LIMS are controlled to protect against data loss. Access to the central computer facility in Denver is restricted by keypad entry used by IT staff. The central computer room is temperature controlled, and has an Uninterrupted Power Supply (UPS) plus a power generator to ensure that the WAN functions are not disrupted by power failures. Backup media, such as tapes and disks, are maintained daily. In addition, full volume backup copies of the raw data are shipped offsite to a commercial facility specially designed to store electronic data.

6.2.2 Local Computer Facilities

Facilities for housing local computer hardware meet manufacturer's recommendations. Electronic data is protected against environmental hazards such as fire, water damage, and strong electromagnetic fields. Backup copies of data files are made at regular intervals to protect against accidental loss through hardware or software failure.

6.2.3 Controlled Software Access

The integrity of data is also assured by maintaining limited access to administrative functions through a hierarchy of operating system shells controlled by passwords. Access is granted by the LAN analyst depending on a persons experience, training, and assigned duties.

Firewalls are in place to protect against unauthorized access from the Internet.

6.2.4 Virus Protection

Commercial virus protection programs are installed on all computers connected to the Internet or email to detect and remove computer viruses. LAN Analysts are notified whenever a virus is detected so that they can isolate any portions of the systems that may be at risk.

6.3 LIMS Raw Data

LIMS raw data and instrument raw data from instrument data systems are stored on the laboratory servers. The Systems Administrator and the LAN Analyst are responsible for maintaining the servers.

The individuals responsible for entering and recording raw data must be uniquely identified in the data, together with the date and time the data were entered. The instrument transmitting raw data must be uniquely identified, together with the date and

time of the transmission. Further data recording requirements exist to document manual integrations (see Policy S-Q-004 for details).

Procedures for verifying raw data are discussed in LQM Section 8.8.

6.4 Software

If computer software is used to acquire, process, or report client data, that software is tested to ensure that it correctly performs its intended function. Software is validated or verified, depending upon its complexity, size, and whether it was purchased or developed by STL. The following definitions are used by STL:

- Validation - the process of establishing documented evidence that provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.

- Verification - the process of checking the accuracy of automatically (electronically) calculated information.

6.4.1 Industry Standard Software

Industry standard software programs are defined as those that are purchased and widely used without modification to the program itself. The program is initially verified for use by using tests with known solutions to demonstrate that the program is operational for the desired application.

All purchased software must be used in accordance with the terms of its software license. Any use of software contrary to its license terms is expressly prohibited by STL.

6.4.2 Testing of STL-Developed Software

For programs used to process client data and developed within STL, and externally prepared programs that are modified by STL, validation or verification must be performed. The process used is dependent upon the function of the software as follows:

- Large complex systems consisting of several programs operating in unison to produce an intended result must be validated.

- For smaller software programs which only perform numerical manipulation, sample sets of numbers for which results are known should be processed and the results verified. In this case, known results are usually generated by performing

hand calculations using the same equations and procedures as the software to verify that the software produces identical results.

- Software that performs as part of instrument operation should be verified as previously described and by processing reference materials through the instrument system. Processed instrument response should be evaluated against expected instrument response and performance.

Refer to the IT SOP governing software development and testing, KNOX-IT-0001.

6.4.3 Software Revalidation

Whenever a program is changed, the change is evaluated to determine if it is significant enough to make revalidation necessary. If features have been added, previous test problems are rerun to demonstrate that their function has not been affected. New test problems are processed, as previously discussed, to verify added performance. If a software revision changes the basic operation of the program, complete revalidation of the program may be required.

Spreadsheets and unprotected software used to acquire, process, or report client data must be documented and reverified when changes are made. The test problems used to provide initial verification are reprocessed and the results compared to demonstrate that performance of the software is unchanged.

Laboratory operations are responsible for the generation of the validation and verification documentation for instrument level software. Completed records are kept as QC records. STL Information Technology is responsible for generation and maintenance of documentation relating to verification and validation of the STL QuantIMS system.

6.4.4 Version Control

The laboratory shall maintain copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of five years from its retirement date. The associated hardware, required to operate the software, must also be retained for the same time period.

6.5 Records Retention

As required by NELAC, electronic raw data and computer documentation are stored for a minimum of five years. See LQM Section 3.0 for further records retention details.

7.0 Contract Review and Project Planning

The generation of environmental analytical data is an intricate process. Success is dependent upon the timely execution of interrelated steps. For many environmental sampling and analysis programs, testing design is site or project specific and is not necessarily the same as the laboratory's standard service. It is STL's intent to provide both standard and customized laboratory services to our clients, provided that any special requirements are documented in writing and that performing the work in this manner does not cause the laboratory to violate relevant regulatory requirements. STL Knoxville has an organizational system in place to ensure that projects are properly planned prior to project initiation. This means that laboratory personnel understand project requirements, that the client clearly understands the lab's capabilities, that the laboratory has the facilities and resources needed to perform the required tests, that samples will be properly handled, that contingency plans are in place, and that analytical data will be reported in accordance with project needs.

7.1 Contract Review

The process of client request for proposal (RFP) and the laboratory's tender of a written response is a process of communication between both parties to understand project requirements and the laboratory's capabilities. All statements of work and contracts for new work entered into by STL Knoxville are reviewed by the Customer Service Manager (CSM) or designee. Agreements for continuing work are the responsibility of laboratory Project Managers (PMs) or the CSM. Depending on the size and scope of the proposed project, the Laboratory Director and other STL management staff can also be involved. Technical staff (Operations Manager, QA Manager, and IT staff) can be called upon to perform a review of the technical and QA/QC requirements. The CSM or PM, with this internal support, will work with clients to align project requirements with laboratory capabilities. Any contract requirement or contract modification communicated to STL verbally is documented and communicated to the client in writing. Any discrepancy between client requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract.

All contracts, Quality Assurance Project Plans (QAPP), Sampling and Analysis Plans (SAPs), contract amendments and documented communications become part of the project record as detailed in Section 3.5.

7.2 Certifications and Approvals

A necessary part of the review and work acceptance procedure is the evaluation of project needs for laboratory certification. The persons reviewing the prospective project must determine if project work plans or regulatory permits are tied to specific laboratory certifications or approvals. Where such requirements exist, the laboratory must have the certifications or approvals in place before the work begins. QA personnel coordinate with

the state certification agencies to maintain or add additional parameters. Copies of current laboratory certifications are maintained by the QA office, and are available upon request.

7.3 Data Collection Process

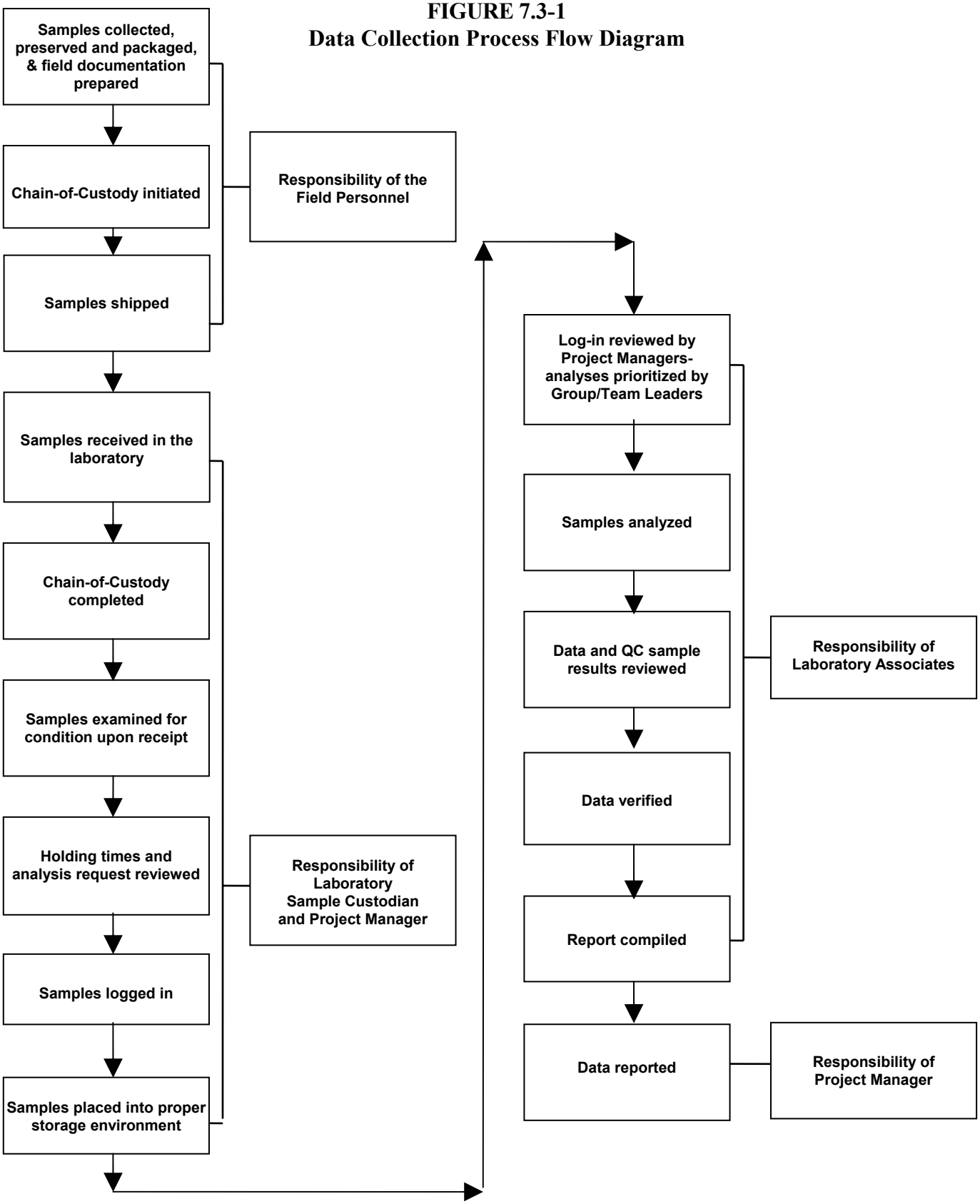
The data collection and data generation processes are shown in Figure 7.3-1. These processes are designed to produce analytical data that accurately reflect the nature of the site or sampling point.

7.4 Project Organizational Responsibilities

Each laboratory client is assigned a single point of contact, usually a PM, to ensure that there is a strong line of communication between the client and STL Knoxville. As a matter of policy, the CSM or designee, PMs, Quality Assurance personnel, the Laboratory Director and the Operations Manager work together to accomplish the following prior to receipt of samples at the laboratory:

- Samples are scheduled for arrival at the laboratory.
- All unique project requirements are identified and communicated to all appropriate personnel.
- Standardized client, state, federal, or STL programs are appropriately selected.
- Fully qualified subcontract laboratories are selected, if needed.
- A review is performed on all pre-project documents such as proposals, contracts, and/or QAPPs to identify the type of tests required and to ensure project requirements are within the scope of the laboratory being used.
- All appropriate and required preparations are made at the laboratory to accommodate or meet project requirements as described in proposals, contracts, and/or QAPPs.
- Determine that the laboratory has the capability and the capacity to analyze the samples including equipment, staff, space and workload.
- Determine that the laboratory is capable of meeting the required sample holding times and is able to report the resulting data within the time line specified by the client.
- All known safety hazards associated with the samples are communicated to all appropriate personnel.

FIGURE 7.3-1
Data Collection Process Flow Diagram



Approval and issuance of a quote, bid or contract document is documentation that this process has occurred. For particularly large or involved projects, STL Knoxville encourages our clients to visit the laboratory and/or participate in kickoff meetings with the laboratory staff. STL has found it very effective to invite the client into the laboratory's project preparations.

7.5 Communicating Project Requirements Internally

STL Knoxville PMs shall document all project-specific requirements prior to receipt of samples. The LIMS system requires the PM to enter a "quote" before any samples can be logged in. In addition to price information, the "quote" is a detailed technical specification of the work to be performed. The quote includes identification of project personnel, numbers and types of samples, tests to be performed, reporting limits, QC to be performed, control limits, data qualifier flags to be used, significant figures to be used, and the types of deliverables required. This is the primary means of communicating routine project requirements to laboratory personnel.

Brief non-routine project requirements are entered into the comments section of the Client Requirements Checklist portion of the quote. The Checklist is reviewed by analysts as analyses are being scheduled and before testing has started. If the special requirements are too lengthy for the Client Requirements Checklist, the PM must prepare a Quality Assurance Summary (QAS) or equivalent, which is a written document describing all requirements that are different than routine work. The QAS is referenced in the LIMS (e.g., the Client Requirements Checklist), and is distributed by the PM to each of the operational groups involved. For complex projects, project kickoff meetings are conducted by the PM with each of the operational groups involved.

7.6 Contingency Planning

An effective QA program must emphasize contingency planning, i.e, actions to prevent problems from recurring, and to ensure timely and effective completion of a measurement effort. The following are considered relative to contingency planning.

7.6.1 Staffing

A primary objective is to ensure that qualified staff are available to perform the necessary analytical work, regardless of employee turnover, vacation, illness, or other absences. STL Knoxville is a relatively large laboratory with multiple staff capabilities for the majority of tests performed. However, other sources of trained personnel are potentially available to assist in the event of unforeseen absences. Given sufficient time for necessary orientation, temporary agency staff can be used. More significantly, STL is a large laboratory network and a large pool of qualified staff can be made available from other STL laboratories.

7.6.2 Backup Instrumentation

Within STL Knoxville, duplicate instrumentation is available for most methods to allow uninterrupted work flow if one piece of equipment fails. The laboratory may also choose to lease equipment. However, in circumstances where a catastrophic instrument failure occurs, alternative, but equivalent, methods may be recommended to the client for approval.

7.6.3 Other Contingency Plan Elements

- Preventive Maintenance - STL's preventive maintenance program is designed to minimize analytical instrument malfunctions, permit simple adjustments, and to ensure fewer and shorter breakdowns of critical analytical equipment. (See Section 8.11, "Preventive Maintenance and Service".)

- STL Laboratories & Subcontractor Laboratories - To support the laboratory during peak periods or in the event of a critical instrument malfunction, STL has the capability to arrange for the use of other STL laboratories or other qualified analytical laboratories as subcontractors for short-term backup analytical support. However, use of a subcontractor laboratory must be approved by the client in writing. For projects requiring NELAC approval, the subcontractor must also be NELAC approved. See Section 5.3 for other procedures related to the control of subcontract laboratory services.

- Uninterruptible Power Supply - An Uninterruptible Power Supply (UPS) system provides line conditioning and backup power to the LIMS computer system/server and critical analytical instruments. This contingency allows sufficient time for the main computer system and instruments to be shut down and for data archival. All electronically generated data that are stored on the main computer systems are backed up at regular intervals.

8.0 Work Processes and Operations

Many activities related to environmental project activities are planned and designed externally to the laboratory or field operation, and are presented to the laboratory in the form of a contract, work plan, sampling and analysis plan (SAP) or QA Project Plan (QAPP). Laboratory and field activities are in turn planned, implemented, and assessed by STL to meet client requirements according to approved procedures and methodologies. The LQM provides the systems to document and implement these activities. The execution and assessment of the implemented operational systems are detailed in STL SOPs. The entire process is assessed on a regular basis for conformance to prescribed requirements.

Standard practices for STL Knoxville operations are detailed in this section. Specific project or program requirements may differ from those described here, but they must be explicitly stated in approved contracts, work plans, QAPPs or other project documents. Special project requirements can generally be accommodated provided that they are properly documented, communicated, and they do not cause the laboratory to violate relevant regulatory requirements.

Table 8-1 lists the test methods performed by STL Knoxville. Table 8-2 lists the SOPs associated with those methods. Table 8-3 provides a list of the major equipment in place at the laboratory, and Figure 8.11-1 (at the end of this section) shows the laboratory floor plan.

8.1 Traceability of Measurements

STL documents all laboratory activities in sufficient detail to allow their reconstruction. To this end, documentation is generated to trace a sample from its point of origin, through receipt in the laboratory, analysis, reporting and disposal.

The required documentation includes, but is not limited, to:

- Chain of custody documenting movement and possession of samples
- Sample preparation
- Sample analysis
- Calibration and QC data associated with the samples
- Instrument maintenance
- Control of ancillary equipment and materials (e.g., deionized water and glassware)
- Sample disposal
- Final reports

These topics are described in this section. Traceability of chemical standards is also discussed in Section 5.2.4.

8.2 Analytical Methods

Whenever possible, STL operations use industry- and regulatory agency-recognized analytical methods from source documents published by agencies such as the Environmental Protection Agency (EPA), Department of Energy (DOE), and the American Society for Testing and

Materials (ASTM) as described in STL's SOPs. Analytical methods performed by STL Knoxville are given in Table 8-1. The methods approved by a NELAC Accrediting Authority are indicated in the table.

Method performance data, as described in Section 8.2.2 below, are developed by the laboratory operations staff to demonstrate method proficiency. The operations staff and the QA staff evaluate and approve the performance data before a methodology is performed routinely. The method must also be described and documented in an SOP.

8.2.1 Standard Operating Procedures

SOPs are required for all repetitive analytical and administrative activities ranging from the receipt of samples in the laboratory through their analysis, reporting, and subsequent disposal. Training, health and safety procedures, QC, method procedures, and instrument and equipment calibrations are included in SOPs. SOP requirements are discussed in the SOP KNOX-QA-0018, "Preparation and Management of SOPs". The specifications in the SOP meet NELAC requirements. Table 8-2 lists laboratory standard operating procedures.

New SOPs and proposed SOP revisions are reviewed by technically qualified lab personnel. SOPs are controlled documents and are distributed and maintained as described in SOP KNOX-QA-0018. Requirements for SOP approval and frequency of review are listed in Tables 2-3 and 2-4. All significant modifications to the published method are described in a section of the SOP. All operations must be performed as described in these SOPs.

Planned changes in procedure, which may occur due to expected sample matrix effects or project requirements, are documented in the project files. These planned changes may be documented using nonconformance memos, NCMs (see discussion of NCMs in section 9.1), project-specific case narratives, or as modifications or additions to associated QAPPs.

Unplanned deviations in the SOPs, which may occur due to sample matrix or other events, are documented in NCMs and in the project-specific case narratives.

8.2.2 Method Validation and Verification

Before analyzing samples by a new method or method modification, the method must be verified or validated and analyst capability must be demonstrated (see Section 4.4).

8.2.2.1 Method Verification

Method verification is required for methods developed by authoritative agencies, such as EPA or ASTM. The level of verification can vary

depending on the type of method or level of modification, but generally should include:

- Determination of method sensitivity,
- determination of working range,
- an initial demonstration of capability (as specified by NELAC), and
- a written SOP or project-specific written protocol.

Each of these is described in the next section.

8.2.2.2 Method Validation

A complete validation is required for methods developed by STL Knoxville. While method validation can take a variety of courses, the following are the key concerns:

Determination of Method Selectivity
Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity
Method sensitivity is normally demonstrated using the 40 CFR Part 136 Appendix B method detection limit protocol (see MDLs, section 8.2.3, below), but can also be based on variance of blank results and signal-to-noise ratios.

Determination of Interferences
This is demonstrated by analyzing samples of the matrix of interest that are known to be free of the analyte(s) of interest.

Determination of Range
In most cases, analytical range is determined and demonstrated by comparison of the response of an analyte at different concentrations to targeted criteria. The curve is used to establish the range of quantitation, with the lower and upper values representing the upper and lower quantitation limits.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analyses of samples of known concentration. The resulting percent recovery and relative standard deviation, or other precision measure are calculated and compared to a set of target criteria.

Documentation of Method
The method is formally documented in an SOP (see SOP KNOX-QA-0018 for details).

Continued Demonstration of Method Performance
Continued ability of the lab to perform the method is addressed in the SOP. Generally this is accomplished with the specified calibration and batch QC requirements.

8.2.3 Method Detection Limits

It is STL Knoxville's policy to follow the specifications in the U.S. EPA 40 CFR Part 136 Appendix B in determining MDLs for chemical tests. The STL requirement for this procedure is detailed in the SOP S-Q-003 "Method Detection Limit Studies," and further defined in the STL Knoxville attachment to the SOP. This procedure requires that the MDLs be determined for each analyte of interest representing the aqueous and solid matrices within the capability of the primary analytical methods. The laboratory's MDLs are subject to update. Current MDLs are available from the laboratory upon request.

8.2.4 Instrument Detection Limits

Instrument Detection Limits (IDLs) are required to be performed quarterly for metals constituents and cyanide when analyses are performed in support of Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) activities or when the USEPA CLP SOW protocol is required. When required, IDLs will be performed as described in the associated laboratory SOPs.

Prior to acceptance and use for reporting purposes, all data from detection limit studies and reporting limits must undergo technical review and approval by the laboratory management and QA staff.

8.2.5 Reporting Limits

Reporting limits are established and modified within STL according to the SOP S-T-002, "Reporting Limits for Laboratories Using QuantIMS." Two reporting limit conventions are discussed in the policy: the standard Reporting Limit (RL) and the Project-Specific Reporting Limit (PSRL). The standard STL RL is the lowest level at which measurements become quantitatively meaningful. The RL is always greater than the statistically determined MDL. PSRLs are used when project data quality objectives (DQO) require a reporting limit other than the standard RL. PSRLs tailor STL's product to meet customer requirements. Higher PSRLs may be established based on maximum contaminant levels (MCLs), applicable or relevant and appropriate requirements (ARARs), or project-specific DQOs. PSRLs below the lab's standard RL may be used, but they must be supported by the MDL and the instrument calibration.

A standard at the PSRL taken throughout the entire preparation and extraction procedure may be used to support a PSRL with QA approval. The STL RLs and PSRLs are maintained in the LIMS. STL Knoxville RLs are provided in Tables 8-4-1 through 8-4-26.

8.2.6 Estimation of Uncertainty Measurement

STL Knoxville has developed and applied a procedure for estimating the measurement uncertainty associated with the results reported. Refer to SOP KNOX-QA-0019, current revision, for details regarding the calculation and reporting of the measurement uncertainty. The estimate of measurement uncertainty is available to clients upon request.

8.3 Data Quality Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994)[1]. Typically, DQOs are identified during project scope and the development of sampling and analysis plans. In this LQM, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. The EPA has established six primary analytical DQOs for environmental studies. These DQOs are precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QC samples and their applications, described in Section 8.4, are selected on the basis of method or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic and organic analyses may include calibration or instrument blanks, method blanks, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), post-digestion spikes (PDSs) and surrogate spikes.

8.3.1 Precision and Accuracy

Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed, similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate

[1] "Guidance for the Data Quality Objectives Process", EPA 600/R-96/005, September 1994.

measurements. Table 8-5 illustrates the formula used to calculate units of precision (i.e., RSD and RPD).

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

The precision and accuracy measures that are to be used in evaluating inorganic and organic constituents at STL are provided in Tables 8-7 through 8-9, in method-specific SOPs, and in the documentation for the analytical method of interest.

Precision and accuracy are determined, in part, by analyzing data from matrix spike and matrix spike duplicates, unspiked duplicates, LCSs, PDSs and single blind audit samples. A description of these QC samples is provided in Section 8.4.

8.3.2 Completeness

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. Typically, a data quality objective of at least 90% completeness is specified in quality assurance project plans.

8.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy, therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At STL, efforts must be made to analyze an aliquot that is representative of the original sample, and to reasonably homogenize the sample before subsampling.

8.3.4 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

8.4 Quality Control Samples

Two types of Quality Control (QC) samples are field QC samples and laboratory QC samples. Field QC samples are collected during the sampling event and are useful in determining sampling precision and accuracy and monitoring for contamination that may

occur during collection, transport or storage of environmental samples. Laboratory QC samples are routinely added at the laboratory to the normal sample stream. Successful analysis of these samples demonstrates that the laboratory is operating within prescribed requirements for accuracy and precision. In addition, by utilizing matrix-specific laboratory QC samples, information regarding the effect of the matrix or field conditions on the analytical results can be obtained. The following sections describe common field and laboratory QC samples.

8.4.1 Field QC Samples

When field QC sample collection and analysis are required for a project, it is the responsibility of the client to ensure that this sampling is performed correctly and at the project-required frequencies. Field QC samples may or may not be identified as such to the laboratory and are considered by the laboratory as field samples for the purpose of QC batching, sample preparation and analysis. Field QC sample results are reported in the same manner as actual field samples, unless a specific deliverable is requested by the client. No correction of the analytical data is done in the laboratory based on the analysis of field QC samples.

Field QC sample types, applicability to organic and inorganic analyses, precision and accuracy applications and by whom they are introduced are summarized in Table 8-6.

8.4.2 Laboratory QC Samples

Laboratory performance QC is required to ensure the laboratory systems (instrumentation, sample preparation, analysis, data reduction, etc.) are operating within acceptable QC guidelines during data generation as required to meet the client's objectives. Laboratory QC samples consist of method blanks (MB), instrument blanks, laboratory control samples (LCS) and calibration verification samples. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. Typically, this includes matrix spikes (MS), matrix spike duplicates (MSD), sample duplicates, PDS/PDSD and the use of surrogate compounds.

Laboratory and matrix-spike QC sample types are summarized in Tables 8-7 through 8-9. The following sections provide descriptions of laboratory QC samples and their frequency of use. Policy QA-003, "Quality Control Program", describes in detail the QC data evaluation process.

8.4.2.1 Quality Control (QC) Batch

The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period. This definition of a QC batch is utilized by STL unless there is clear regulatory guidance, contract

specifications, or differing client requirements that are explicitly documented. Further details and requirements for the application of the definition of QC batch are described in Policy QA-003.

8.4.2.2 Method Blank

The method blank (MB) is a QC sample that consists of all reagents specific to the method and is carried through every aspect of the procedure, including preparation, cleanup, and analysis. The method blank is used to identify any interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Potential sources of contamination include solvents, reagents, glassware, other sample processing hardware, or the laboratory environment. In general, the method blank is a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples, that is processed as a sample. In the event that no appropriate solid matrix exists, deionized water may be used. The volume or weight of the method blank must be approximately equal to the sample volume or sample weight processed. A method blank shall be prepared with each group of samples processed.

8.4.2.3 Instrument/ Calibration Blank

The instrument blank is an unprocessed aliquot of reagent used to monitor the contamination of the analytical system at the instrument. System contamination may lead to the reporting of elevated analyte concentrations or false positive data. The instrument blank does not undergo the entire analytical process and generally consists of an aliquot of the same reagent(s) used for a sample dilution. Instrument blanks are also referred to as initial calibration blanks (ICBs) and continuing calibration blanks (CCBs).

8.4.2.4 Laboratory Control Sample

A laboratory control sample (LCS) is a laboratory-prepared, suitable clean matrix sample that is fortified with target analytes or a solid reference material purchased from an approved vendor. The LCS contains selected target analytes specified in the method, and must contain the same analytes as the matrix spike and matrix spike duplicate. For certain regulatory or client programs, an LCS may contain a full list of analytes. However, in these cases, a subset of analytes, as defined by the program, may be used to determine the acceptability of a batch of sample data. The LCS recovery data are used to monitor the analytical method performance in terms of analytical accuracy. On-going evaluation of the LCS recoveries demonstrates that the laboratory is performing the method within statistical control (i.e., accuracy and precision) in the absence of matrix interference. The LCS results, coupled with MS data, help determine whether the laboratory performed the method correctly and if the sample matrix affected

the analytical results. When a laboratory control sample duplicate (LCSD) is required, a percent recovery for each target analyte is calculated, as well as a relative percent difference (RPD) between the LCS and the LCSD.

8.4.2.5 Matrix Spike

A matrix spike (MS) is an environmental sample to which known concentrations of selected target analytes have been added. MS samples are analyzed to evaluate the effect of the sample matrix on the analytical methodology. MS samples are generated by taking a separate aliquot of an actual field sample and spiking it with the selected target analyte(s) prior to sample extraction. The MS sample then undergoes the same extraction and analytical procedures as the unfortified client sample. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked and not on samples collected at other locations that are included in the QC batch.

8.4.2.6 Matrix Spike Duplicate

A matrix spike duplicate (MSD) is a second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with the associated sample and MS sample. The results of the MS and MSD are used together to determine the effect of a matrix on the accuracy and precision of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results may have immediate bearing only on the specific sample spiked and not all samples in the QC batch.

8.4.2.7 Sample Duplicate

A sample duplicate is a second aliquot of an environmental sample taken from the same sample container that is processed identically with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The results are compared to determine the sample homogeneity and the precision of the analytical process.

8.4.2.8 Surrogates

Surrogates are organic compounds that are similar in chemical composition and behavior to the target analytes but that are not normally found in environmental samples. Surrogates are added to all appropriate samples and QC samples being tested for organic analytes to monitor the effect of the sample matrix and the procedure on the accuracy of the process.

8.4.2.9 Post-digestion Spike

A post-digestion spike is created by spiking target analytes into a prepared portion of a sample just prior to analysis. It provides information on matrix effects encountered during analysis such as suppression or enhancement of instrument signal levels. It is most often used in elemental analysis involving various forms of atomic emission or atomic absorption spectroscopy. A single analytical spike serves as a single point application of the "method of standard additions" or MSA.

8.4.2.10 Interference Check Sample

An interference check sample (ICS) is a solution containing known concentrations of both interfering and analyte elements. Analysis of this sample by ICP can be used to verify background and interelement correction factors.

8.4.2.11 Internal Standards

An internal standard (IS) is a compound or element with similar chemical characteristics and behavior in the analysis process to the target analytes, but is not normally found in environmental samples. The internal standard is usually added after sample preparation. The primary function of the internal standard is quantitation; however, it also provides a short-term indication of instrument performance. For isotope dilution methods, internal standards are added during sample preparation and are used for quantitation.

8.5 Data Collection Operations

Laboratory analyses are designed to produce data that are representative of existing conditions present at the time the sample was obtained. The data collection design includes field sampling events, sample handling and custody, analytical operations, data recording procedures, data assessments, data verification, and data reporting requirements and techniques to assess limitations of data use. These operations are discussed in this section through section 8.10.

8.5.1 Field Collection and Shipment

In order to provide a sample that most accurately represents the test matrix, field sample collection personnel must abide by the sample collection guidelines and procedures established by involved regulatory agencies. A significant part of the efforts of regulatory agencies include the use of appropriate sample containers, chemical and physical preservation techniques, and observance of specified holding times. It is imperative that all samples be collected and preserved according to the appropriate analytical method specified in the QAPP (if one exists). Although the sampling may be performed by non-STL personnel, the importance of sampling and transportation of the sample to the laboratory is understood and must be considered during data validation.

The sample acceptance policy must be made available to sample collection personnel (refer to section 8.5.3.2).

Field personnel are responsible for labeling each individual sample collected with the following information:

- Project name
- Unique client sample number
- Sample location (including as appropriate: borehole and depth or grid coordinates)
- Sampling date and time
- Sample preservation
- Analysis required.

An overriding consideration for the resulting analytical data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody, and disposal must be documented to accomplish this. Figure 8.5-1 shows an example Chain-of-Custody (COC) form that is used by the STL laboratory to document this evidence. Field personnel are responsible for initiating the COC form.

The prompt shipment of samples to the laboratory is necessary to ensure that required holding times are met. Samples should be shipped by an overnight carrier, be hand-delivered, or transported in a manner that assures prompt delivery to the laboratory. Some sites require an extensive radioactive screening process before a sample may be shipped. In these cases, it is imperative for the Project Manager to maintain good communication with the client to assure proper staffing of the laboratory in response to a decreased holding time.

Radioactive samples that are shipped to STL Knoxville must be screened prior to delivery to the laboratory and found not to contain radioactivity that exceeds the level stated in the laboratory's radiological license. Samples received by STL Knoxville containing radioactivity exceeding the license limit will immediately be returned to the project site.

FIGURE 8.5-1
Example STL Chain-of-Custody Form

Reference Document No. _____
Page 1 of _____

Project Name/No.: _____
Sample Team Members: _____
Profit Center No.: _____
Project Manager: _____
Purchase Order No.: _____
Required Report Date: _____

Sample Shipment Date: _____
Lab Destination: _____
Lab Contact: _____
Project Contact/Phone: _____
Carrier/Waybill No.: _____

Bill To: _____

Report To: _____

ONE CONTAINER PER LINE

Sample Number	Sample Description/Type	Date/Time Collected	Container Type	Sample Volume	Preservative	Requested Testing Program	Condition on Receipt	Disposal Record No.

Special Instructions:

Possible Hazard Identification:
Non-hazard ❑ Flammable ❑ Skin Irritant ❑ Poison B ❑ Unknown ❑

Sample Disposal:
Return to Client ❑ Disposal by Lab ❑ Archive _____ (mos.)

Turnaround Time Required:
Normal ❑ Rush ❑

QC Level:

1. Relinquished by: *(Signature/Affiliation)*	Date: _____ Time:	1. Received by: *(Signature/Affiliation)*	Date: _____ Time:
2. Relinquished by: *(Signature/Affiliation)*	Date: _____ Time:	2. Received by: *(Signature/Affiliation)*	Date: _____ Time:
3. Relinquished by: *(Signature/Affiliation)*	Date: _____ Time:	3. Received by: *(Signature/Affiliation)*	Date: _____ Time:

Comments:

8.5.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times

8.5.2.1 Sample Containers

A sample container is defined as the sealed enclosure, usually made of plastic or borosilicate glass, that the sample is collected in and stored in until analysis. All sample containers provided by STL operations for environmental sampling are new, with the exception of some air sampling canisters, which must be recertified before reuse, and demonstrated to be clean for their appropriate use. All documentation certifying sample container cleanliness must be maintained by the laboratory or the vendor and can be provided to the client upon request. The sample containers to be supplied are listed in Tables 8-10 through 8-12. Container volumes listed in these tables may be decreased with the approval of the laboratory QA Manager or Technical Director to accommodate reduced sample volumes required by the facility SOP.

8.5.2.2 Shipping Containers

Shipping containers are defined as the sealed enclosure in which the sample containers are stored during shipment from the sample collection site to the analytical laboratory. Shipping containers must be of sufficient number and size to accommodate the samples in an upright condition. Shipping containers must also meet all requirements for the shipment of environmental and/or radioactive samples.

Packaged samples must be shipped to the analytical laboratory in a safe manner that preserves the integrity of the samples. The most common method of sample shipment employs coolers or ice chests that are sealed with custody tape and shipping tape. These coolers must be durable and resistant to crushing during shipment. All coolers must be well maintained and cleaned to prevent cross-contamination of the samples. It is the ultimate responsibility of the person collecting and packaging the sample for shipment to ensure that the shipping containers are clean and functional.

To help prevent sample breakage during shipment, additional consideration must be given to providing shock absorbency to all samples packaged inside the shipping container. Use of bubble-wrap around each sample container is the best way to provide this protection. Foam packing materials and vermiculite are also successfully used.

8.5.2.3 Sample Preservatives

Most analytes have a finite holding time in a given sample matrix. Sample

preservation is the chemical or physical means by which samples are treated during and/or following sample collection to aid in the stability of the analytes of interest in that matrix. Sample holding times are adversely affected when samples are improperly preserved or shipped unpreserved. The preservation of samples at the time of sample collection follows the requirements of the analytical methods used. This preservation includes the addition of reagents to deter chemical and biochemical degradation and the maintenance of refrigeration during transit and ultimate storage in the laboratory. The required preservatives for the analyses to be performed on each matrix are included in Tables 8-10 through 8-12.

8.5.2.4 Sample Holding Times

Holding time is defined as the maximum allowable time a sample can be stored after sample collection and preservation (or laboratory receipt for CLP) until appropriate processing occurs (preparation or analysis). The holding time may vary according to method or client requirements. Tests designated with holding times as "analyze immediately or ASAP" are considered parameters that should be tested by field personnel or on-site. A system is in place to ensure that holding times are monitored by each group within the operating unit. It is the responsibility of each STL associate processing the sample to assure that holding times are met. STL is responsible for meeting all holding times for properly preserved samples received within 48 hours of collection or if less than half the holding time has passed. If these conditions are not met, STL will attempt to expedite sample analysis as soon as possible. Sample holding times are listed in Tables 8-10 through 8-12.

8.5.3 Sample Handling

STL Knoxville's SOP KNOX-SC-0003 describes the sample receipt and log-in process in detail. The following sections describe the general policies followed by STL.

8.5.3.1 Sample Receipt

Samples shall be received and logged in at STL by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of gloves, a lab coat and safety glasses.
- Examine the shipping containers to verify that the custody tape is intact.
- Examine all sample containers for damage.
- Open shipping containers in adequately ventilated areas to assure worker

safety.

- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the COC.
- Compare samples received against those listed on the COC.
- Verify that sample holding times have not been exceeded.
- Examine all shipping records for accuracy and completeness.
- Verify sample pH if it is required preservation for the scheduled analysis (except VOA samples) and record on the COC. Also check for residual chlorine (if required) and note on the COC.
- Sign and date the COC immediately (only after shipment is accepted) and retain the waybill and/or the waybill number.
- Note any problems associated with the coolers and samples on the COC, immediately initiate a Condition Upon Receipt Report (CUR) or equivalent form, and notify the PM who, in turn, notifies the client.
- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test.
- Place the samples in proper laboratory storage.

A CUR or an equivalent form/system is generated by sample control during the sample log-in process and is used to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and may not require corrective actions to be taken within the laboratory. The affected client shall be notified by the PM or designee of all CUR anomalies for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. The completed CUR form shall be stored in the project file. An example CUR is shown in Figure 8.5-2. The report narrative will include an explanation of sample receiving related anomalies. Further details are given in SOP KNOX-AD-0004.

8.5.3.2 Exceptions or Discrepancies

STL reserves the right to reject samples, as required by NELAC, for any of the following reasons:

- No custody seals as required by project.
- No chain of custody documentation provided.
- Preservation inappropriate for analysis requested.
- Sample container inappropriate for analysis requested.
- Sample received out of holding time for analysis requested.
- Incomplete sample information provided.
- Discrepancies between COC and sample labels.
- Samples have high levels of polychlorinated dibenzo-p-dioxins/ dibenzo furans (PCDD/PCDFs.)

- Samples have a high level gross alpha or beta radiation.
- Samples are from a site known to contain chemical warfare agents (CWAs) and the samples have not been screened for them.

These or any other project exceptions or discrepancies are discussed with the client and agreed upon actions are taken.

8.5.3.3 Sample Log-in

Sample log-in activities at STL are fully documented in SOP KNOX-SC-0003. The following is a general description of the log-in process:

- Enter the samples in the LIMS which contains the following information at a minimum:
  - Project name or identification number
  - Unique sample numbers (both client and internal laboratory)
  - Type of samples
  - Required tests
  - Date and time of laboratory receipt of samples
  - Field ID supplied by field personnel
- Notify the PM and appropriate Group/Team Leader(s) of sample arrival
- Place the completed COCs, waybills, and any additional documentation in the project file.

8.5.3.4 Sample Storage

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required.
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples, when known, should be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests should be monitored twice per month by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial).
- Security of samples within the laboratory.

The requirements listed in Tables 8-10 through 8-12 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. The PM or designee shall have the responsibility of notifying the Group/Team Leaders or their designees if there are any samples that must be analyzed immediately because of holding time requirements.

FIGURE 8.5-2
Example STL Knoxville Condition Upon Receipt Anomaly Report (CUR)

STL KNOXVILLE SAMPLE RECEIPT/CONDITION UPON RECEIPT ANOMALY CHECKLIST

Client: _____ **Project:** _____ **Lot Number:** _____

Review Items	Yes	No	NA	If No, what was the problem?	Comments/Actions Taken
1. Do sample container labels match COC? (IDs, Dates, Times)				☐ **1a** Do not match COC ☐ **1b** Incomplete information ☐ **1c** Marking smeared ☐ **1d** Label torn ☐ **1e** No label ☐ **1f** COC not received ☐ **1g** Other:	
2. Is the cooler temperature within limits? (North Carolina, 1668, 1613B: 0-4°C; VOST: 10°C) (Cooler temp should be used only if no temp blank.)				☐ **2a** Temp Blank = _____ ☐ **2b** Cooler Temp = _____	
3. Were samples received with correct chemical preservative (excluding Encore)?				☐ **3a** Sample preservative =	
4. Were custody seals present/intact on cooler and/or containers?				☐ **4a** Not present ☐ **4b** Not intact ☐ **4c** Other:	
5. Were all of the samples listed on the COC received?				☐ **5a** Samples received-not on COC ☐ **5b** Samples not received-on COC	
6. Were all of the sample containers received intact?				☐ **6a** Leaking ☐ **6b** Broken	
7. Were VOA samples received without headspace?				☐ **7a** Headspace (VOA only)	
8. Were samples received in appropriate containers?				☐ **8a** Improper container	
9. Did you check for residual chlorine, if necessary?					
10. Were samples received with ≥½ HT remaining?				☐ **10a** Holding time expired ☐ **10b** <½ HT left	
11. For rad samples, was sample activity info. provided?				☐ Incomplete information	
12. For SOG water samples (1613B, 1668A, 8290, LR PAHs), do samples have visible solids present?				If yes, was SOG notified? _____	
13. Are the coolers intact?				☐ **13a** Leaking ☐ **13b** Other:	
14. Was COC relinquished? (Signed/Dated/Timed)				☐ **14a** Not relinquished by client	
15. Are tests/parameters listed for each sample?				☐ **15a** Incomplete information	
16. Is the matrix of the samples noted?				☐ **15a** Incomplete information	
17. Is the date/time of sample collection noted?				☐ **15a** Incomplete information	
18. Is the client and project name/# identified?				☐ **15a** Incomplete information	
19. Was the sampler identified on the COC?				☐ **15a** Incomplete information	

Quote #: _____ PM Instructions: _____

Sample Receiving Associate: _____ Date: _____

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8.5.3.5 Internal Sample Chain-of-Custody and Interlaboratory Transfers

Sample custody within STL Knoxville is described in SOP KNOX-SC-0004. Internal COC may be required for programs defined by state or federal agency. The sample custody documentation shall include the following minimum requirements:

- Name of associate taking custody of the sample from the sample storage area for preparation or analysis.
- Dates sample removed from and returned to the sample storage area.
- Identification of tests to be performed on the sample aliquot(s) selected by the associate.
- Sample matrix.
- Laboratory sample numbers.
- Sample storage location.

Access at STL Knoxville is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

Samples transferred to a different laboratory than the original receiving facility are transferred under chain-of-custody (COC). The COC is maintained whether the laboratory is another STL facility or a subcontracted laboratory. If all sample containers are transmitted, the original copy of the client's COC form will be used to document the relinquishing of the samples and will accompany the samples to the destination. A copy of the completed COC form shall be retained in the laboratory project file. In the case where an aliquot of a sample is shipped from the laboratory, a new COC will be generated by the laboratory and shipped with the sample aliquot. The original COC will be retained in the project file at the site holding the original sample container until the sample results are reported.

Samples are not transferred to other STL facilities or to subcontractor laboratories without prior approval of the client.

8.5.3.6 Subsampling

Sample preparation procedures are referenced in the method SOPs. SOP KNOX-QA-0006 describes processes for obtaining representative subsamples.

8.5.3.7 Sample Disposal and Return Chain-of-Custody

After the requested analyses on the samples have been completed, any remaining portions of the samples will be maintained by the sample custodian until the

samples are disposed of or returned to the client. Sample disposal procedures and documentation are described in SOP KNOX-SC-0005. STL's routine sample retention period is at least thirty days after the analytical report is issued to the client, unless otherwise specified by the client.

For Nuclear Regulatory Commission (NRC) or state licensed laboratories, a real-time inventory of all radioactive isotopes contained in the laboratory (including radioactive samples), as required by the NRC or state, is maintained by the Radiation Safety Officer (RSO). If the quantities of radioactive materials in-house approach the limits stipulated by the laboratory NRC or state license, appropriate action must be taken to ensure the licensed level is not exceeded. This may involve returning samples to clients immediately.

If samples are returned to the client rather than disposed of by the laboratory, the original COC or a new COC is used to document custody transfer back to the client from the laboratory. A copy of the completed COC is retained in the laboratory project records.

8.5.4 Calibration Procedures and Criteria

All equipment and instruments used at STL operations for quantitative measurements are controlled by a formal calibration program. Table 8-3 lists the lab's major analytical instrumentation. Calibrations may be periodic or operational. These are described in the lab's method SOPs. The Policy P-T-001, "Selection of Calibration Points", is applicable when the number of data points is not described in the method. At a minimum, these calibration procedures shall include:

- Instrument to be calibrated.
- Reference standards used for calibration.
- Calibration technique (e.g., linear, quadratic).
- Acceptable performance tolerances and corrective actions required if specifications are not met.
- Frequency of calibration.
- Calibration documentation requirements.

Whenever possible, recognized procedures such as those published by ASTM or the USEPA or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed, considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operation error on the quantities measured.

8.5.4.1 Physical Reference Standards

Physical reference standards associated with periodic calibrations include weights for calibrating balances and certified thermometers for calibrating working thermometers. Whenever possible, physical reference standards shall be calibrated by a body that can provide traceability to nationally or internationally

recognized standards. If these standards are not available, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration procedures and shall be stored separately from equipment used for analysis.

8.5.4.2 Chemical Reference Standards and Reagents

Chemical reference standards are generally associated with operational calibration. These standards include reference materials traceable to recognized standards suppliers. This may include vendor-certified materials traceable to national or international standard reference materials (e.g., NIST). This topic is also discussed in the Section on "Procurement of Supplies and Services" (see Section 5).

All chemical reference standards maintained in the laboratory for use in calibrations (or as QC spiking solutions) and reagents prepared in the laboratory shall be labeled or referenced to appropriate documentation (hard copy or electronic) with the following information at a minimum:

- A unique identification including concentration (solutions containing several analytes can be identified such that the solution constituents and concentrations can be referenced to a logbook).
- Medium prepared in (if non-aqueous).
- Preparation date.
- Expiration date.
- Initials of preparer.

Vials containing standard solutions that are not large enough to accommodate labels listing the above information may be referenced to a laboratory logbook/notebook entry or standards software. The expiration date of the working standard and reagent must not exceed the expiration date of the original material. These records should provide sufficient detail to allow one to reproduce the standard or reagent.

Records for all purchased standards and reagents shall include the date of receipt, the date opened, and, where applicable, the expiration date.

8.5.4.3 Standard Verification

When possible, reference standards are purchased from a STL preapproved vendor. Standards are verified by quantitation against a second known standard before reporting data. The standard for verification must meet the laboratory's criteria for the independent/second source initial calibration verification (ICV). Therefore, the verification of a new initial calibration standard with a second source ICV meets this verification requirement. Realizing that some "bad acting" analytes may not meet these criteria, alternative criteria must be approved by the

QAM before use. Standard spiking solutions and surrogates shall be verified by analyzing an LCS with the new standards and verifying against historical control limits. Special standards that are obtained from another source must also be independently verified at the lab. Verification by the laboratory of a reference standard from neat materials is also necessary.

An independent or second source standard is used to verify initial calibrations. An independent/second source standard is defined as a standard composed of the same target constituents as, but from a different source than, those used in the standards for the initial calibration. An independent standard may be a laboratory-prepared or a certified independent standard solution. Independence of reference material can be achieved by: (1) purchasing reference materials from two separate vendors, (2) using a different lot from the same vendor that is certified by the vendor as an independent standard or (3) having two separate individuals prepare the calibration and verification standard solutions if independent sources are not available.

To extend the use of an expired standard**,** reverification is necessary, provided that the new analysis produces acceptable data. Note that not all programs may allow this. The reverification of an expired standard is performed against a current, independent standard reference material by analyzing within a valid calibration. The analysis of the expired standard must meet the QC criteria.

Stock and working standards and reagents are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard and reagent solutions. Standards and reagents are always stored separately from samples.

8.5.4.4 Periodic Calibration

Periodic calibration is performed at prescribed intervals. In general, equipment that can be calibrated periodically are distinct, singular purpose units and are relatively stable in performance. These include balances, micropipettors, counters, thermometers, refrigerators, freezers, and ovens. Equipment employed at STL requiring periodic calibration are listed along with their respective calibration requirements in Tables 8-13. NELAC requires mechanical volumetric dispensing devices (except Class A glassware) to be checked for accuracy on at least a quarterly basis if in use. Refer to SOP KNOX-QA-0012 for the calibration of mechanical volumetric dispensing devices.

8.5.4.5 Operational and Continuing Calibration

Operational calibration is routinely performed as part of instrument usage, such as the development of a standard calibration curve. The accuracy of

initial calibrations is verified prior to sample analysis through the use of an independent standard.

Detailed requirements for operational and continuing calibration are contained in method-specific SOPs.

When an initial calibration is not performed on the day of analysis, the initial calibration must be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch.

- A continuing instrument calibration verification must be analyzed at the beginning and end of each analytical batch, as defined by the laboratory SOPs. The concentrations of the calibration verification shall be varied within the established calibration range on an annual basis for each NELAC accredited analytical method. If an internal standard is used for organic analyses, only one continuing instrument calibration verification must be analyzed per analytical batch, unless otherwise specified by the method.

- Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, and calibration curve or response factor.

- If the continuing instrument calibration verification results obtained are outside the established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications or a new initial instrument calibration must be performed. If the laboratory has not demonstrated acceptable performance, sample analyses must not occur until a new initial calibration curve is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

    - When the acceptance criteria for the continuing calibration verification is exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

    - When the acceptance criteria for the continuing calibration verification is exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit. Otherwise the samples affected by

the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

8.5.4.6 Calibration Failure

Equipment or instruments that fail calibration or become inoperable during use shall be tagged as necessary to prevent inadvertent use. Such instruments or equipment shall be repaired and successfully recalibrated before reuse. Following recalibration or verification, back to control will be documented in the injection/run log and/or maintenance logbook through the routine identification of the required calibration runs specified by the standard operating procedure.

8.5.4.7 Calibration Records

Calibration shall be documented for each piece of equipment subject to calibration. All calibration records (periodic and operational) directly affect data and may not be limited to one project. These records shall be stored in either the quality records or the associated project files. Project files that include sample data shall either include the calibration records or include reference to them.

8.6 Quality Assessment

The effectiveness of the QA practices is measured by the quality of data generated by the laboratory. Procedures are in place to detect, prevent, and correct quality problems and to ensure quality improvement. Items and processes that do not meet established requirements must be investigated to determine their cause. Improvements must be implemented in the operations that will prevent a recurrence of these quality problems and provide overall quality performance. All phases of laboratory work should be designed with the objective of preventing problems and improving quality on a continuous basis.

8.6.1 Data Quality Assessment

Data quality is judged in terms of precision, accuracy, representativeness, completeness and comparability. The areas of representativeness, comparability, and completeness for an overall project, inclusive of sampling issues, may be beyond the control of the laboratory. The elements over which the laboratory has direct control are precision, accuracy, and completeness relative to analytical testing results.

Precision and accuracy assessments are made as part of the evaluation of laboratory QC data generated during sample preparation and analysis. The QC samples employed at STL Knoxville as part of routine sample analysis are summarized in Section 8.4 of this document. Table 8-5 shows the precision and accuracy measurements employed. Analytical method SOPs and Policy QA-003 include information on requirements for the type of QC samples, frequencies, and acceptance criteria. Additionally, the SOPs and

policy describe the appropriate actions to be taken when a QC sample result does not meet acceptance criteria.

8.6.2 Statistical Evaluation of Data

In-house limits for QC data must be evaluated at least annually and compared to the appropriate limits published in the methods for applicable matrices. Method limits may be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points is recommended to establish the in-house QC limits. Calculated results of the QC (e.g. LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the nonconformance system, to review trends.

Precision and accuracy measurements employed by STL Knoxville are shown in Table 8-5.

8.7 Data Recording Procedures

To ensure data integrity, all documentation of data, and records generated or used during the process of data generation, must be performed in compliance with SOP KNOX-QA-0013, "Instrument Records and Logbooks".

8.8 Data Reduction and Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100 percent review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed. This procedure is outlined in Figure 8.8-1.

8.8.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, sample amounts, etc.

- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.).
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers.
- The appropriate SOPs have been followed and are identified in the project records.
- Proper documentation procedures have been followed.
- All nonconformances have been documented.
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets.
- Input of raw data for computer processing.
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to produce good chromatography, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy S-Q-004, “Acceptable Manual Integration Practices”.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation and processed, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants/RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The initial verification of the data reduction (level 1 review) by the analyst is documented on a data review checklist, which is signed and dated by the analyst. Data review requirements are described in Section 5.3.6 of the QMP.

8.8.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through level 1 review is performed by an experienced peer, supervisor, or designee. This check (level 2 review) is performed to ensure that level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. This review includes an evaluation of all items required in the raw data package. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

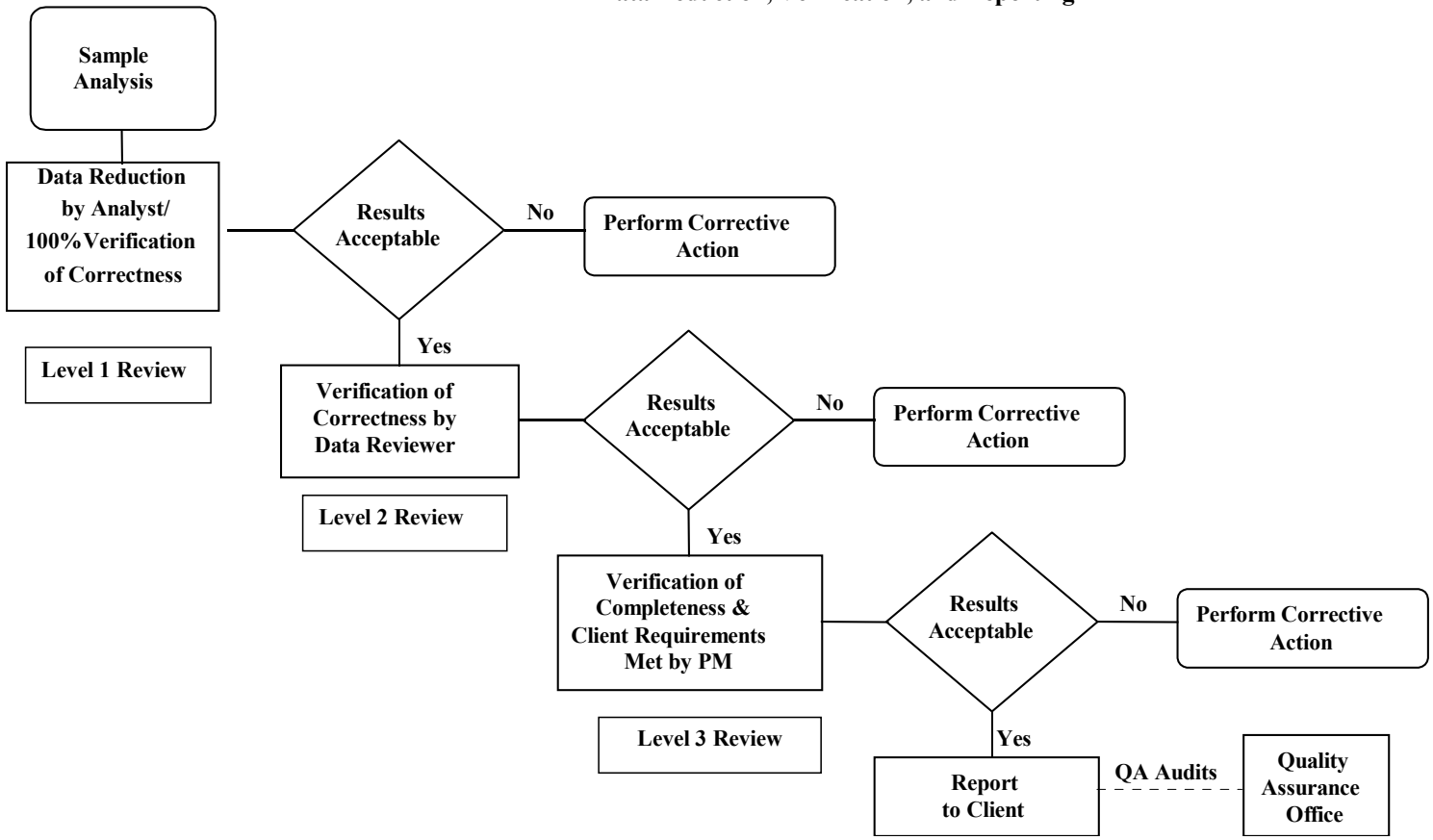
- Adherence of the procedure used to the requested analytical method SOP.
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly).
- Correct identification and quantitation of constituents with appropriate qualifiers.
- Numerical correctness of calculations and formulas (checked randomly).
- Acceptability of QC data.
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.).
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

The level 2 review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and specific instructions given on the project QAS or equivalent summary form.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo.
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct.
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the level 2 review may vary according to the analytical method, but this review of the data must be documented by signing the data review checklist. Data review requirements are described in Section 5.3.6 of the QMP.

FIGURE 8.8-1
Data Reduction, Verification, and Reporting



8.8.3 Completeness Verification

A third-level review is performed by the PM. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG).
- Every parameter or target compound requested is reported with either a value or reporting limit.
- The correct units and correct number of significant figures are utilized .
- All nonconformances and receipt variances with the resolution, including holding time violations and data evaluation statements that impact the data quality, are accompanied by clearly expressed comments from the laboratory.
- The final report is legible, contains all the supporting documentation required by the project, and is in either the standard STL format or in the client-required format.
- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)
- A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

8.9 Data Reporting

8.9.1 Data Reports

STL Knoxville is capable of developing a variety of data deliverable reports. Standard reports will contain:

- Cover Letter/Narrative - Information on sample types, tests performed, any problems encountered, and general comments are provided.
- Analytical Data - Results are reported by sample or by test with the appropriate significant figures and reporting limits, and with the proper adjustment for dilution, if appropriate. Pertinent information including dates sampled, received, prepared, extracted, and analyzed are provided.
- Laboratory Performance QC Information - The results of LCSs and method blanks analyzed with the project are listed. Any data or QC anomalies are discussed in the narrative.
- Matrix-Specific QC Information - Results of any client sample duplicates and MS/MSDs analyzed with the samples as batch QC are reported, as requested by the client. Other project-specific QC requested by the client

are also reported. The results include supporting information such as amount spiked, percent recovery, or percent difference/RPD.

- Methodology - Reference for analytical methodology used is cited.
- Other Deliverables - Other deliverables available include disk deliverables, electronic data transfer, sample raw data packages, complete deliverable packages, and custom report formats.

8.9.2 Final Report Details

STL Knoxville will provide paginated reports or a uniquely defined, identifiable certificate/report (i.e., electronic file, CD). The report will include:

a) Report title, name, address and phone number of the laboratory.
b) Name and address of client/project name/client identification number.
c) Description (lab ID of sample).
d) Dates and time of sample collections (if known) and receipt, and date of preparation and analysis. If the required holding time is 48 hours or less, time of sample preparation and analysis.
e) Method identifiers traceable to all procedures used.
f) Reporting limit.
g) Test result with appropriate units and how reported (wet weight/dry weight). Any results outside of quantitation limits will be identified. When required, a statement of the estimated uncertainty of the test result should be added.
h) If appropriate, description of any QC failures or deviations from SOPs.
i) Signature and title of the individual responsible for the report. Electronic signature is acceptable.
j) Date of issue.
k) All subcontract work is clearly identified, and name and address of subcontractor laboratory noted.
l) Where relevant, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory.
m) Where relevant, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory.
n) NELAC statement, as described in SOP KNOX-AD-0004, current revision.

After the final report, any correction, addition, or deletion must clearly identify its purpose and meet the above reporting requirements as appropriate.

8.9.3 Verbal Results

STL Knoxville, as a policy, discourages the release of data verbally or without

full data review. If however, the client requests analytical results to be communicated verbally or by facsimile prior to final review, they must be clearly identified as "Preliminary" results. The client must understand that the data have not undergone the required levels of review and may potentially change.

8.9.4 Reporting Analytical Results

Sample results are reported according to analytical method SOPs or client specifications. Normally, the laboratory uses the STL Knoxville Reporting Limit (RL) at which any analyte of interest detected at or above that level is reported as a positive value and any analyte of interest not detectable or detected below that level is reported as "not detected" at the RL. The laboratory will report results from within the calibration range; however, any reported results outside of the calibration range will be flagged in the final report.

If a QC measurement is out of control and the data is to be reported, data qualifiers are reported with samples associated with failed QC measurements.

The laboratory must certify that the test results meet all NELAC requirements or provide reasons and/or justification if they do not.

In some cases a contract, QAPP, or documented client request may require the laboratory to report sample results in a specified manner. Some examples are given below:

- The laboratory may be requested to report all analytes of interest that are less than the laboratory's RL but are greater than the MDL. This data will be flagged with an appropriate qualifier or noted in the report case narrative. (See precautions in "Reporting Limits for STL Laboratories Using QuantIMS", Policy Number S-T-002).
- The laboratory may be requested to report any tentatively identified compounds (TICs). These data will be flagged with an appropriate qualifier.
- The laboratory may be requested to report sample results using an RL that is higher than the standard STL Knoxville RL. In this case, only the analytes of interest found at or above the higher RL would be reported as positive values. In this case, the laboratory will report to the project-specific RL (PSRL) rather than the standard RL. All analytes of interest not detected or detectable below that level would be reported as "not detected" at the PSRL.

  In any of these situations, the laboratory must include documentation in the project file that supports the reporting procedure employed. It is the responsibility of the laboratory to provide a reporting system that assures that any problems associated with an analysis are properly documented on a

nonconformance memo, communicated to the appropriate STL Knoxville staff, and addressed appropriately in the data report.

8.9.5 Reissued Deliverables

If, after issuance of a report, STL Knoxville observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified. After issuance of the report, the original laboratory report remains unchanged. Any material amendments to a report after issue must be in the form of a further document or further data transfer and must be identified as a revised or supplemental report. For further discussion, see SOP KNOX-AD-0004, "Data Reporting".

8.9.6 Client Confidentiality

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence, unless such information is generally available to the public or is in the public domain. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of our clients, and are not released to a third party without written consent from the client. Data confidentiality is also discussed Section 3.6.

8.10 Data Validation

Data validation for STL refers to data reviews conducted in accordance with the USEPA CLP "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses", or modifications thereof, for non-CLP type analyses.

This form of data validation provides an impartial evaluation of the laboratory's results. Data validation may be requested by the client for a percentage of data and is performed by a third party, one that was not involved with the sample analysis. Qualifiers are assigned to data, when required during data validation, according to the requirements of the data validation protocol being used.

8.11 Preventive Maintenance and Service

Facilities, instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within STL, preventive maintenance, coupled with vendor service agreements, is an organized program of actions taken to maintain facilities and equipment in control.

8.11.1 Analytical Instrumentation and Equipment

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

Each instrument or piece of equipment shall be uniquely identified. The laboratory maintains the following:

- Instrument/equipment inventory list with serial number of the item or equipment
- Instrument/equipment major spare parts inventory
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit.

The records of routine maintenance and non-routine maintenance shall include at a minimum:

- Name of the item or equipment
- Details of maintenance performed
- Dates and results of recalibrations/reverifications indicating return to control (this may be done in the analytical run log)
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative.

Any item or equipment that does not perform to specifications or is found to be defective shall be taken out of service (and tagged as necessary to prevent inadvertent use) until it has been repaired and shown by calibration/verification to perform satisfactorily.

8.11.2 Frequency of Equipment Maintenance

The frequency of maintenance must consider manufacturer's recommendations and previous experience. Frequency of preventive maintenance along with the recommended preventive maintenance schedules are given in STL Knoxville SOP KNOX-QA-0003, “Preventive Maintenance” for analytical instrumentation and equipment. Frequency of maintenance for the facility systems is documented in the STL Corporate Safety Manual.

8.11.3 Facilities

Another important aspect of the laboratory operation is the existence and maintenance of adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme temperatures, waste disposal, and a source of

stable power. The facility floor plan is provided in Figure 8.11-1.

The maintenance and use of these facilities and proper operations are described in the STL Corporate Safety Manual. The Laboratory Director has responsibility for ensuring a properly maintained facility. The Laboratory Director also has the responsibility for ensuring that facilities are available to store samples properly without contamination, work areas are equipped with adequate bench, hood and operational space, and that procedures are in place to ensure the areas are free from chemical and radiological contamination that may affect analytical results.

8.11.4 Facility Security

The laboratory building is a limited access, secure facility. To ensure that only authorized personnel are able to enter the building from an entrance that is not monitored, entry into each building is limited in one or more of the following ways at a minimum:

- The use of key pads or electronic locks activated by swipe or magnetic cards which are issued only to authorized personnel
- Locking doors and issuing keys only to authorized personnel
- Alarm systems to detect unauthorized entrance

During business hours, entry is possible only through the main entrance. This entrance is monitored at all times, usually by a receptionist. All guests are required to sign in by using a visitor logbook.

8.12 Requirements for Ancillary Equipment and Materials

8.12.1 Water

High purity water (e.g., ASTM reagent grade or equivalent water) will be used in all metals, wet chemistry, and organic analyses. Demonstration of contaminant-free water is shown through the routine analysis for the analyte of interest of method blanks consisting of the reagent water used. This water is obtained by the use of either a commercial ion-exchange deionizing, distillation, or reverse osmosis unit plus an appropriate polishing unit. The resulting water has a maximum conductivity of 1.0 umho-cm at 25°C or a minimum resistivity of 1.0 Mohm at 25°C. Conductivity or resistivity will be monitored and documented daily or on each day that water is dispensed for analytical use.

For volatile analyses, the water may be further purified by purging with an inert gas before use to remove potential traces of organic solvents.

8.12.2 Compressed Air and Gases

Ultra high-purity compressed gases from pre-approved vendors or in-house gas generators will be used when required for instrumentation. These air and gases must meet the requirements and specifications of the analytical methods performed. In-line filters will be used when appropriate to minimize contamination and moisture from the gases.

8.12.3 Glassware Preparation

Glassware preparation procedures implemented at operating units are designed to ensure that contaminants are not introduced during sample analysis. Procedures describing glassware preparation are detailed in SOP KNOX-QA-0002.

8.12.4 Chemical Storage

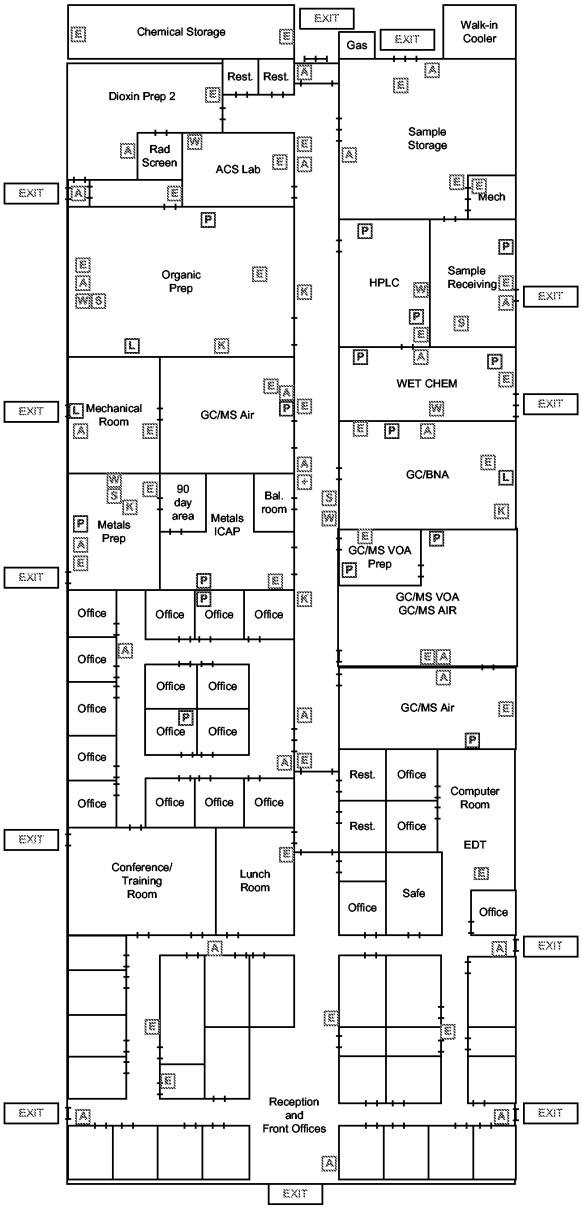
Storage of chemicals shall be conducted in a manner to minimize the potential for fire or release of hazardous material resulting from an unplanned chemical reaction. Flammable solvents shall be stored in appropriate cabinets meeting all necessary codes. All chemicals are stored according to chemical compatibility. Further details regarding chemical storage are provided in the STL Corporate Safety Manual.

8.12.5 Waste Management

The goal of STL's policy for waste management is to ensure that laboratory wastes are disposed of safely and in a manner consistent with applicable federal, state and local regulations. The waste disposal program is designed to assure that minimal harm to people and the environment shall result from the disposal of laboratory chemicals. This goal is accomplished by requiring that the laboratory comply with the procedures presented in the STL Corporate Safety Manual.

Figure 8.11 -1 Floor Plan

STL Knoxville Laboratory
Main Building



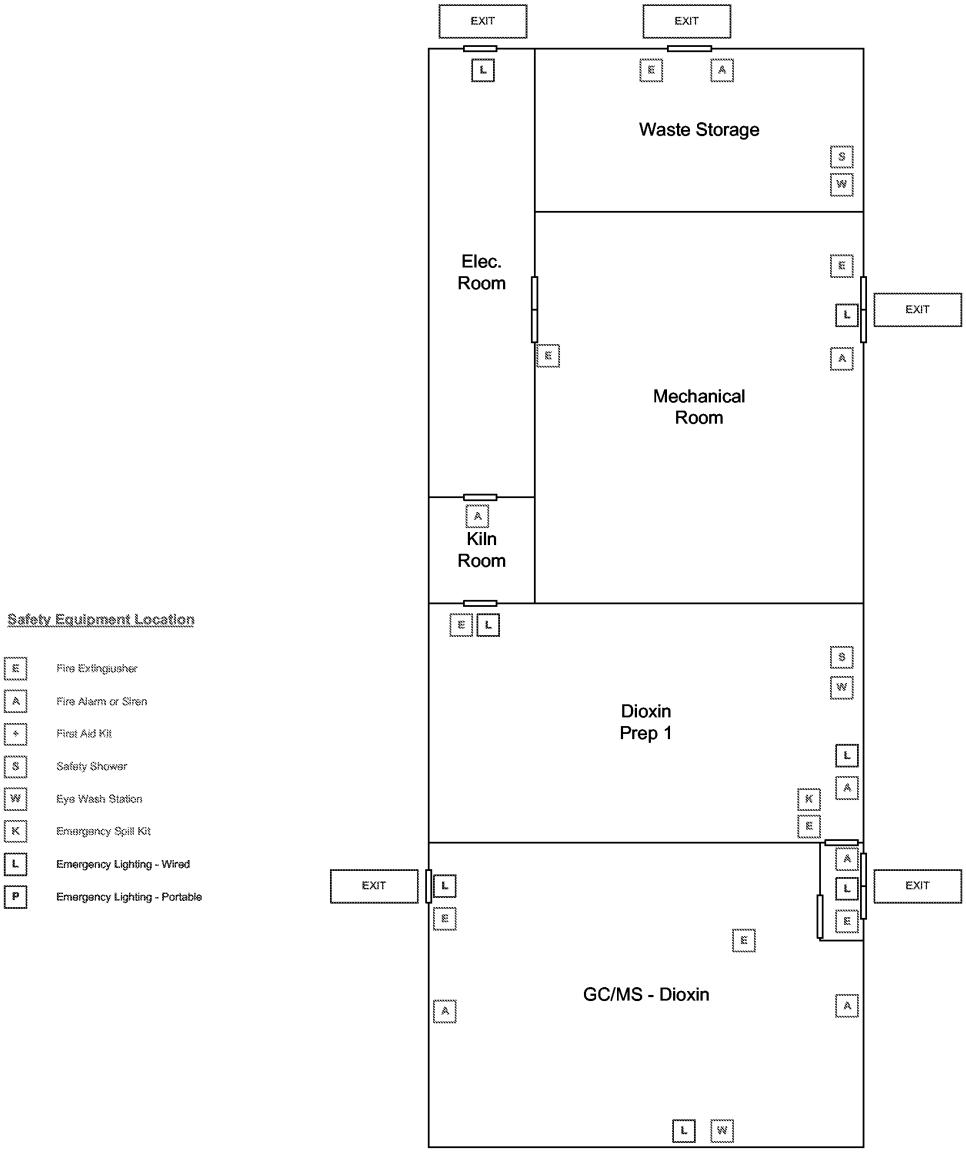
Safety Equipment Location

- E Fire Extingiusher
- A Fire Alarm or Siren
- + First Aid Kit
- S Safety Shower
- W Eye Wash Station
- K Emergency Spill Kit
- L Emergency Lighting - Wired
- P Emergency Lighting - Portable

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030905.vsd Page 1

Figure 8.11 -1 Floor Plan, continued

STL Knoxville Laboratory
Building 2 - Specialty Organics Group Lab



9.0 Quality Assessment and Response

9.1 Nonconformances

A nonconformance is an unplanned deviation from an established protocol or plan and in some cases may be exceptionally permitted departures from the documented policies and procedures or from standard specifications. The deviation may be the result of STL's actions as a systematic error, then termed a deficiency. A single isolated event or event beyond the control of STL is termed an anomaly.

Nonconformances can be identified on the basis of internal or external systems or performance audits, sample processing, routine calibration and monitoring of analytical and support equipment, or QC sample analyses. The Technical Director, Operations Manager, Project Manager, QA Manager, Team Leader, and Analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected, the issue is immediately brought to the attention of QA.

9.1.1 Nonconformance Memo (NCM)

Nonconformances, deficiencies and anomalies, are documented via an electronic process or on a paper form that meets NCM requirements as approved by QA. An allowed exception is log-in conformance problems, which are documented on a Condition Upon Receipt Form (see Section 8.5). A detailed description of the procedure and responsibilities associated with nonconformance documentation, communication, and resolution is described in SOP KNOX-QA-0008.

The Clouseau NCM program, available on the local-area network throughout the laboratory, is the main vehicle for documenting and communicating NCMs. The program allows anyone in the laboratory to document a nonconformance, explain the cause of the problem, and link to the LIMS system to identify the samples and clients involved. The program uses the local e-mail to automatically notify the person's supervisor, the Project Managers associated with the samples, and the QA department. The program is used to document approval and completion of the immediate corrective actions for the samples involved, and can be used to document long-term corrective actions. It provides a place to document resolution of problems with the clients, and it provides routines to query the associated database to examine trends and prepare management reports. An electronic record of the nonconformance shall be kept in the Clouseau Program database. Nonconformances affecting client sample results are addressed in the project narrative.

9.2 Client Complaints

Client inquiries and complaints are generally received through the PM or Customer Services Manager. Typically, the PM or CSM communicates with the client to determine the details of the inquiries, including technical data problems, deliverable issues, turn-around-time problems, etc. Technical and deliverable issues are coordinated by the PM and usually involve input from operations, QA, and management staff. A formal written response to the client may be coordinated by the PM, CSM, QAM, Technical Director or Laboratory Director. Details of the types and levels of complaints and required documentation are provided in Section 4.8 of the QMP. Client complaints are recorded as a type of NCM in the Clouseau database, which are summarized in the monthly QA Reports to Management (see Section 9.7 for more about the monthly QA reports).

9.3 Corrective Actions

Corrective actions are measures taken to rectify conditions adverse to quality and, where possible, to prevent their recurrence. Investigations of potential problems and corrective actions should be timely, determine the root cause, and evaluate any propagation of the error or problem. Whenever a systematic error is discovered that affects the accuracy or defensibility of results reported to STL's clients, Corporate QA notification and written client notification will be part of the corrective action.

Corrective actions should be implemented with an understanding of the technology and work activities associated with the quality element, and may include appropriate training of STL associates, and should be monitored for progress and success. The occurrence of the problem, the corrective action employed, and results of the corrective action must be documented properly. On-the-spot actions are used to correct minor problems, such as recalibration, retuning or a minor repair (e.g., replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used by an analyst. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Similarly, routine instrument maintenance, malfunctions, and power failures are also documented in the appropriate instrument maintenance logbooks. These events do not require a formal NCM process, provided reported analytical results are not affected. Corrective actions specific to quality controls for analytical methods are discussed in the operational-specific SOPs.

9.3.1 Monitoring Corrective Actions

All formal corrective action documentation is maintained by the QA department, either in the Clouseau database or in paper files. The QA department reviews all corrective actions and may implement a spot assessment audit. The purpose of spot assessments is not only to monitor the implementation of the corrective action, but also to assess laboratory adherence to procedures, policies, etc.

9.4 Preventive Action

Preventive action is defined as noting and correcting a problem before it happens, because of a weakness in a system, method or procedure. When potential problems are identified, preventive action is initiated to effectively address the problem to eliminate or reduce the risk identified. STL Knoxville implements preventive action as a part of its quality system. For example, preventive action is inherent in the Preventive Maintenance SOP (KNOX-QA-0003) which helps ensure that instruments remain operational and in the Project Set-up SOP (KNOX-AD-0005) which helps ensure that laboratory capabilities meet client data quality objectives. Section 7, Contract Review and Project Planning, and specifically Section 7.6, Contingency Planning, are also part of the preventive action process. Documentation of preventive action may also be documented as part of the corrective action process.

9.5 Internal Audits

Internal audits are performed to assess the degree of adherence to established policies, procedures and standards. These assessments are conducted by STL personnel who are independent of the area being evaluated. Audits can identify areas for improvement with regard to compliance with policies, procedures and standards. Audits also provide a means for correction prior to system failure.

Audits and assessments are generally conducted through the use of checklists and relevant reference documents. The findings of all audits and assessments are documented as is the laboratory response and any corrective actions. Follow-up checks are performed and the status of implementation of corrective actions is documented for all categories of audits and assessments. This cycle continues until all findings are closed.

When the audit findings cast doubt on the validity of the laboratory test results, the laboratory shall take timely and appropriate corrective action. Upon completing the review of the audit finding, if it is determined that a data recall is needed due to a systematic error, the laboratory shall follow the requirements of STL SOP S-Q-005, Data Recall, current revision. The client must be notified in writing no later than 30 days from the completion of the review which determined invalid data were reported.

9.5.1 Audit Types and Frequency

The following types of audits are performed at STL Knoxville:

Figure 9.5-1 Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data	QA Department or designee	**Data Report Review:** As necessary to ensure an effective secondary review process
		Analyst Data Audits: 100% of all analysts annually
		Electronic Data Audits: 100% of all organic instruments
Special	QA Department or Designee	As Needed

Note that some programs, e.g., AFCEE, require project data audits conducted by QA at a defined frequency.

9.5.2 Systems Audits

Facility systems audits are comprehensive technical and systems evaluations covering each operational and support area at least once per year. The audit report is issued by the auditor within 30 calendar days of the audit. The audit report is addressed to the Laboratory Director and copied to the STL Corporate Director of Quality Assurance, the Regional Manager and Laboratory QA Manager. A written audit response is required within 30 calendar days of the date of the audit report. The audit response from the lab must follow the format of the original audit report. The audit response is directed to all individuals copied on the audit report. When a corrective action is not completed by the time of the audit response, the target date for the corrective action is stated. Evidence of corrective action is submitted to the QA department either at the time of the audit response or in the agreed upon time frame. Refer to SOP S-Q-002 for details regarding internal systems audits.

9.5.3 Data Audits

Data audits assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria. Data Audits may be accomplished through electronic instrument data audits, analyst data authenticity audits or through final project report reviews.

Records of the data audits shall be kept, and the frequency of data audits shall be included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of differentiating between systematic and isolated

errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client. The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for process evaluation and change.

Where specific clients and regulatory programs require more frequent data auditing, the individual facility must meet the data auditing frequency for that program.

9.5.3.1 Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from the operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. The laboratory will report the percentage of analysts reviewed (for the year) in their monthly QA report and should average about 8% per month.

9.5.3.2 Electronic Data Audits

Electronic data audits shall be performed on 100% of all organic instruments by the QA department or a designate independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The laboratory will report the percentage of instruments reviewed (for the year) in their monthly QA report and should average about 8% of instruments per month. Electronic data audits must include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to QMP Section 5.3.6.

9.5.4 Spot Assessments

Spot assessments, equivalent to special audits as described in the STL QMP, are conducted on as needed basis, and may be conducted as a follow up to specific issues such as client complaints, validator concerns, corrective actions, control chart or NCM trends, proficiency testing results, data audits, or external audit issues. Spot assessments are focused on a specific issue. The frequency, report format, distribution, and timeframes are tailored to address the nature of the issue.

9.5.5 Proficiency Testing

Proficiency testing samples (PTs) are analyzed to verify the ability of the laboratory to correctly identify and quantitate compounds in PT samples. PT samples may be supplied internally or externally as single-blind or double-blind samples. They can be used to assess if a deficiency has been corrected, to document the proficiency of the analyst to perform the analysis, or to assess the overall performance of an analytical method.

PT samples are handled and tested in the same manner as environmental samples. It is not acceptable to run multiple replicates that would not otherwise be performed; it is not acceptable to average multiple results; and PT results cannot be shared among labs in advance of the close of the study. PT test sample data is archived using the same requirements as for project and raw data record retention.

STL Knoxville participates in a number of PT studies, as shown in Table 9-1. The NELAC PT program involves a minimum of two PT rounds each year for each NELAC field of testing for which the lab is maintaining certification.

STL Knoxville also participates in a double-blind performance evaluation annually. An external vendor is contracted to submit double-blind samples to the lab. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor. The PT contractor provides a detailed report to the Corporate Director of QA and to STL Knoxville.

9.6 External Audits

STL Knoxville is regularly audited by clients and external regulatory authorities. STL Knoxville is available for these audits, and makes every effort to provide the auditors with the personnel, documentation and assistance they require. STL Knoxville recommends that all audits be scheduled with the QA department so that all necessary personnel are available on the day of the audit. As required, deficiencies reported to the laboratory must be responded to within the time frame specified in the audit report. It is the responsibility of the QA Manager to coordinate the response to the audit report. The development and implementation of the corrective actions is the responsibility of the operations management of the affected areas. All responses must be approved by the Laboratory Director or Operations Manager prior to submitting the final response. It is the responsibility of the QA Manager to verify implementation of the corrective actions and inform the responsible manager of the closure of all deficiencies from the audit.

9.7 Management Reviews

9.7.1 Quality Reports to Management

A monthly QA report is prepared by the QA Manager and forwarded to the Laboratory Director, the Regional General Manager, and the Corporate QA Manager. The reports include metrics (i.e., frequency and number of revised reports, frequency and number of client complaints) to assess the effectiveness of the Quality System. The contents of the monthly report include:

- Audits
  Internal System Audits
  External System Audits
  Analyst & Instrument Data Authenticity Audits
- Revised Reports / Client Complaints
  Frequency of revised reports
  Total number of client complaints, reason, and resolution
  Client Compliments
- Certification/Parameter Changes
- Proficiency Testing
  Score for each PT as a percentage of maximum score
  Note repeat failures and/or significant problems
- SOP Status
  # of active SOPs, # needing to be written and # reviewed/revised in the past 24 months
- Miscellaneous QA and Operational Issues
  Narrative outlining improvements, regulatory compliance issues, general concerns
- Metrics Spreadsheet
  Summarize metrics in the template provided by the Corporate Quality Director.

This information is compiled by the Corporate QA Manager together with similar information from and about other STL laboratories, which is then presented in a report to the STL Chief Operating Officer.

9.7.2 Management Review of QA Systems

The Laboratory Director will conduct an annual management quality system review to assess the adequacy of the quality systems in the laboratory and plan any changes in laboratory organization, policies, practices, certifications, accreditations in order to achieve operational efficiencies, meet regulatory requirements and client expectations. The evaluation shall consider:

- The suitability of policies and procedures
- Reports from managerial and supervisory personnel

- The outcome of recent internal audits
- Corrective and preventative actions
- Assessments by external bodies
- The results of interlaboratory comparisons and proficiency tests
- Status of QA documents
- Changes in the volume and type of work and the effects on QA systems
- Client feedback
- Complaints
- Quality control activities
- Resources and staff training

Acronyms and Initialisms

A2LA	American Association for Laboratory Accreditation
AA	Atomic Absorption
ANSI	American National Standards Institute
AR/COC	Analysis Request/Chain-of-Custody
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BLK	Blank
BOD	Biochemical Oxygen Demand
CCC	Calibration Check Compound
CEO	Chief Executive Officer
CF	Calibration Factor
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
CLP	Contract Laboratory Program
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act (Superfund)
COC	Chain-of-Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CSM	Customer Service Manager
CSRM	Certified Standard Reference Material
CUR	Condition Upon Receipt
CV	Coefficient of Variation
CVAA	Cold Vapor Atomic Absorption (Spectroscopy)
DFTPP	Decafluorotriphenylphosphine
DOC	Dissolved Organic Carbon
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective

Acronyms and Initialisms (continued)

EH&S	Environmental Health and Safety
EPA	(U. S.) Environmental Protection Agency
FLAA	Flame Atomic Absorption (Spectroscopy)
FTIR	Fourier Transform Infrared (Spectrometry)
GC	Gas Chromatograph(y)
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption (Spectroscopy)
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
ICAP	Inductively Coupled Argon Plasma (Spectroscopy)
ICAP/MS	Inductively Coupled Argon Plasma/Mass Spectrometry
ICS	Interference Check Sample
IDL	Instrument Detection Limit
IR	Infrared (Spectroscopy)
IS	Information Systems
IS	Internal Standard
ISO	International Organization for Standardization
IT	Information Technology
KRI	Key Result Indicator
LAN	Local Area Network
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LQM	Laboratory Quality Manual
LRGC	Low Resolution Gas Chromatography
LRMS	Low Resolution Mass Spectrometry
LWL	Lower Warning Limit
MBAS	Methylene Blue Active Substance

Acronyms and Initialisms (continued)

MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NCM	Nonconformance Memo
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards Technology
NMOC	Non-Methane Organic Compounds
NPDES	National Pollutant Discharge Elimination System
NRC	Nuclear Regulatory Commission
NRM	National Reference Material
PAH	Polynuclear Aromatic Hydrocarbons (or PNA)
PC	Personal Computer
PCB	Polychlorinated Biphenyls
PDS	Post Digestion Spike
PEM	Performance Evaluation Mixture
PM	Project Manager
PQL	Practical Quantitation Limit
PSRL	Project-Specific Reporting Limit
PT	Proficiency Testing
PUF	Polyurethane Foam
QA	Quality Assurance
QMP	Quality Management Plan
QAPP	Quality Assurance Project Plan or Quality Assurance Program Plan
QAS	Quality Assurance Summary
QC	Quality Control
QS	Quality System

Acronyms and Initialisms (continued)

QuantIMS	STL Knoxville Laboratory Information Management System
QRI	Quality-Related Item
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFP	Request for Proposal
RFQ	Request for Quote
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compounds
SPLP	Synthetic Precipitation Leaching Procedure
SRL	Standard Reporting Limit
SRM	Standard Reference Material
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TOX	Total Organic Halides
UCL	Upper Control Limit
UPS	Uninterruptable Power Supply
USEPA	United States Environmental Protection Agency
UWL	Upper Warning Limit
VOA	Volatile Organic Analysis
VOST	Volatile Organic Sampling Train
WAN	Wide Area Network
WS	Water Supply
WP	Water Pollution

Glossary

acceptance limits
Data quality limits specified for analytical method performance.

accuracy
Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

aliquot, aliquant
A measured portion of a sample taken for analysis.

analytical spike
A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. (Also see matrix spike.)

anomaly
See nonconformance.

areas needing improvement
Represent isolated instances of noncompliance or issues that are judged to have a less immediate impact on data quality. Laboratory management must correct the situation or otherwise ensure that the condition does not recur. This term replaces the previous term used “Observations.”

arithmetic mean
The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

where: $\bar{x}$ = the mean
x_i = the i^{th} data value
n = number of data values

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

Glossary (continued)

assessment
The evaluation process used to measure the performance or effectiveness of a system and its elements. Assessment is used as an all-inclusive term to denote any of the following: performance, systems, data and compliance audits, management systems reviews, peer reviews, inspections, or spot assessments.

associate
Employee.

audit
A planned and documented investigative evaluation of an item or process to determine its adequacy and effectiveness as well as compliance with established procedures, instructions, drawings, quality management plans, and other applicable documents.

benchmarking
A step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices.

bias
A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.

blind performance evaluation sample
A sample either submitted to the laboratory or prepared in the laboratory whereby the concentrations of parameters of concern are known by the preparer and not by the laboratory.

calibration
Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels at which actual measurements are to be made. Calibration is also the act of making a scheduled comparison of instrument performance against national standards for instruments which measure physical parameters such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.
calibration curve

Glossary (continued)

The graphical relationship between the known values for a series of calibration standards and instrument responses.

calibration factor (CF)
The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations.

$$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$

calibration standard
A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to standard reference materials (provided by NIST, or other recognized standards agencies) or a primary standard.

certified reference material
A reference material accompanied by a certificate issued by an organization certifying the contents and concentration(s) of the material. (See also standard reference material.)

chain-of-custody (COC)
A system of documentation demonstrating the physical custody and traceability of samples.

check standard analyses
A standard (often a midpoint standard) analyzed at a frequency specified in the method or in a SOP to verify the continuing calibration of the standard curve.

client
Any individual or organization for whom items or services are furnished or work is performed in response to defined requirements and expectations.

Glossary (continued)

client sample
The material or collection media submitted to the laboratory for analysis. Field QC samples are considered client samples but laboratory QC samples are not counted as client samples when counting samples for QC batches.

coefficient of variation (relative standard deviation)
A measure of precision (relative dispersion). It is equal to the standard deviation (s) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.

$$CV\ (RSD) = \left(\frac{s}{\bar{x}}\right) x\ 100$$

collocated samples
Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

comparability
Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

completeness
Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed. It is usually expressed as a percentage:

$$\%\ Completeness = \frac{V}{n} x\ 100$$

where: V = number of measurements judged valid
n = total number of measurements

composite
A sample composed of two or more increments.
control chart

Glossary (continued)

A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.

controlled document
A document for which the distribution is known. Updates of the document are sent to the original recipients, unless the copy distributed is an uncontrolled copy.

corrective action
A measure taken to rectify conditions adverse to quality and, where necessary, to preclude their recurrence.

correlation coefficient
The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.

customer
See client.

data quality objectives (DQO)
Data quality objectives (DQOs) are quantitative and qualitative measures of principal quality attributes, including precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). Typically, DQOs are identified during project scope and development of sampling and analysis plans. In this QA manual, however, we refer to only the analytical DQIs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. STL Knoxville incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in Section 8.4 and are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and

Glossary (continued)

performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs) and surrogate spikes.

data validation
See validation - data.

data verification
See verification - data.

deficiency
A practice that is not compliant with procedure or policy. See nonconformance or finding.

degrees of freedom
The number of independent deviations used in calculating an estimate of the standard deviation.

double blind performance evaluation sample
A sample that contains select parameters at defined levels. The levels are unknown to the laboratory. The laboratory is also unaware that the sample is a performance evaluation sample.

duplicate sample analyses
Different aliquots of the same sample are analyzed to evaluate the precision of an analysis.

error
The difference between an observed or measured value and its true value.

field blank
A blank that is prepared and handled in the field and analyzed in the same manner as its corresponding client samples.

field matrix spike
A sample created by spiking target analytes into a sample in the field at the point of sample acquisition.

Glossary (continued)

finding
Noncompliant practices or policies which have significant adverse impact on data quality, technical defensibility, or regulatory acceptance of data. Findings require immediate attention by the laboratory management and must be resolved to comply with STL Knoxville's quality documents and laboratory-established procedures.

geometric mean
The n^{th} root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much population data.

initial calibration
Analysis of a series of analytical standards at different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds prior to the analysis of samples.

inspection
Examination or measurement of an item or activity to verify conformance to specific requirements.

instrument detection limit (IDL)
IDL is a calculated estimate of instrument detectability defined by the USEPA Contract Laboratory Program (CLP).

Glossary (continued)

internal standard (IS)
A compound added to every standard, QC sample, client sample, or sample extract at a known concentration prior to analysis for the purpose of quantitation. For example, internal standards are used as the basis for quantitation of the target compounds by GC/MS.

Laboratory Quality Manual (LQM)
A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

linear regression
A statistical method for finding a straight line that best fits a set of two or more data points, thus providing a relationship between two or more variables.

matrix
The component or substrate which contains the analyte(s) of interest. Examples of matrices are water, soil or sediment, and air. Matrix is not synonymous with phase (liquid or solid).

matrix effect
An interference in the measurement of analyte(s) in a sample that is caused by materials in the sample. Matrix effects may cause elevated reporting limits or may prevent the acquisition of acceptable results.

matrix spike (MS)
An aliquot of a matrix fortified (spiked) with known quantities of specific compounds and subjected to an entire analytical procedure in order to indicate the appropriateness of the method for a particular matrix. The percent recovery for the respective compound(s) is then calculated.

matrix spike duplicate (MSD)
A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

may
Denotes permission but not a requirement.

mean

Glossary (continued)

See arithmetic mean.

measurement
The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

median
The middle value of a set of data when the data set is ranked in increasing or decreasing order.

method
An assemblage of techniques.

method blank (MB)
An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. Examples of method blanks are a volume of deionized or distilled laboratory water for water samples, a purified solid matrix for soil/sediment samples, or a generated zero air.

method detection limit (MDL)
The minimum concentration of an analyte that, in a given matrix and with a specific method, can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is operationally defined as:

$$MDL = st_{(n-1,\ \alpha= 0.99)}$$

where:

s = the standard deviation of a number of measurements of a blind or sample matrix containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1,\ \alpha= 0.99)}$ = the student's value for a one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99% confidence level and n-1 degrees of freedom.

modified method
A standard or reference method which has been changed to meet project or matrix requirements.

Glossary (continued)

must
Denotes a requirement is mandatory and has to be met.

notable practices
Laboratory practices that increase effectiveness and quality and represent improvements with respect to conventional laboratory operations.

nonconformance
An unplanned deviation from an established protocol or plan. The deviation may be the result of STL Knoxville's actions, then termed a deficiency. If the deviation is the result of events beyond the control of STL Knoxville, it is termed an anomaly.

operational calibration
Routinely performed as part of instrument usage, such as the development of a standard calibration curve. Operational calibration is generally performed for instrument systems.

outlier
A result excluded from the statistical calculations due to being deemed "suspicious" when applying the "Grubbs Test" (or equivalent).

parameter
A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

percent difference
When two independent measurements of the same characteristics are available, it is possible to use the percent difference instead of the coefficient of variation to measure precision.

$$\%D = \left| \frac{X_1 - X_2}{X_1} \right| x\ 100\%$$

where: $\%D$ = percent difference
X_1 = first value
X_2 = second value

Glossary (continued)

percent recovery
A measure of accuracy determined from the comparison of a reported spike value to its true spike concentration.

$$\%R = \frac{observed\ conc. - sample\ conc.}{true\ spike\ conc.} \times 100\%$$

performance audit
See performance evaluation.

performance testing (PT)
A type of audit in which a known or characterized value is compared to the result obtained through the routine analysis of the sample in the laboratory to evaluate the proficiency of an analyst or laboratory. This was formerly referred to as a PE.

periodic calibration
A calibration that is performed at prescribed intervals for equipment such as balances, thermometers, and balance weights. In general, they are performed on equipment that are distinct, singular purpose units, and are relatively stable in performance.

population
A generic term denoting any finite or infinite collection of individual things, objects, or events.

practical quantitation limit (PQL)
The lowest concentration a method can reliably achieve within limits of precision and accuracy and is derived from empirical, matrix-free method performance studies.

precision
Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements.

preventive maintenance

Glossary (continued)

An organized program within STL Knoxville of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use.

primary standard
A material having a known, stable property that can be accurately measured or derived from established physical or chemical constants. It is readily reproducible and can be accepted (within stated limits) and used to establish the same value of another substance or item.

procedure
Detailed instructions to permit replication of a method. (See standard operating procedure.)

proficiency testing
A series of planned tests which will determine the ability of field technicians or laboratory analysts to perform routine analyses. The results from this testing may be used for comparison against established criteria or for relative comparisons among the data from a group of technicians or analysts.

project-specific reporting limit (PSRL)
See reporting limit.

protocol
Methodology specified in regulatory, authoritative, or contractual situations.

QC batch
The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period.

Glossary (continued)

QC check sample
A reference matrix containing known concentrations of parameters of interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria, corrective actions are taken.

qualification (personnel)
The characteristics of abilities gained through education, training, or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

quality
The sum of features and properties/characteristics of a process, item, or service that bears on its ability to meet the stated needs of the user.

quality assurance (QA)
An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

Quality Management Plan (QMP)
A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality Assurance Project or Program Plan (QAPP)
A formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure the results of the work performed will satisfy the stated performance criteria.

quality control (QC)
The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that it meets the stated requirements established by the client or by STL.

quality improvement

Glossary (continued)

The process of improving the quality of operations. This process encourages worker recommendations for improvement of work processes and requires timely management evaluation and feedback or implementation.

quality management
That aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality management system.

quality management system (QMS)
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

random error
Variations of repeated measurements that are random in nature and individually not predictable.

range
The difference between the largest and smallest numbers in a set of numbers.

raw data
All documentation associated with the original recording of analytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

reagent water
Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. The reagent water's purity and acceptability is verified by analysis with each set of samples.

recovery
See percent recovery.

Glossary (continued)

reference method
A method of known and demonstrated accuracy.

regression coefficients
The quantities describing the slope and intercept of a regression line.

relative error
An error expressed as a percentage of the true value or accepted reference value.

relative percent different (RPD)
Statistic for evaluating the precision of a replicate set. For replicate results:

$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$

where: X_1 = first observed concentration or recovery
X_2 = second observed concentration or recovery

Glossary (continued)

relative response factor (RRF)
A measure of the relative mass spectral response of a compound compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. Because a RRF is the comparison of two responses, it is a unitless number. RRFs are determined by the following equation:

$$RRF = \frac{A_x}{A_{IS}} x \frac{C_{IS}}{C_x}$$

where: A = area of the characteristic ion measured
C = concentration
IS = internal standard
x = analyte of interest

relative standard deviation (RSD)
See coefficient of variation.

reporting limit (RL)
One of two types of reporting limit conventions within STL. The Reporting Limit (RL) is a uniform reporting limit based on an evaluation of the PQLs at STL Knoxville and the expected method performance in routine water and soil matrices. Project Specific Reporting Limits (PSRLs) are reporting limits that are defined by project requirements.

representative sample
A sample taken to represent a lot or population as accurately and precisely as possible.

representativeness
Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At STL Knoxville, every effort must be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

Glossary (continued)

reproducibility
The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

response factor (RF)
A factor derived from the calibration of a compound that is used in the quantitation calculation of sample analytes. A response factor may be derived from an external standard calibration (then called a Calibration Factor) or from an internal standard calibration (then called a Relative Response Factor).

secondary standard
A material having a property that is calibrated against a primary standard.

self assessment
Assessments of work conducted by individuals, groups, or organizations directly responsible for overseeing or performing the work.

shall
Denotes a requirement that is mandatory and has to be met.

should
Denotes a guideline or recommendation.

standard addition
The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish, by extrapolation of the plotted responses, the level of the analyte of interest present in the original sample.

standard deviation
A measure of the dispersion about the mean of the elements in a population. The square root of the variance of a set of values:

Glossary (continued)

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

where: s = standard deviation
Σ = sum of
X = observed values
n = number of observations

standardization
The establishment of the value of a potential standard with respect to an established or known standard.

standard method
A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.

standard operating procedure (SOP)
A written document that details an operation, analysis, or action, with prescribed techniques and steps, that is officially approved as the method for performing certain routine or repetitive tasks.

standard reference material (SRM)
A material produced in quantity, of which certain properties have been certified by the National Institute of Standards and Technology (NIST), formerly NBS, or other agencies to the extent possible to satisfy its intended use.

standard verification
Standard is checked by STL or the vendor versus a known specification.

statistic
A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.

stock solution

Glossary (continued)

A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.

subsample
A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

supplier
See vendor.

surrogate (surrogate standard)
Compounds, when required by a method, that are used added to every blank, sample, LCS, matrix spike, matrix spike duplicate, and standard. They are used to evaluate analytical efficiency by measuring recovery. Surrogates include brominated, fluorinated, or isotopically-labeled compounds that are not expected to be detected in environmental media.

systematic error
The condition of a consistent deviation of the results of a measurement process from the reference or known level.

systems audit or evaluation
A systematic on-site qualitative review of facilities, procedures, equipment, training, record keeping, data verification, and reporting aspects of a quality assurance system to arrive at a measure of the capability of the system. Within STL, system audits or evaluations are performed on a periodic basis.

technique
Physical or chemical principle for characterizing materials of chemical systems.

traceability of data
The entire documented chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction, and storage activities. The documentation will allow complete reconstruction of the data.

traceability of samples

Glossary (continued)

During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers. These sample numbers shall be accompanied by documentation (chain-of-custody form) which clearly identifies all parameters associated with sample acquisition. All additional sample numbering systems applied to the sample must be clearly cross-referenced to the field sample number to provide for traceability of samples from acquisition to reporting of sample results.

traceability of standards
The ability of an analytical standard material used for calibration purposes to be traced to its source. The standards used by STL must be traceable via written documentation to sources which produce or sell verified or certified standards.

validation - computer software
The process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.

validation - data
The process of a second party performing a systematic review of the raw and final data produced by a laboratory using predetermined criteria to ascertain the validity of the data with respect to the criteria.

vendor
Any individual or organization furnishing items or services or performing work according to a procurement document. This is an all-inclusive term used in place of any of the following: supplier, seller, contractor, subcontractor, or consultant.

verification - computer software
The process of checking the accuracy of manually entered or automatically (electronically) calculated information.

Glossary (continued)

verification - data

The process of reviewing data to ensure that data reduction has been correctly performed and that analytical results to be reported correspond to the data acquired and processed.

Table Section

TABLE 2-1
LQM Source Documents Requirements Matrix

STL LQM	EPA QA/R-2[1]	ANSI/ASQC E4-1994[2]	NQA-1[3], [7]	414.1A[4]	ANSI N 13.30[5]	ANSI/ASQC Q2-1991[6], [8]
1.0 Management Commitment and Organization	3.2 Management and Organization	2.1 Management and Organization	1 Organization	4 b 1)(a) Program	1.1 Introduction 1.2 Purpose 1.3 Scope	5.0 Management Responsibility
2.0 Quality System and Description	3.3 Quality System and Description	2.2 Quality System and Description	2 Quality Assurance Program	4 b 1)(a) Program	2.1 Special Word Usage 2.2 Specific Terms 5.1 Quality Assurance 5.2 Quality Control	5.2 Quality System
3.0 Document Control and Records Management	3.6 Documentation and Records	2.5 Documents and Records	6 Document Control 17 Quality Assurance Records	4 b 1)(d) Documents and Records	3.6 Direct Bioassay-Record Retention 4.5 Indirect Bioassay Record Retention	8.4 Quality Documentation and Records
4.0 Staff Qualification, Orientation and Training	3.4 Personnel Qualification and Training	2.3 Personnel Training and Qualification		4 b 1)(b) Personnel Training and Qualification	3.2 Personnel Preparation	14.0 Personnel
5.0 Procurement of Supplies and Services	3.5 Procurement of Items and Services	2.4 Procurement of Items and Services	4 Procurement Document Control 7 Control of Purchased Items and Services	4 b 2)(c) Procurement	N/A	7.0 Quality in Procurement 13.0 Subcontracting
6.0 Computer Hardware and Software	3.7 Computer Hardware and Software	2.6 Computer Hardware and Software	3 Design Control 11 Test Control	N/A	N/A	ISO 9000-3[9]

TABLE 2-1
LQM Source Documents Requirements Matrix (continued)

STL LQM	EPA QA/R-2[1]	ANSI/ASQC E4-1994[2]	NQA-1[3], [7]	414.1A[4]	ANSI N 13.30[5]	ANSI/ASQC Q2-1991[6], [8]
7.0 Contract Review and Project Planning	3.8 Planning	2.7 Planning 3.1 Planning and Scoping 3.3 Implementation of Planned Operations	2 Quality Assurance Program 3 Design Control 5 Instructions, Procedures, and Drawings 8 Identification and Control of Items 9 Control of Processes 11 Test Control 13 Handling, Storage, and Shipping	4 b 1) (a) Program 4 b 2 (b) Design	3.1 Facility Criteria 3.4 Direct Bioassay-Performance Criteria for Service Laboratories 3.5 Direct Bioassay-Reporting Results 4.1 Indirect Bioassay-Responsibilities of the Service Laboratory Customer 4.2 Indirect Bioassay-Analytical Methodology 4.3 Indirect Bioassay-Performance Criteria for Service Laboratories 5.2 Quality Control	6.3.3 Quality Plans
8.0 Work Processes and Operations	3.9 Implementation of Work Processes	2.8 Implementation of Work Processes	1 Organization 5 Instructions, Procedures, and Drawings	4 b 2 (a) Work Processes 4 b 2 (b) Design	3.1 Facility Criteria	8.0 Laboratory Operations Quality Assurance 9.0 Control of Measuring and Test Equipment 10.0 Data Validation 15.0 Use of Statistical Methods

TABLE 2-1
LQM Source Documents Requirements Matrix (continued)

STL LQM	EPA QA/R-2[1]	ANSI/ASQC E4-1994[2]	NQA-1[3], [7]	414.1A[4]	ANSI N 13.30[5]	ANSI/ASQC Q2-1991[6], [8]
	3.9 Implementation of Work Processes (Continued)	3.2 Design of Data Collection Operations	10 Inspection 12 Control of Measuring and Test Equipment 14 Inspection, Test, and Operating Status	4 b 2 (d) Inspection and Acceptance Testing		
9.0 Quality Assessment and Response	3.10 Assessment and Response 3.11 Quality Improvement	2.9 Assessment and Response 3.4 Assessment and Response 3.5 Assessment and Verification of Data Usability 2.10 Quality Improvement	2 Quality Assurance Program 13 Handling, Storage, and Shipping 15 Control of Non-conforming Items 16 Corrective Action 18 Audits	4 b 3 (a) Management Assessment 4 b 3 (b) Independent Assessment 4 b 1 c) Quality Improvement	3.3 Direct Bioassay-Interpretation of Measurements 3.5 Direct Bioassay-Reporting Results 4.4 Indirect Bioassay-Reporting Results 6.1 Direct Bioassay Measurements 6.2 Indirect Bioassay Measurements	16.0 Nonconformity 17.0 Corrective Action 18.0 Auditing the Quality System

Footnotes

(1) EPA Requirements for Quality Management Plans, U.S. Environmental Protection Agency, EPA QA/R-2, March 2001
(2) Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, American Society for Quality Control, Energy and Environmental Quality Division, Environmental Issues Group, ANSI/ASQC E4-1994 (Formerly EQA-1), January 1994
(3) Quality Assurance Program Requirements for Nuclear Facilities, The American Society of Mechanical Engineers, ASME NQA-1-1989 edition
(4) Quality Assurance, Office of Nuclear Energy & Office of Environment, Safety, and Health, United States Department of Energy, DOE ORDER 414.1A, September 1999
(5) Performance Criteria for Radiobioassay, ANSI N 13.30, September 1989
(6) Quality Management and Quality System Elements for Laboratories - Guidelines, American National Standard, American Society for Quality Control, ANSI/ASQC Q2-1991
(7) Section II, "Basic Requirements."
(8) Technically equivalent to ISO 9001.
(9) Quality Management and Quality Assurance Standards, ISO 9000, Part 3, "Guidelines for the Application of ISO 9001 to the Development, Supply and Maintenance of Software."

TABLE 2-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
a) A quality policy statement, including objectives and commitments, by top management	LQM Chapter 1
b) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts	LQM Chapter 1
c) The relationship between management, technical operations, support services and the quality system	LQM Chapter 1 LQM Chapter 7
d) Procedures to ensure that all records required under this chapter are retained as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force	LQM Chapter 2 LQM Chapter 3
e) Job descriptions of key staff and reference to the job descriptions of other staff	LQM Chapter 1 and Chapter 4 Separate document (hardcopy and/or electronic) provides job descriptions
f) Identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed concurrence, (with appropriate titles) of all responsible parties including the QA officer, technical director, and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager	LQM Title/Approval Page
g) The laboratory's procedures for achieving traceability of measurements	LQM Chapter 8

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, Approved July 12, 2002

TABLE 2-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals (continued)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
h) A list of all test methods under which the laboratory performs its accredited testing	LQM Table 8-1
i) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	LQM Chapter 7
j) Reference to the calibration and/or verification test procedures used	LQM Chapter 8 Tables 8-17 through 8-20
k) Procedures for handling submitted samples	LQM Sections 8.5.2 and 8.5.3
l) Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests	Equipment list is Table 8-3 LQM Sections 5.2.4, 8.1, 8.5.4, 8.11 to 8.12
m) Reference to procedures for calibration, verification and maintenance of equipment	Calibrations in Tables 8-17 through 8-20 LQM Sections 8.5.4, 8.11 to 8.12
n) Reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes	LQM Section 9.4.5
o) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	LQM Chapter 9 SOP KNOX-QA-0008, "Nonconformance and Corrective Action"

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems.

TABLE 2-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals (continued)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
p) The laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	LQM Section 7.4 and 9.1
q) Procedures for dealing with complaints	LQM Section 9.2
r) Procedures for protecting confidentiality and proprietary rights (including national security concerns)	LQM Section 8.9.6
s) Procedures for audits and data reviews	LQM Section 5.3.6, 8.8 and 9.4
t) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and/or receive any needed training	LQM Chapter 4 SOP KNOX-QA-0009, "Employee Orientation and Training"
u) Ethics policy statement developed by the laboratory and process/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions	LQM Section 1.4 SOP S-Q-004 – Acceptable Manual Integration Practices P-T-001 – Selection of Data Points Required for Initial Calib.
v) Reference to procedures for reporting analytical results	LQM Section 8.9 SOP S-T-004 "Reporting Limits"

TABLE 2-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals (cont.)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
w) A table of contents and applicable list of references, glossaries, and appendices	LQM Table of Contents Table 8-2 Table 2-1
x) Data integrity procedures defined in detail including • Data integrity training • Signed data integrity documentation for all laboratory employees • In depth, periodic monitoring of data integrity • Data integrity procedure documentation (see NELAC 5.4.2.6)	LQM Section 4 Ethics Policy, P-L-006 LQM, Section 9

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, Approved May 25, 2001

TABLE 2-3
STL Knoxville Quality Documents and Required Approval

Quality Document	Required Approvals
Laboratory Quality Manual (LQM)	• Laboratory Director • Technical Directors • Quality Assurance Manager
STL Knoxville Policies	• Laboratory Director • Quality Assurance Manager
STL Knoxville Standard Operating Procedures (SOPs)	• Laboratory Director • Technical Specialist • Laboratory Health and Safety Coordinator(1) • Quality Assurance Manager

(1) Required only if procedure encompasses more than standard office safety requirements.

TABLE 2-4
STL Quality Document Review Frequency

Document Type	Frequency of Review	Responsible Party
Laboratory Quality Manual (LQM)	Every Year	Quality Assurance Manager
Standard Operating Procedures (SOP) and Policies	Every Two Years (1)	Quality Assurance Manager & Operations Manager, along with Group (Area) Leaders

(1) SOPs for analytical methods performed in support of DOD (i.e., NFESC and USACE) must be reviewed annually.

TABLE 5-1
List of STL Knoxville Quality-Related Items that Require Evaluation Prior to Use

Quality-Related Item
Acetone
Dichloromethane
Hexane
Hydrochloric acid
Methanol
Nitric acid
Hydrogen Peroxide
Sulfuric acid
Toluene

TABLE 6-1
GALP Cross Reference To LQM

GALP Section	GALP Guidance	STL Document
8.1 Laboratory Management	8.1.1 ensure that personnel clearly understand the functions they are to perform	LQM 1.6.2, 1.6.4, and 4.0
	8.1.2 ensure that QAU monitors computer activities	LQM 9.5
	8.1.3 ensure that personnel, resources, and facilities are adequate and available as scheduled	LQM 1.6.1-1.6.4
	8.1.4 receive reports of QAU inspection and audit reports, and ensure corrective actions are promptly taken in response to any deficiency	LQM 9.5
	8.1.5 approve SOPs related to the computer activities, and ensure that devations to the SOPs are documented	LQM 3.3 and 9.1.1
	8.1.6 assure that GALP provisions are followed	LQM 6.0, KNOX-IT-0001
8.2 Personnel	8.2.1 must have adequate education, training, and experience to perform assigned IT functions	LQM 4.0, KNOX-QA-0009
	8.2.2 a summary of training, experience, and job description must be maintained	LQM 4.1
	8.2.3 personnel must be of sufficient number for timely and proper operation of the computer systems	LQM 1.6.1 – 1.6.4
8.3 Quality Assurance Personnel	8.3.1 shall be separate and independent of IT personnel, and shall report directly to laboratory management	LQM 1.6.1
	8.3.2 shall have immediate access to the computer data, SOPs, and other records	LQM 1.61 and 9.5
	8.3.3 inspect the LIMS at intervals to ensure the integrity of LIMS raw data, and shall present inspection reports to management	LQM 9.5
	8.3.4 determine that no deviations from approved SOPs were made without proper authorization and documentation	LQM 9.1.1
	8.3.5 periodically audit raw data to ensure their integrity	LQM 9.5.3
	8.3.6 maintain adequate records of the QAU operations	LQM 9.5
8.4 LIMS Raw Data	8.4.1 LIMS raw data and the storage media on which they reside must be identified and documented. The documentation shall be included in the lab's SOPs.	KNOX-IT-0001
	8.4.2 the individual(s) responsible for entering and recording LIMS raw data must be uniquely identified, together with the date and time the data were entered	KNOX-QA-0013

TABLE 6-1
GALP Cross Reference To LQM (cont.)

GALP Section	GALP Guidance	STL Document
8.4 LIMS Raw Data, continued	8.4.3 the instrument transmitting raw data must be uniquely identified in the record, together with the date and time of transmission	KNOX-QA-0013
	8.4.4 procedures and practices used to verify LIMS raw data must be documented in controlled SOPs	KNOX-IT-0001; LQM 8.8-8.8.3
	8.4.5 procedures and practices for making changes to LIMS raw data are documented and provide evidence of change, preserve the original recorded documentation are dated, indicate the reason for the change, identify the person who made the change and, if different, the person who authorized the change. These procedures shall be included in the laboratory's SOPs.	KNOX-IT-0001, KNOX-QA-0013
8.5 Software	8.5.1 SOPs shall be established for: a. software development b. software testing c. change control d. version control e. maintaining historical file	KNOX-IT-0001
	8.5.2 documentation shall be maintained for: a. software description & functional requirements b. algorithms and formulas c. testing and quality assurance	KNOX-IT-0001
	8.5.3 all documentation is readily available in the facility where the software is used and SOPs are readily available where procedures are performed	KNOX-IT-0001
	8.5.4 a historical file of software and documentation shall be retained	KNOX-IT-0001
8.6 Security	Laboratory management shall ensure that security practices are adequate to assure the integrity of data	LQM 6.2;
8.7 Hardware	8.7.1 must be of adequate design and capacity, and a documented description maintained	LQM 6.1; KNOX-IT-0001
	8.7.2 must be installed in accordance with manufacturer's recommendations, and undergo documented acceptance testing as described in a laboratory SOP	KNOX-IT-0001; LQM 6.1
	8.7.3 testing, maintenance, and repair must be described in a laboratory SOP	KNOX-IT-0001
8.8 Comprehensive Testing	Management shall ensure that comprehensive testing shall be documented at least every 24 months or more frequently as a result of software changes.	KNOX-IT-0001

TABLE 6-1
GALP Cross Reference To LQM (continued)

GALP Section	GALP Guidance	STL Document
8.9 Records Retention	Procedures must be in place for the retention of LIMS raw data and documentation and records pertaining to LIMS	QMP; LQM 3.4-3.5
8.10 Facilities	8.10.1 the environmental conditions of the facility housing the LIMS must be controlled to protect against data loss	LQM 6.2
	8.10.2 environmental conditions for storing LIMS raw data and records must be adequate	LQM 6.2
8.11 SOPs	8.11.1 SOPs, as described above, must be maintained and readily available where the procedure is performed	LQM 3.1-3.2; SOP Index
	8.11.2 SOPs must be reviewed periodically to ensure that they are accurate	LQM 3.3
	8.11.3 SOPs must be authorized and controlled, with all changes subject to the same approvals and control	LQM 3.3
	8.11.4 an historical file of SOPs must be maintained	LQM 3.5

TABLE 8-1
Analytical Methods and Matrices

Analytical Parameters	Fields of Testing[1]				
	Drinking Water (SDWA)	**Non-Potable Water (CWA/RCRA)**	**Solid and Chemical Materials (RCRA-SW846)**	**Air and Emissions (CAA)**	**Biological Tissue**
Anions (Br^-, Cl^-, F^-, NO_2, NO_3, o-PO_4, SO_4	**EPA 300.0**	**EPA 300.0** **EPA 9056**	**EPA 9056**	Mod Method 26A/0050 (9057 Mod)	---
Ash	---	---	ASTM D482	---	---
Cyanide (Amenable)	---	**EPA 9012A** EPA 335.1	**EPA 9012A**	---	---
Cyanide (Total)	---	**EPA 9012A** EPA 335.4	**EPA 9012A**	---	---
Density	---	---	ASTM D1963, D854	---	---
Halogens	---	---	ASTM E442, EPA 5050	---	---
Heat of Combustion (Btu)	---	---	ASTM D5865, D240	---	---
Hexavalent Chromium	---	---	---	Method 0061/7199	---
Perchlorate	**EPA 314.0**	EPA 314.0		---	---
pH	**EPA 150.1**	**EPA 9040B** **EPA 150.1**	**EPA 9040B** **EPA 9045C**	---	---
Hardness	SM 2340B	**SM 2340B**	---	---	---
Particulates	---	---	---	PM-10, 40CFR, Part 50, App. J & App. B Method 0050/5 40CFR Part 60 App. A	---
Mercury	**EPA 245.1**	**EPA 245.1** **EPA 7470A**	**EPA 7470A** **EPA 7471A**	**EPA 0060/7470A** ASTM D6784-02 EPA IO-3.1/7470A **EPA Method 29/7470A**	EPA 7471A
Metals	**EPA 200.7**	**EPA 200.7** **EPA 6010B**	**EPA 6010B**	**EPA 0060/6010B** EPA IO-3.1/6010B **EPA Method 29/6010B**	EPA 6010B
Toxicity Characteristic Leaching Procedure (TCLP) & Synthetic Precipitation Leaching (SPLP)	---	---	**EPA 1311** **EPA 1312**	---	---
Viscosity	---	---	ASTM D445	---	---

TABLE 8-1 (continued)
Analytical Methods and Matrices

Analytical Parameters	Fields of Testing[1]				
	Drinking Water (SDWA)	**Non-Potable Water (CWA/RCRA)**	**Solid and Chemical Materials (RCRA-SW846)**	**Air and Emissions (CAA)**	**Biological Tissue**
Volatiles by GC/MS	---	**EPA 8260B**	**EPA 8260B**	**TO-14A, TO-15 EPA 0031/5041A (VOST)**	---
Volatiles by GC	---	---	---	EPA 0040 Condensates ASTM D1946-90	---
Semivolatiles by GC/MS	---	**EPA 8270C**	**EPA 8270C**	**TO-13A EPA 3542/8270C**	---
Semivolatiles by GC	---	**Pesticides EPA 8081A, PCBs EPA 8082, Congeners EPA 8082**	**Pesticides EPA 8081A, PCBs EPA 8082, Congeners EPA 8082**	**TO-4A**, TCO/Grav	PCBs EPA 8082, **Congeners EPA 8082**
Explosives by HPLC	---	**EPA 8330 EPA 8332**	**EPA 8330 EPA 8332**	---	---
Dioxins/Furans by HRGC/HRMS	**EPA 1613B**	**EPA 1613B EPA 8290**	**EPA 8290**	**TO-9A**, Method 23/8290 **EPA 0023A/8290**	**EPA 8290 EPA 1613B**
PCBs by HRGC/HRMS	---	**EPA 1668A**	**EPA 1668A**	EPA 1668A	**EPA 1668A**
Dioxins/Furans by Immunoassay Screen	---	---	EPA 4000, EPA 4025 Mod	---	---
PCBs by Immunoassay Screen	---	---	EPA 4000	---	---
Polycyclic Aromatic Hydrocarbons (PAH)	---	---	---	KNOX-ID-0016 (CARB 429 Mod, HRGC/LRMS)	---

Footnotes

(1) **NELAC accredited methods are highlighted in bold font.**

Table 8-2 Standard Operating Procedures

SOP No.	Rev. No.	Rev. Date	Title
Administrative			
KNOX-AD-0001	6	2/22/06	Document Storage
KNOX-AD-0002	8	4/28/05	Project Records
KNOX-AD-0003	3	4/28/05	Documenting Project Requirements
KNOX-AD-0004	7	1/12/06	Data Reporting
KNOX-AD-0005	2	1/30/06	Project Quote and Setup
KNOX-AD-0006	0	4/28/03	QDS Report Form Generation
Enzyme Immunoassay			
KNOX-EI-0001	0	2/1/06	Screening for Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans and Polychlorinated Biphenyls by Determination of Enzyme Immunoassay Toxic Equivalency Quotients (EIA-TEQs)
GC Laboratory			
KNOX-GC-0003	3	7/7/05	Permanent Gas Analysis by GC/TCD
KNOX-GC-0010	4	12/14/05	Analysis of Extractable Semivolatile and Nonvolatile Total Organics by Gas Chromatography and Gravimetric Analysis
KNOX-GC-0011	6	8/30/05	Analysis of Polychlorinated Biphenyl Congeners Based on SW-846 Method 8082
KNOX-GC-0014	5	8/30/05	Analysis of Organochlorine Pesticides Based on Method 8081A
KNOX-GC-0015	8	11/17/05	Analysis of PCBs Based on Method 8082 and TO-4A
KNOX-GC-0016	1	4/5/04	Analysis of Method 0040 Condensates by Gas Chromatography for Total Unspeciated Mass in the Volatile Range
KNOX-GC-0017	1	1/3/05	Analysis of Tedlar Bag Samples Collected by SW-Method 0040 by Gas Chromatography for Total Unspeciated Mass in the Volatile Range
Health & Safety			
KNOX-HS-0001	6	8/4/04	Radiological Sample Screening and Classification
KNOX-HS-0002	4	1/25/05	Waste Characterization and Categorization
KNOX-HS-0005	4	3/5/04	Waste Minimization Program
KNOX-HS-0006	3	8/11/04	Safety Precautions for Samples Containing Chemical Warfare Materials
KNOX-HS-0007	4	5/24/05	Facility Engineering Control Maintenance Program
KNOX-HS-0008	3	8/20/02	Safety Inspections and Audits
KNOX-HS-0010	5	1/16/04	Radiation Protection Program
KNOX-HS-0012	2	8/20/02	Radiation Safety Training
KNOX-HS-0014	1	6/11/01	Background and Source Check and Use of Portable Survey Instruments
KNOX-HS-0015	3	2/14/06	Operation of Ludlum 3030e Alpha Beta Counting Instrumentation
KNOX-HS-0016	3	1/6/06	Facility Radiological Surveys and Monitoring
KNOX-HS-0018	2	10/7/03	Inventory and Leak Testing of Sealed Sources
KNOX-HS-0019	1	6/11/01	Radioactive Sample Inventory
KNOX-HS-0021	0	8/21/01	Respiratory Protection Program
Specialty Organics Laboratory			
KNOX-ID-0004	6	9/27/05	Analysis of Polychlorinated Dioxins/Furans by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) Based on Methods 8290, 1613B, 23, 0023A and TO-9A
KNOX-ID-0012	2	4/6/06	Method 0023A and Method 0010 Sampling Train Pre-Test Preparation

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SOP No.	Rev. No.	Rev. Date	Title
KNOX-ID-0013	4	9/21/05	Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS
KNOX-ID-0016	4	4/11/06	Extraction and Isotope Dilution Analysis of Polynuclear Aromatic Hydrocarbons and Selected Semivolatile Organic Compounds by High Resolution Gas Chromatography-Low Resolution Selected Ion Monitoring Mass Spectrometry (HRGC/LRMS-SIM)
Information Technology			
KNOX-IT-0001	1	11/10/04	Good Automated Laboratory Practices
KNOX-IT-0002	3	9/9/04	Electronic Data and Software Backup
KNOX-IT-0003	2	12/21/05	Electronic Data Deliverable
KNOX-IT-0005	1	12/9/054	Laptop Computer Security
HPLC Laboratory			
KNOX-LC-0001	14	1/23/06	Analysis of Nitroaromatic and Nitramine Explosives by HPLC
KNOX-LC-0005	2	1/5/06	Analysis of Nitroglycerin by HPLC
KNOX-LC-0006	0	3/29/06	Extraction and Analysis of 2,4-Diaminotoluene, 2,6-Diaminotoluene and 4,4'-Methylenedianiline by HPLC
GCMS Laboratory			
KNOX-MS-0001	8	4/6/06	VOA Canister Analysis
KNOX-MS-0010	3	1/13/05	Volatile Analyte Screening by Purge and Trap
KNOX-MS-0011	5	7/26/05	VOST Analysis
KNOX-MS-0012	2	6/23/05	Unspeciated Mass Procedure
KNOX-MS-0014	3	12/29/05	Tentatively Identified Compounds (TICs)
KNOX-MS-0015	8	1/26/06	Determination of Volatile Organics by GCMS Based on Method 8260B
KNOX-MS-0016	6	1/30/06	GCMS Analysis Based on Methods 8270C and 625
KNOX-MS-0017	1	11/29/04	GCMS Analysis of Polyaromatic Hydrocarbons in Ambient Air Based on EPA Method TO13A
KNOX-MS-0018	1	7/22/05	Methanol Extraction of VOST Tubes
Metals Laboratory			
KNOX-IP-0002	2	1/23/06	Metals Sample Preparation based on Method 3030C
KNOX-IP-0003	4	1/9/06	Digestion of Total Metals in Filters
KNOX-IP-0004	3	1/20/06	Acid Digestion of Soils, SW846 Method 3050B
KNOX-IP-0005	3	4/22/04	Acid Digestion of Aqueous Samples by SW846 and EPA Method 200.7
KNOX-IP-0006	2	3/15/05	Ontario Hydro Mercury Sampling Train Preparation
KNOX-IP-0008	1	11/10/05	Preparation of Organic Waste Feed Samples for Total Metals Analysis
KNOX-MT-0004	3	1/11/06	Microwave-Assisted Acid Digestion of Sediments , Soils and Oils by Method 3051 and 3052
KNOX-MT-0005	2	4/9/03	Hardness by Calculation
KNOX-MT-0006	9	12/15/05	Multi-Metals (MMT) Sampling Train Preparation
KNOX-MT-0007	5	11/10/05	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method For Trace Element Analyses, SW-846 Method 6010B and EPA Method 200.7
KNOX-MT-0008	2	6/3/04	Sequential Extraction Procedure for Arsenic
KNOX-MT-0009	4	1/13/06	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1
KNOX-MT-0010	4	1/23/06	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption, SW-846 7471A and MCAWW 245.5

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SOP No.	Rev. No.	Rev. Date	Title
KNOX-MT-0013	1	12/20/04	Sequential Extraction Procedure for TAL Metals for EA Engineering, Science and Technology
KNOX-MT-0014	0	11/8/04	Metals Laboratory Data Review and Reporting
Organic Prep Laboratory			
KNOX-OP-0002	6	3/22/05	Extraction of PUF/XAD-2 or PUF Air Sampling Cartridges for TO-13A and TO-4 Analyses
KNOX-OP-0009	3	1/11/06	Method 0010 Sample Train (Modified Method 5) Component Extraction
KNOX-OP-0011	6	7/7/05	Extraction and Cleanup of Organic Compounds From Waters and Soils, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series Methods
KNOX-OP-0012	3	7/7/05	Toxicity Characterisitic Leaching Procedure and Synthetic Precipitation Leaching Procedure
KNOX-OP-0014	3	6/3/054	Extraction and Cleanup of PCB Congeners from Waters, Wastes and Soils
Quality Assurance			
KNOX-QA-0001	6	12/27/05	Standard/Reagent Labeling and Documentation
KNOX-QA-0002	6	1/31/06	Glassware Cleaning
KNOX-QA-0003	5	2/14/06	Preventive Maintenance
KNOX-QA-0004	3	10/24/03	Control Limits and Control Charting
KNOX-QA-0005	4	12/16/05	Balance Calibration and Weight Verification
KNOX-QA-0006	1	2/9/04	Subsampling
KNOX-QA-0007	4	6/14/05	Quality Assurance Records
KNOX-QA-0008	2	9/22/04	Nonconformance and Corrective Action System
KNOX-QA-0009	3	1/30/06	Personnel Orientation and Training
KNOX-QA-0010	5	4/12/06	Temperature Monitoring and Thermometer Calibration
KNOX-QA-0011	3	6/16/05	Document Control and Distribution
KNOX-QA-0012	3	10/24/03	Pipetter Calibration
KNOX-QA-0013	3	10/20/03	Instrument Records and Logbooks
KNOX-QA-0014	0	3/3/03	Air Sampling Media Tracking and Quality Control
KNOX-QA-0015	3	6/16/05	Facility Security
KNOX-QA-0016	3	6/13/05	Routine Monitoring of Deionized Water
KNOX-QA-0017	6	6/9/054	Audits
KNOX-QA-0018	3	8/29/05	Preparation and Management of Standard Operating Procedures
KNOX-QA-0020	1	11/8/05	Selection and Evaluation of Subcontract Laboratories
S-Q-002	3.1	2/3/05	System Audits
S-Q-003	1	4/18/02	Method Detection Limits Studies
S-Q-004	2	9/17/04	Acceptable Manual Integration Practices
S-Q-005	1.1	2/3/05	Data Recall Process
S-Q-007	1	2/3/05	Data Authenticity Audit Procedure
S-T-001	2	11/8/04	Testing of Solvents and Acids
S-T-002	2	11/8/04	Reporting Limits for STL Laboratories Using QuantIMS
Sample Receiving			
KNOX-SC-0001	7	6/6/05	Canister Cleaning and Preparation
KNOX-SC-0003	10	7/26/05	Samples Receipt and Log In

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SOP No.	Rev. No.	Rev. Date	Title
KNOX-SC-0004	5	6/21/05	Internal Chain of Custody
KNOX-SC-0005	3	10/6/03	Sample Disposal, Return and Archive
KNOX-SC-0006	1	7/31/03	Bottle and Cooler Preparation
Wet Chemistry Laboratory			
KNOX-WC-0001	6	3/12/04	Cyanide Analysis
KNOX-WC-0003	1	1/21/04	Analysis of Hexavalent Chromium Based on SW-846 Methods 0061/7199
KNOX-WC-0005	6	3/23/04	Anion Analysis by Ion Chromatography
KNOX-WC-0006	2	3/8/06	Method 0050/5 Train Particulate
KNOX-WC-0007	4	3/20/03	Ph Analysis
KNOX-WC-0010	1	11/13/03	Heat of Combustion (Gross Calorific Value)
KNOX-WC-0011	1	11/12/03	Determination of Viscosity
KNOX-WC-0012	4	1/9/04	Percent Moisture
KNOX-WC-0013	2	11/12/03	Ash Determination
KNOX-WC-0014	5	3/24/04	Determination of Perchlorate by Ion Chromatography
KNOX-WC-0015	1	11/14/03	Density
KNOX-WC-0016	1	11/14/03	Halogen Determination
KNOX-WC-0017	0	9/15/05	Perchlorate Data Review and Reporting
KNOX-WC-0018	0	10/13/05	Ion Chromatography Data Review and Reporting
KNOX-WC-0028	5	4/7/05	Total Suspended Particulate (TSP) and PM-10

TABLE 8-3 Instrument List

Instrument Type	Manufacturer/ Serial Number (S/N)[1]	Model	Purchase/ Installation Date	Condition When Purchased	Autosampler
GCMS	Hewlett Packard S/N 3435A01974-MT	5972A	1999	New	Yes
	Hewlett Packard S/N US82321805-MA	5973	1999	New	Yes
	Hewlett Packard S/N US80310976-MZ	5973	1998	New	Yes
	Hewlett Packard S/N US80210991-MX	5973	1998	New	Yes
	Hewlett Packard S/N US91911868-MJ	5973	2000	New	Yes
	Agilent Technologies S/N US10129076-ME	5973 Network	2001	New	Yes
	Agilent Technologies S/N US10451834-MD	5973 Network	2001	New	Yes
	Agilent Technologies S/N US10345111-MK	5973 Network	2003	New	Yes (Solatek 72 autosampler (Tekmar/ Dohrmann)
	Agilent Technologies S/N US44621247-MG	5973 Network	2005	New	Yes
GCMS Specialty Organics	Hewlett Packard S/N US94260093-MY	5973	2000	New	Yes
	Finnigan S/N 2631	MAT-95	1991	New	Yes
	Finnigan S/N 2725	MAT-95S	1998	New	Yes

TABLE 8-3 Instrument List

Instrument Type	Manufacturer/ Serial Number (S/N)[1]	Model	Purchase/ Installation Date	Condition When Purchased	Autosampler
GCMS Specialty Organics, continued	Finnigan S/N (GC) US00001760	MAT-90S	2001	Used	Yes
Prep Specialty Organics	Isco S/N 212287 (Pump)	Combi Flash Sq 1600	2001	New	Yes
GC	Varian S/N 3961-GR	3400 TCD	1989	New	Yes
	Hewlett Packard S/N US00021272-ST	6890 ECD	1987	New	Yes
	Agilent Technologies S/N US00041050-GH	6890 ECD	2001	New	Yes
	Hewlett Packard S/N 2618A07840-MN	5890 ECD	1986	Used	Yes
	Agilent Technologies S/N US10346012-CD	6890 ECD	2003	New	Yes
	Varian S/N 4644 GTO	3400 FID	1987	Used	No
Metals	Leeman Labs, Inc. Install #62434	Hydra AA	2001	New	Yes
	Thermo Jarrell Ash S/N 512990	ICAP 61E Trace	2000	New	Yes
	Thermo Jarrell Ash S/N 248490	ICAP 61E Trace	1994	New	Yes

TABLE 8-3 Instrument List

Instrument Type	Manufacturer/ Serial Number (S/N)[1]	Model	Purchase/ Installation Date	Condition When Purchased	Autosampler
HPLC	Hewlett Packard S/N DE91606293-Ace	Series 1100	1999	New	Yes
	Hewlett Packard S/N US72102530-Semeka	Series 1100	1998	New	Yes
Wet Chemistry	Lachat S/N A83000-2135	Quick Chem 8000	2003	New	Yes
	Dionex S/N 00070167 Lil Mike	DX 320	2000	New	Yes
	Dionex S/N 02070458 Aragorn	DX 600	2002	New	Yes
	Dionex S/N 03100244	ICS-1500	2003	New	Yes
Organic Prep	J_2 Scientific S/N 05L-1179-4.0 UP	GPC Accuprep	2006	New	Yes
	Accumet S/N C0025140	Model 15 pH meter	1998	New	No
ACS	Varian S/N 0295	3600 FID	1988	New	No
	Varian S/N 3890	3300 FID	1987	New	No
	Parr S/N M5650	1266 Calorimeter	2003	New	No

[1] Information about preventive maintenance can be found in the STL Knoxville SOP KNOX-QA-0003, current revision, Preventive Maintenance.

Table 8-4-1
Anion Methods and Reporting Limits (RL) [1]

Method 300.0/9056			
Parameter	**CAS Number**	**Water RL (mg/L, except as noted)**	**Solid RL[1] (mg/Kg, except as noted)**
Bromide	24959-67-9	1.0	10
Chloride	16887-00-6	1.0	10
Fluoride	16984-48-8	1.0	10
Nitrate	---	0.50	5.0
Nitrite	---	0.50	5.0
Orthophosphate	---	1.0	10
Sulfate	14808-79-8	1.0	10

Methods 9057Mod, 0050/26A Mod[2]	
Parameter	**Air RL ug**
Chlorine	100
Hydrogen Chloride	100

(1) Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.
(2) Reporting limits for anions for Methods 26A (Mod)/9057 (Mod) are dependent on the impinger volume. RLs represent 100 mL impinger volume.
NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-2
Physical Parameter Methods and Reporting Limits (RL) [1]

Parameter	Method	RL[1] (mg/Kg, except as noted)
Ash	D482	200
Density	D1963, D854	0.01 g/cm^3
Heat of Combustion (Btu)	D240, D5865	1800 Btu/lb
Viscosity	D445	0.35 cSt
Total Halogens	E442, SW-846 5050 & 9056	200

(1) Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis. NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-3
Wet Chemistry Methods
Reporting Limits

Parameter	CAS Number	Method	Water RL (mg/L, except as noted)	Solid RL[(1)] (mg/Kg, except as noted)	Air RL (ug)[(2)]
Cyanide	57-12-5	335.4/ 9012A (LACHAT)	0.010	0.5	--
Cyanide, amenable	--	335.1/ 9012A (LACHAT)	0.010	NA	--
Hexavalent Chromium	18540-29-9	0061/7199	--	--	0.05 ug
Perchlorate	14797-73-0	314.0	4.0 ug/L	40 ug/kg	--
Percent Moisture	-	160.3 Mod	NA	0.1%	--
pH	-	150.1/ 9040B	0.10 Standard Units	NA	--
	-	9045C	NA	0.10 Standard Units	--

(1) Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.
NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

(2) Air reporting limit is based on 100 mL impinger volume.

Table 8-4-4
Particulate Reporting Limits (RL)

Parameter	Method	RL
Particulate Matter as PM-10	PM-10, 40 CFR Part 50, App. J and App. B	5000 ug
Particulate, total	40 CFR Part 60, App. A/ 0050/5	0.5 mg

Table 8-4-5
ICP Metals by EPA Methods 6010B, 200.7[(1)], 0060, 29 and IO-3.1
Reporting Limits (RL)

Element	CAS Number	Water RL 6010B/200.7 (µg/L)	Soil RL[(2)] 6010B (mg/Kg)	Air RL, Front Half/ Back Half of Air Train 0060/29 (ug)	PM-10 Filter RL IO-3.1 (ug)
Aluminum	7429-90-5	400	40	20	90.0
Antimony	7440-36-0	60	6.0	6	13.5
Arsenic	7440-38-2	20	2.0	1.0	2.3
Barium	7440-39-3	10	1.0	20	45.0
Beryllium	7440-41-7	10	1.0	0.5	1.1
Boron	7440-42-8	200	20	---	180
Cadmium	7440-43-9	5.0	0.50	0.5	1.1
Calcium	7440-70-2	5000	500	500	2250
Chromium	7440-47-3	15	1.5	1.0	9.0
Cobalt	7440-48-4	50	5.0	5.0	11.3
Copper	7440-50-8	25	2.5	2.5	5.6
Iron	7439-89-6	200	20	10	180
Lead	7439-92-1	15	1.5	1.0	2.3
Lithium	7439-93-2	50	5.0	5.0	---
Magnesium	7439-95-4	5000	500	500	1130
Manganese	7439-96-5	15	1.5	1.5	3.4
Molybdenum	7439-98-7	40	4.0	4.0	---
Nickel	7440-02-0	40	4.0	4.0	9.0
Phosphorus	7723-14-0	300	30	30	540
Potassium	7440-09-7	5000	500	500	1130
Selenium	7782-49-2	15	1.5	1.0	2.3
Silicon	7440-21-3	500	---	---	---
Silver	7440-22-4	30	3.0	2.0	4.5
Sodium	7440-22-4	5000	500	500	4500
Strontium	7440-24-6	50	5.0	5.0	---
Thallium	7440-28-0	35	3.5	2.0	4.5
Tin	7440-31-5	100	10	10	90
Titanium	7440-32-6	50	5.0	5.0	---
Vanadium	7440-62-2	25	2.5	5.0	18.0
Zinc	7440-66-6	60	6.0	2.0	18.0

[(1)] Method 200.7 is for waters only.
[(2)] Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.
NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-6
Hardness by Standard Methods 2340B
Reporting Limit (RL)

Element	Water RL (mg/L)
Hardness	33

Table 8-4-7
CVAA Mercury[1] Reporting Limits (RL)

Element	CAS Number	Method	Water RL (ug/L)	Soil RL[2] (mg/kg)
Mercury	7439-97-6	245.1/245.5, 7470A/7471A	0.2	0.033

Ontario Hydro						
Element	CAS Number	Method	Front Half RL (Filter) (ug)	Back Half RL[3] HNO_3/H_2O_2 (ug)	Back Half RL[3] $KMnO_4$ (ug)	Back Half RL[3] KCl (ug)
Mercury	7439-97-6	ASTM D6784-02	0.01	0.5	0.2	0.2

Method 29/0060							
Element	CAS Number	Method	Front Half RL (Filter) (ug)	Back Half RL Empty[3] (ug)	Back Half RL HNO_3/H_2O_2[3] (ug)	Back Half RL $KMnO_4$[3] (ug)	Back Half RL HCl[3] (ug)
Mercury	7439-97-6	29/0060	0.20	0.40	0.20	0.04	0.10

PM-10 Filter			
Element	CAS Number	Method	Reporting Limit (ug)
Mercury	7439-97-6	IO-3.1/7470A	0.45

[1] Mercury is analyzed by cold vapor atomic absorption spectroscopy.
[2] Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.
[3] Back Half Reporting limits in ug depend on the impinger volume. The reporting limits presented are based on a 1 liter volume.

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.
RLs and MDLs for source air testing are also available upon request.

Table 8-4-8
Pesticides by GC EPA Method 8081A
Reporting Limits (RL)

Analyte	CAS Number	Water RL (µg/L)	Soil RL [1] (µg/Kg)	Waste RL (µg/Kg)	Air RL (µg/sample)
α -BHC	319-84-6	0.05	1.7	500	0.50
β -BHC	319-85-7	0.05	1.7	500	0.50
δ -BHC	319-86-8	0.05	1.7	500	0.50
γ -BHC(Lindane)	58-89-9	0.05	1.7	500	0.50
α -chlordane	5103-71-9	0.05	1.7	500	0.50
γ -chlordane	5103-74-2	0.05	1.7	500	0.50
Heptachlor	76-44-8	0.05	1.7	500	0.50
Aldrin	309-00-2	0.05	1.7	500	0.50
Heptachlor Epoxide	1024-57-3	0.05	1.7	500	0.50
Endosulfan I	959-98-8	0.05	1.7	500	0.50
Dieldrin	60-57-1	0.05	1.7	500	0.50
4,4'-DDE	72-55-9	0.05	1.7	500	0.50
Endrin	72-20-8	0.05	1.7	500	0.50
Endosulfan II	33213-65-9	0.05	1.7	500	0.50
4,4'-DDD	72-54-8	0.05	1.7	500	0.50
Endosulfan sulfate	1031-07-8	0.05	1.7	500	0.50
4,4'-DDT	50-29-3	0.05	1.7	500	0.50
Methoxychlor	72-43-5	0.1	3.3	1000	1.0
Endrin Aldehyde	7421-93-4	0.05	1.7	500	0.50
Endrin Ketone	53494-70-5	0.05	1.7	500	0.50
Chlordane (Technical)	57-74-9	0.5	17	5000	NA
Toxaphene	8001-35-2	2.0	67	20000	NA

[1] Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-9
PCBs by GC EPA Methods 8082 and TO-4A
Reporting Limits (RL)

Analyte	CAS Number	Water RL (µg/L)	Soil RL [1] (µg/Kg)	Air RL (ug)	Tissue RL (µg/Kg)
Aroclor 1016	12674-11-2	1.0	33	1.0	10
Aroclor 1221	11104-28-2	1.0	33	1.0	10
Aroclor 1232	11141-16-5	1.0	33	1.0	10
Aroclor 1242	53469-21-9	1.0	33	1.0	10
Aroclor 1248	12672-29-6	1.0	33	1.0	10
Aroclor 1254	11097-69-1	1.0	33	1.0	10
Aroclor 1260	11096-82-5	1.0	33	1.0	10

[1] Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-10
PCB Congeners by GC EPA Method 8082
Reporting Limits (RL)

Analyte			Reporting Limit				
CAS Number	BZ[1]	Compound Name	Aqueous (ug/L)	Low-level Aqueous (ug/L)	Solid (ug/kg, Dry)	Tissue (ug/kg)	Waste ug/kg
2051-60-7	1	2-Chlorobiphenyl	0.2	0.02	20	20	2000
2051-62-9	3	4-Chlorobiphenyl	0.2	0.02	20	20	2000
16605-91-7	5	2,3-dichlorobiphenyl	0.01	0.001	1	1	100
34883-43-7	8	2,4'-Dichlorobiphenyl	0.01	0.001	1	1	100
2050-68-2	15	4,4'-Dichlorobiphenyl	0.05	0.005	5	5	500
37680-65-2	18	2,2',5-Trichlorobiphenyl	0.01	0.001	1	1	100
7012-37-5	28	2,4,4'-Trichlorobiphenyl	0.01	0.001	10	10	100
16606-02-3	31	2,4',5-trichlorobiphenyl	0.01	0.001	10	10	100
38444-90-5	37	3,4,4'-Trichlorobiphenyl	0.01	0.001	1	1	100
41464-39-5	44	2,2',3,5'-Tetrachlorobiphenyl	0.01	0.001	1	1	100
70362-45-7	45	2,2',3,6-Tetrachlorobiphenyl	0.01	0.001	1	1	100
41464-40-8	49	2,2',4,5'-Tetrachlorobiphenyl	0.01	0.001	1	1	100
35693-99-3	52	2,2',5,5'-Tetrachlorobiphenyl	0.01	0.001	1	1	100
41464-43-1	56	2,3,3',4'-Tetrachlorobiphenyl	0.01	0.001	1	1	100
32598-10-0	66	2,3',4,4'-Tetrachlorobiphenyl	0.01	0.001	1	1	100
32598-11-1	70	2,3',4',5-Tetrachlorobiphenyl	0.01	0.001	1	1	100
32690-93-0	74	2,4,4',5-Tetrachlorobiphenyl	0.01	0.001	1	1	100
32598-13-3	77	3,3',4,4'-Tetrachlorobiphenyl	0.01	0.001	1	1	100
70362-50-4	81	3,4,4',5-Tetrachlorobiphenyl	0.01	0.001	1	1	100
38380-02-8	87	2,2',3,4,5'-Pentachlorobiphenyl	0.01	0.001	1	1	100
68194-07-0	90	2,2',3,4',5-Pentachlorobiphenyl	0.01	0.001	1	1	100
38379-99-6	95	2,2',3,5',6-Pentachlorobiphenyl	0.01	0.001	1	1	100
38380-01-7	99	2,2',4,4',5-Pentachlorobiphenyl	0.01	0.001	1	1	100
37680-73-2	101	2,2',4,5,5'-Pentachlorobiphenyl	0.01	0.001	1	1	100
32598-14-4	105	2,3,3',4,4'-Pentachlorobiphenyl	0.01	0.001	1	1	100
38380-03-9	110	2,3,3',4',6-Pentachlorobiphenyl	0.01	0.001	1	1	100
74472-37-0	114	2,3,4,4',5-Pentachlorobiphenyl	0.01	0.001	1	1	100
74472-38-1	115	2,3,4,4',6-Pentachlorobiphenyl	0.01	0.001	1	1	100
31508-00-6	118	2,3',4,4',5-Pentachlorobiphenyl	0.01	0.001	1	1	100
56558-17-9	119	2,3',4,4',6-Pentachlorobiphenyl	0.01	0.001	1	1	100
65510-44-3	123	2,3',4,4',5'-Pentachlorobiphenyl	0.01	0.001	1	1	100
57465-28-8	126	3,3',4,4',5-Pentachlorobiphenyl	0.01	0.001	1	1	100
38380-07-3	128	2,2',3,3',4,4'-Hexachlorobiphenyl	0.01	0.001	1	1	100
38380-05-1	132	2,2',3,3',4,6'-Hexachlorobiphenyl	0.01	0.001	1	1	100
35065-28-2	138	2,2',3,4,4',5'-Hexachlorobiphenyl	0.01	0.001	1	1	100
52712-04-6	141	2,2',3,4,5,5'-Hexachlorobiphenyl	0.01	0.001	1	1	100
51908-16-8	146	2,2',3,4',5,5'-Hexachlorobiphenyl	0.01	0.001	1	1	100
38380-04-0	149	2,2',3,4',5',6-Hexachlorobiphenyl	0.01	0.001	1	1	100
52663-63-5	151	2,2',3,5,5',6-Hexachlorobiphenyl	0.01	0.001	1	1	100
35065-27-1	153	2,2',4,4',5,5'-Hexachlorobiphenyl	0.01	0.001	1	1	100
38380-08-4	156	2,3,3',4,4',5-Hexachlorobiphenyl	0.01	0.001	1	1	100
69782-90-7	157	2,3,3',4,4',5'-Hexachlorobiphenyl	0.01	0.001	1	1	100
74472-42-7	158	2,3,3',4,4',6-Hexachlorobiphenyl	0.01	0.001	1	1	100
52663-72-6	167	2,3',4,4',5,5'-Hexachlorobiphenyl	0.01	0.001	1	1	100
59291-65-5	168	2,3',4,4',5',6-Hexachlorobiphenyl	0.01	0.001	1	1	100
32774-16-6	169	3,3',4,4',5,5'-Hexachlorobiphenyl	0.01	0.001	1	1	100
35065-30-6	170	2,2',3,3',4,4',5-Heptachlorobiphenyl	0.01	0.001	1	1	100

Table 8-4-10
PCB Congeners by GC EPA Method 8082
Reporting Limits (RL), continued

Analyte			Reporting Limit				
CAS Number	BZ[1]	Compound Name	Aqueous (ug/L)	Low-level Aqueous (ug/L)	Solid (ug/kg, Dry)	Tissue (ug/kg)	Waste ug/kg
38411-25-5	174	2,2',3,3',4,5,6'-Heptachlorobiphenyl	0.01	0.001	1	1	100
52663-70-4	177	2,2',3,3',4,5',6'-Heptachlorobiphenyl	0.01	0.001	1	1	100
35065-29-3	180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	0.01	0.001	1	1	100
52663-69-1	183	2,2',3,4,4',5',6-Heptachlorobiphenyl	0.01	0.001	1	1	100
74472-48-3	184	2,2',3,4,4',6,6'-Heptachlorobiphenyl	0.01	0.001	1	1	100
52663-68-0	187	2,2',3,4',5,5',6-Heptachlorobiphenyl	0.01	0.001	1	1	100
39635-31-9	189	2,3,3',4,4',5,5'-Heptachlorobiphenyl	0.01	0.001	1	1	100
41411-64-7	190	2,3,3',4,4',5,6-Heptachlorobiphenyl	0.01	0.001	1	1	100
35694-08-7	194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	0.01	0.001	1	1	100
52663-78-2	195	2,2',3,3',4,4',5,6-Octachlorobiphenyl	0.01	0.001	1	1	100
40186-71-8	200	2,2',3,3',4,5',6,6'-Octachlorobiphenyl	0.01	0.001	1	1	100
52663-75-9	201	2,2',3,3',4,5,5',6'-Octachlorobiphenyl	0.01	0.001	1	1	100
2136-99-4	202	2,2',3,3',5,5',6,6'-Octachlorobiphenyl	0.01	0.001	1	1	100
40186-72-9	206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	0.01	0.001	1	1	100
52663-79-3	207	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	0.01	0.001	1	1	100
52663-77-1	208	2,2',3,3',4',5,5',6,6'-Nonachlorobiphenyl	0.01	0.001	1	1	100
2051-24-3	209	2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	0.01	0.001	1	1	100

Table 8-4-11
Fixed Gases by GC
Method D1946-90
Reporting Limits

Analyte	CAS Number	RL (%)
Carbon Dioxide	124-38-9	0.1
Carbon Monoxide	630-08-0	0.1
Methane	74-82-8	0.08
Nitrogen	7727-37-9	0.1
Oxygen	7782-44-7	0.1
Ethylene	74-85-1	0.1
Ethane	74-84-0	0.1

Table 8-4-12
Total Chromatographable Organics/Gravimetric Analysis (TCO Grav)
Method TCO/Grav
Reporting Limits

Analyte	Combined Train Fractions RL (mg)
Total Chromatographable Organics	0.15
Gravimetric Analysis	1.5

Table 8-4-13
Method 0040 Condensate
Reporting Limits

Analyte	Whole Sample RL* (ng)
C4	40
C5	40
C6	40
C7	40

* Based on 40 ml sample volume.

Table 8-4-14
Explosives by HPLC
EPA Method 8330
Reporting Limits

Analyte	CAS Number	Water RL, SPE[1] (µg/L)	Soil RL (mg/Kg, dry weight)[2]
HMX	2691-41-0	0.20	0.20
RDX	121-82-4	0.20	0.20
1,3,5-Trinitrobenzene	99-35-4	0.20	0.20
1,3-Dinitrobenzene	99-65-0	0.20	0.20
Tetryl	479-48-8	0.20	0.20
Nitrobenzene	98-95-3	0.20	0.20
2,4,6-Trinitrotoluene	118-96-7	0.20	0.20
2,6-Dinitrotoluene	606-20-2	0.20	0.20
2,4-Dinitrotoluene	121-14-2	0.20	0.20
2-Nitrotoluene	88-72-2	0.60	0.60
4-Nitrotoluene	99-99-0	0.60	0.60
3-Nitrotoluene	99-08-1	0.50	0.50
4-Amino-2,6-Dinitrotoluene	1946-51-0	0.30	0.50
2-Amino-4,6-Dinitrotoluene	35572-78-2	0.20	0.30
Nitroglycerin	55-63-0	2.5	2.5
PETN	78-11-5	2.5	2.5

[1] SPE = Solid phase extraction
[2] Soil samples are analyzed on a dry weight basis.

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-15
Nitroglycerin by HPLC
EPA Method 8332
Reporting Limits

Analyte	CAS Number	Water RL (µg/L)	Soil RL (mg/Kg dry weight) (1)
Nitroglycerin	55-63-0	500	2.5

(1) Soil samples are analyzed on a dry weight basis.

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-16
Volatiles by GCMS EPA Method 8260B
Water Reporting Limits (RL)

Analyte	CAS Number	Water RL (µg/L)
Acetone	67-64-1	10
Benzene	71-43-2	1.0
Bromobenzene	108-86-1	1.0
Bromochloromethane	74-97-5	1.0
Bromodichloromethane	75-27-4	1.0
Bromoform	75-25-2	1.0
Bromomethane	74-83-9	2.0
2-Butanone	78-93-3	5.0
n-Butylbenzene	104-51-8	1.0
sec-Butylbenzene	135-98-8	1.0
tert-Butylbenzene	98-06-6	1.0
Carbon Disulfide	75-15-0	1.0
Carbon Tetrachloride	56-23-5	1.0
Chlorobenzene	108-90-7	1.0
Chlorodibromomethane	124-48-1	1.0
Chloroethane	75-00-3	2.0
Chloroform (Trichloromethane)	67-66-3	1.0
Chloromethane	74-87-3	2.0
2-Chlorotoluene	95-49-8	1.0
4-Chlorotoluene	106-43-4	1.0
1,2-Dibromo-3-chloropropane	96-12-8	2.0
1,2-Dibromoethane	106-93-4	1.0
Dibromomethane	74-95-3	1.0
1,2-Dichlorobenzene	95-50-1	1.0
1,3-Dichlorobenzene	541-73-1	1.0
1,4-Dichlorobenzene	106-46-7	1.0

Table 8-4-16
Volatiles by GCMS EPA Method 8260B
Water Reporting Limits (RL)
(continued)

Analyte	CAS Number	Water RL (µg/L)
Dichlorodifluoromethane	75-71-8	2.0
1,1-Dichloroethane	75-34-3	1.0
1,2-Dichloroethane	107-06-2	1.0
1,1-Dichloroethene	75-35-4	1.0
cis-1,2-Dichloroethene	156-59-2	1.0
trans-1,2-Dichloroethene	156-60-5	1.0
1,2-Dichloroethene (total)	540-59-0	1.0
1,2-Dichloropropane	78-87-5	1.0
1,3-Dichloropropane	142-28-9	1.0
2,2-Dichloropropane	594-20-7	1.0
1,1-Dichloropropene	563-58-6	1.0
cis-1,3-Dichloropropene	10061-01-5	1.0
trans-1,3-Dichloropropene	10061-01-5	1.0
Ethyl Benzene	100-41-4	1.0
Hexachlorobutadiene	87-68-3	1.0
2-Hexanone	591-78-6	5.0
Isopropylbenzene	98-82-8	1.0
p-Isopropyltoluene	99-87-6	1.0
Methylene Chloride	75-09-2	2.0
4-methyl-2-pentanone	108-10-1	5.0
Naphthalene	91-20-3	1.0
n-Propylbenzene	103-65-1	1.0
Styrene	100-42-5	1.0
1,1,1,2-Tetrachloroethane	630-20-6	1.0
1,1,2,2-Tetrachloroethane	79-34-5	1.0

Table 8-4-16
Volatiles by GCMS EPA Method 8260B
Water Reporting Limits (RL)
(continued)

Analyte	CAS Number	Water RL (µg/L)
Tetrachloroethene	127-18-4	1.0
Toluene	108-88-3	1.0
1,2,3-Trichlorobenzene	87-61-6	1.0
1,2,4-Trichlorobenzene	120-82-1	1.0
1,1,1-Trichloroethane	71-55-6	1.0
1,1,2-Trichloroethane	79-00-5	1.0
Trichloroethene	79-01-6	1.0
Trichlorofluoromethane	75-69-4	2.0
1,2,3-Trichloropropane	96-18-4	1.0
1,2,4-Trimethylbenzene	95-63-6	1.0
1,3,5-Trimethylbenzene	108-67-8	1.0
Vinyl chloride	75-01-4	2.0
o-Xylene	95-47-6	1.0
p-/m-Xylenes	136777-61-2	2.0
Xylenes (total)	1330-20-7	3.0

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-17
Volatiles by GCMS EPA Method 8260B
Low Level and Medium Level Encore Soil Samples
Reporting Limits (RL)

Analyte	CAS Number	Low Level RL[1] (µg/Kg)	Medium Level RL[1] (µg/Kg)
Acetone	67-64-1	20	1000
Benzene	71-43-2	5.0	250
Bromobenzene	108-86-1	5.0	250
Bromochloromethane	74-97-5	5.0	250
Bromodichloromethane	75-27-4	5.0	250
Bromoform	75-25-2	5.0	250
Bromomethane	74-83-9	10	500
2-Butanone	78-93-3	20	1000
n-Butylbenzene	104-51-8	5.0	250
sec-Butylbenzene	135-98-8	5.0	250
tert-Butylbenzene	98-06-6	5.0	250
Carbon Disulfide	75-15-0	5.0	250
Carbon Tetrachloride	56-23-5	5.0	250
Chlorobenzene	108-90-7	5.0	250
Chlorodibromomethane	124-48-1	5.0	250
Chloroethane	75-00-3	10	500
Chloroform (Trichloromethane)	67-66-3	5.0	250
Chloromethane	74-87-3	10	500
2-Chlorotoluene	95-49-8	5.0	250
4-Chlorotoluene	106-43-4	5.0	250
1,2-Dibromo-3-chloropropane	96-12-8	10	500
1,2-Dibromoethane	106-93-4	5.0	250
Dibromomethane	74-95-3	5.0	250
1,2-Dichlorobenzene	95-50-1	5.0	250

Table 8-4-17
Volatiles by GCMS EPA Method 8260B
Low Level and Medium Level Encore Soil Samples
Reporting Limits (RL)
(continued)

Analyte	CAS Number	Low Level RL[1] (µg/Kg)	Medium Level RL[1] (µg/Kg)
1,3-Dichlorobenzene	541-73-1	5.0	250
1,4-Dichlorobenzene	106-46-7	5.0	250
Dichlorodifluoromethane	75-71-8	10	500
1,1-Dichloroethane	75-34-3	5.0	250
1,2-Dichloroethane	107-06-2	5.0	250
1,1-Dichloroethene	75-35-4	5.0	250
cis-1,2-Dichloroethene	156-59-2	5.0	250
trans-1,2-Dichloroethene	156-60-5	5.0	250
1,2-Dichloroethene (total)	540-59-0	5.0	250
1,2-Dichloropropane	78-87-5	5.0	250
1,3-Dichloropropane	142-28-9	5.0	250
2,2-Dichloropropane	594-20-7	5.0	250
1,1-Dichloropropene	563-58-6	5.0	250
cis-1,3-Dichloropropene	10061-01-5	5.0	250
trans-1,3-Dichloropropene	10061-01-5	5.0	250
Ethyl Benzene	100-41-4	5.0	250
Hexachlorobutadiene	87-68-3	5.0	250
2-Hexanone	591-78-6	20	1000
Isopropylbenzene	98-82-8	5.0	250
p-Isopropyltoluene	99-87-6	5.0	250
4-Methyl-2-pentanone	108-10-1	20	1000
Methylene Chloride	75-09-2	5.0	250
Naphthalene	91-20-3	5.0	250

Table 8-4-17
Volatiles by GCMS EPA Method 8260B
Low Level and Medium Level Encore Soil Samples
Reporting Limits (RL)
(continued)

Analyte	CAS Number	Low Level RL[(1)] (µg/Kg)	Medium Level RL[(1)] (µg/Kg)
n-Propylbenzene	103-65-1	5.0	250
Styrene	100-42-5	5.0	250
1,1,1,2-Tetrachloroethane	630-20-6	5.0	250
1,1,2,2-Tetrachloroethane	79-34-5	5.0	250
Tetrachloroethene	127-18-4	5.0	250
Toluene	108-88-3	5.0	250
1,2,3-Trichlorobenzene	87-61-6	5.0	250
1,2,4-Trichlorobenzene	120-82-1	5.0	250
1,1,1-Trichloroethane	71-55-6	5.0	250
1,1,2-Trichloroethane	79-00-5	5.0	250
Trichloroethene	79-01-6	5.0	250
Trichlorofluoromethane	75-69-4	10	500
1,2,3-Trichloropropane	96-18-4	5.0	250
1,2,4-Trimethylbenzene	95-63-6	5.0	250
1,3,5-Trimethylbenzene	108-67-8	5.0	250
Vinyl chloride	75-01-4	10	500
o-Xylene	95-47-6	5.0	250
p-/m-Xylenes	136777-61-2	10	500
Xylenes (total)	1330-20-7	15	750

(1) Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-18
Volatile Organics by GCMS EPA Method TO-14A/TO-15
Reporting Limits (RL)

Analyte	CAS Number	Air RL ppb (V/V)
Dichlorodifluoromethane	75-71-8	0.20
Chlorodifluoromethane	75-45-6	0.20
1,2-Dichlorotetrafluoroethane	76-14-2	0.20
Chloromethane	74-87-3	0.50
Vinyl chloride	75-01-4	0.20
n-Butane	106-97-8	0.40
1,3-Butadiene	106-99-0	0.40
Bromomethane	74-83-9	0.20
Chloroethane	75-00-3	0.20
Trichlorofluoromethane	75-69-4	0.20
Pentane	109-66-0	1.0
1,1-Dichloroethene	75-35-4	0.20
1,1,2-Trichlorotrifluoroethane	76-13-1	0.20
Carbon disulfide	75-15-0	0.50
3-Chloropropene	107-05-1	0.20
Methylene Chloride	75-09-2	0.50
trans-1,2-Dichloroethene	156-60-5	0.20
n-Hexane	110-54-3	0.50
1,1-Dichloroethane	75-34-3	0.20
cis-1,2-Dichloroethene	156-59-2	0.20
Chloroform	67-66-3	0.20
1,1,1-Trichloroethane	71-55-6	0.20
Cyclohexane	110-82-7	0.50
Carbon Tetrachloride	56-23-5	0.20
Benzene	71-43-2	0.20
1,2-Dichloroethane	107-06-2	0.20
n-Heptane	142-82-5	0.50

Table 8-4-18
Volatile Organics by GCMS EPA Method TO-14A/TO-15
Reporting Limits (RL)
(continued)

Analyte	CAS Number	Air RL ppb (V/V)
Trichloroethene	79-01-6	0.20
1,2-Dichloropropane	78-87-5	0.20
Dibromomethane	74-95-3	0.40
Bromodichloromethane	75-27-4	0.20
cis-1,3-Dichloropropene	10061-01-5	0.20
Toluene	108-88-3	0.20
n-Octane	111-65-9	0.40
trans-1,3-Dichloropropene	10061-01-5	0.20
1,1,2-Trichloroethane	79-00-5	0.20
Tetrachloroethene	127-18-4	0.20
Dibromochloromethane	124-48-1	0.20
1,2-Dibromoethane	106-93-4	0.20
Chlorobenzene	108-90-7	0.20
Ethyl Benzene	100-41-4	0.20
p-/m-Xylenes	136777-61-2	0.20
Nonane	111-84-2	0.50
o-Xylene	95-47-6	0.20
Styrene	100-42-5	0.20
Bromoform	75-25-2	0.20
Cumene	98-82-8	0.40
1,1,2,2-Tetrachloroethane	79-34-5	0.20
n-Propylbenzene	103-65-1	0.40
1,3,5-Trimethylbenzene	108-67-8	0.20
n-Decane	124-18-5	1.0
alpha-Methlystyrene	98-83-9	0.40
1,2,4-Trimethlybenzene	95-63-6	0.20

Table 8-4-18
Volatile Organics by GCMS EPA Method TO-14A/TO-15
Reporting Limits (RL)
(continued)

Analyte	CAS Number	Air RL ppb (V/V)
1,3-Dichlorobenzene	541-73-1	0.20
1,4-Dichlorobenzene	106-46-7	0.20
Benzyl Chloride	100-44-7	0.40
1,2-Dichlorobenzene	95-50-1	0.20
n-Undecane	1120-21-4	1.0
n-Dodecane	112-40-3	1.0
1,2,4-Trichlorobenzene	120-82-1	1.0
Hexachlorobutadiene	87-68-3	1.0
Naphthalene	91-20-3	0.50
Methanol	67-56-7	10
Ethyl ether	60-29-7	2.0
Acetone	67-64-1	5.0
Acrylonitrile	107-13-1	2.0
Vinyl acetate	108-05-4	1.0
2-Butanone	78-93-9	1.0
1-Butanol	71-36-3	2.0
4-Methyl-2-pentanone	108-10-1	0.50
2-Hexanone	591-78-6	0.50
Methyl-tert-butyl ether	1634-04-4	1.0
Acrolein	107-02-8	0.80
Acetonitrile	75-05-8	1.0

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-19
Volatiles by GCMS EPA Method 0031/5041A (VOST)
Reporting Limits (RL)

Analyte	CAS Number	VOST RL ug
Acetone	67-64-1	0.100
Benzene	71-43-2	0.025
Bromobenzene	108-86-1	0.025
Bromochloromethane	74-97-5	0.025
Bromodichloromethane	75-27-4	0.025
Bromoform	75-25-2	0.025
Bromomethane	74-83-9	0.050
2-Butanone	78-93-9	0.100
n-Butylbenzene	104-51-8	0.025
sec-Butylbenzene	135-98-8	0.025
tert-Butylbenzene	98-06-6	0.025
Carbon disulfide	75-15-0	0.025
Carbon Tetrachloride	56-23-5	0.025
Chlorobenzene	108-90-7	0.025
Chlorodibromomethane	124-48-1	0.025
Chloroethane	75-003	0.050
Chloroform	67-66-3	0.025
Chloromethane	74-87-3	0.050
2-Chlorotoluene	95-49-8	0.025
4-Chlorotoluene	106-43-4	0.025
1,2-Dibromoethane	106-93-4	0.025
Dibromomethane	74-95-3	0.025
1,2-Dichlorobenzene	95-50-1	0.025
1,3-Dichlorobenzene	541-73-1	0.025

Table 8-4-19
Volatiles by GCMS EPA Method 0031/5041A (VOST)
Reporting Limits (RL)
(continued)

Analyte	CAS Number	VOST RL ug
1,4-Dichlorobenzene	106-46-7	0.025
Dichlorodifluoromethane	75-71-8	0.050
1,1-Dichloroethane	75-34-3	0.025
1,2-Dichloroethane	107-06-2	0.025
cis-1,2-Dichloroethene	156-59-2	0.025
trans-1,2-Dichloroethene	156-60-5	0.025
1,1-Dichloroethene	75-35-4	0.025
1,2-Dichloropropane	78-87-5	0.025
1,3-Dichloropropane	142-28-9	0.025
2,2-Dichloropropane	594-20-7	0.025
cis-1,3-Dichloropropene	10061-01-5	0.025
trans-1,3-Dichloropropene	10061-01-5	0.025
1,1-Dichloropropene	563-58-6	0.025
Ethylbenzene	100-41-4	0.025
Hexachlorobutadiene	87-68-3	0.025
2-Hexanone	591-78-6	0.100
Isopropylbenzene	98-82-8	0.025
p-Isopropyltoluene	99-87-6	0.025

Table 8-4-19
Volatiles by GCMS EPA Method 0031/5041A (VOST)
Reporting Limits (RL)
(continued)

Analyte	CAS Number	VOST RL ug
Methylene chloride	75-09-2	0.025
4-Methyl-2-pentanone	108-10-1	0.100
Naphthalene	91-20-3	0.025
1,2-Dibromo-3-chloropropane	96-12-8	0.050
n-Propylbenzene	103-65-1	0.025
Styrene	100-42-5	0.025
1,1,1,2-Tetrachloroethane	630-20-6	0.025
1,1,2,2-Tetrachloroethane	79-34-5	0.025
Tetrachloroethene	127-18-4	0.025
Toluene	108-88-3	0.025
1,2,3-Trichlorobenzene	87-61-6	0.025
1,2,4-Trichlorobenzene	120-82-1	0.025
1,1,1-Trichloroethane	71-55-6	0.025
1,1,2-Trichloroethane	79-00-5	0.025
Trichloroethene	79-01-6	0.025
Trichlorofluoromethane	75-69-4	0.050
1,2,3-Trichloropropane	96-18-4	0.025
1,2,4-Trimethylbenzene	95-63-6	0.025
1,3,5-Trimethylbenzene	108-67-8	0.025
Vinyl chloride	75-01-4	0.050
m- &p-xylene	136777-61-2	0.050
o-xylene	95-47-6	0.025

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request.

Table 8-4-20
Semivolatile Organics by GCMS Method 8270C
Reporting Limits (RL)

Analyte	CAS Number	Water RL (µg/L)	Soil RL [1] (µg/Kg)
Acenaphthene	83-32-9	10	330
Acenaphthylene	208-96-8	10	330
Anthracene	120-12-7	10	330
Benzo(a)anthracene	56-55-3	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(g,h,i)perylene	191-24-2	10	330
Benzo(a)pyrene	50-32-8	10	330
bis(2-Chloroethoxy)methane	111-91-1	10	330
bis(2-Chloroethyl)ether	111-44-4	10	330
bis(2-chloroisopropyl)ether	108-60-1	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4-Bromophenyl-phenylether	101-55-3	10	330
Butyl benzyl phthalate	85-68-7	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	10	330
4-Chloro-3-Methylphenol	59-50-7	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Chlorophenol	95-57-8	10	330
4-Chlorophenyl phenylether	7005-72-36	10	330
Chrysene	218-01-9	10	330
Dibenzo(a,h)anthracene	53-70-3	10	330
Dibenzofuran	132-64-9	10	330
Di-n-butylphthalate	84-74-2	10	330

Table 8-4-20
Semivolatile Organics by GCMS Method 8270C
Reporting Limits (RL)
(continued)

Analyte	CAS Number	Water RL (µg/L)	Soil RL [1] (µg/Kg)
1,2-Dichlorobenzene	95-50-1	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	10	330
2,4-Dimethylphenol	105-67-9	10	330
Dimethyl Phthalate	131-11-3	10	330
4,6-Dinitro-2-Methylphenol	534-52-1	50	1600
2,4-Dinitrophenol	51-28-5	50	1600
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Di-n-Octylphthalate	117-84-0	10	330
Fluoranthene	206-44-0	10	330
Fluorene	86-73-7	10	330
Hexachlorobenzene	118-74-1	10	330
Hexachlorobutadiene	87-68-3	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
Hexachloroethane	67-72-1	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Isophorone	78-59-1	10	330
2-Methylnaphthalene	91-57-6	10	330
2-Methylphenol	95-48-7	10	330
4-Methylphenol	106-44-5	10	330

Table 8-4-20
Semivolatile Organics by GCMS Method 8270C
Reporting Limits (RL)
(continued)

Analyte	CAS Number	Water RL (µg/L)	Soil RL [1] (µg/Kg)
Naphthalene	91-20-3	10	330
2-Nitroaniline	88-74-4	50	1600
3-Nitroaniline	99-09-2	50	1600
4-Nitroaniline	100-01-6	50	1600
Nitrobenzene	98-95-1	10	330
2-Nitrophenol	88-75-5	10	330
4-Nitrophenol	100-02-7	50	1600
n-Nitrosodiphenylamine	86-30-6	10	330
n-Nitroso-di-n-propylamine	621-64-7	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Phenol	108-95-2	10	330
Pyrene	129-00-0	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2,4,6-Trichlorophenol	88-06-2	10	330

[1] Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

NOTE: Method detection limits (MDLs) are subject to updating. Current MDLs are available upon request. RLs and MDLs for source air testing are also available upon request.

Table 8-4-21
Semivolatile Organics by GCMS Method TO-13A
Reporting Limits (RL)

Analyte	CAS Number	Air RL (µg)
Acenaphthene	83-32-9	5
Acenaphthylene	208-96-8	5
Anthracene	120-12-7	5
Benzo(a)anthracene	56-55-3	5
Benzo(a)pyrene	50-32-8	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(g,h,i)perylene	191-24-2	5
Benzo(k)fluoranthene	207-08-9	5
Chrysene	218-01-9	5
Dibenzo(a,h)anthracene	53-70-3	5
Fluoranthene	206-44-0	5
Fluorene	86-73-7	5
Indeno(1,2,3-cd)pyrene	193-39-5	5
Naphthalene	91-20-3	5
Phenanthrene	85-01-8	5
Pyrene	129-00-0	5

Table 8-4-22
Modified Method 5 Train (0010/3542/8270C)
Reporting Limits (RL)

Analyte	CAS Number	Filter/Rinse RLs (ug)	Back Half RLs (ug)	Condensate RLs (ug)
Acenaphthene	83-32-9	10	10	10
Acenaphthylene	208-96-8	10	10	10
Acetophenone	98-86-2	10	10	10
Aniline	62-53-3	20	20	20
Anthracene	120-12-7	10	10	10
Benzidine	92-87-5	100	100	100
Benzoic acid	65-85-0	100	100	50
Benzo(a)anthracene	56-55-3	10	10	10
Benzo(a)pyrene	50-32-8	10	10	10
Benzo(b)fluoranthene	205-99-2	10	10	10
Benzo(ghi)perylene	191-24-2	10	10	10
Benzo(k)fluoranthene	207-08-9	10	10	10
Benzyl alcohol	100-51-6	100	100	10
bis(2-Chloroethoxy)methane	111-91-1	10	10	10
bis(2-Chloroethyl) ether	111-44-4	10	10	10
bis(2-Ethylhexyl) phthalate	117-81-7	10	20	10
4-Bromophenyl phenyl ether	101-55-3	10	10	10
Butyl benzyl phthalate	85-68-7	10	10	10
Carbazole	86-74-8	10	10	10
4-Chloro-3-methylphenol	59-50-7	10	10	10
4-Chloroaniline	106-47-8	10	20	10
2-Chloronaphthalene	91-58-7	10	10	10
2-Chlorophenol	95-57-8	10	10	10
4-Chlorophenyl phenyl ether	7005-72-36	10	10	10
Chrysene	218-01-9	10	10	10
Di-n-butyl phthalate	84-74-2	10	20	10
Di-n-octyl phthalate	117-84-0	10	10	10

Table 8-4-22
Modified Method 5 Train (0010/3542/8270C)
Reporting Limits (RL), continued

Analyte	CAS Number	Filter/Rinse RLs (ug)	Back Half RLs (ug)	Condensate RLs (ug)
Dibenz(a,h)anthracene	53-70-3	10	10	10
Dibenzofuran	132-64-9	10	10	10
1,2-Dichlorobenzene	95-50-1	10	10	10
1,3-Dichlorobenzene	541-73-1	10	10	10
1,4-Dichlorobenzene	106-46-7	10	10	10
3,3'-Dichlorobenzidine	91-94-1	50	50	50
2,4-Dichlorophenol	120-83-2	10	10	10
Diethyl phthalate	84-66-2	10	10	10
Dimethyl phthalate	84-66-2	10	10	10
2,4-Dimethylphenol	105-67-9	10	10	10
4,6-Dinitro-2-methylphenol	534-52-1	50	50	50
2,4-Dinitrophenol	51-28-5	50	50	50
2,4-Dinitrotoluene	121-14-2	10	10	10
2,6-Dinitrotoluene	606-20-2	10	10	10
1,2-Diphenylhydrazine	122-66-7	10	10	10
Fluoranthene	206-44-0	10	10	10
Fluorene	86-73-7	10	10	10
Hexachlorocyclopentadiene	77-47-4	50	50	50
Hexachlorobenzene	118-74-1	10	10	10
Hexachlorobutadiene	87-68-3	10	10	10
Hexachloroethane	67-72-1	10	10	10
Indeno(1,2,3-cd)pyrene	193-39-5	10	10	10
Isophorone	78-59-1	10	10	10
2-Methylnaphthalene	91-57-6	10	10	10
2-Methylphenol	95-48-7	10	10	10
3-Methylphenol & 4-Methylphenol	108-39-4 & 106-44-5	10	10	10
N-Nitrosodi-n-propylamine	621-64-7	10	10	10

Table 8-4-22
Modified Method 5 Train (0010/3542/8270C)
Reporting Limits (RL), continued

Analyte	CAS Number	Filter/Rinse RLs (ug)	Back Half RLs (ug)	Condensate RLs (ug)
N-Nitrosodimethylamine	62-75-9	10	10	10
N-Nitrosodiphenylamine	86-30-6	10	10	10
Naphthalene	91-20-3	10	10	10
2-Nitroaniline	88-74-4	50	50	50
3-Nitroaniline	99-09-2	50	50	50
4-Nitroaniline	100-01-6	50	50	50
Nitrobenzene	98-95-1	10	10	10
2-Nitrophenol	88-75-5	10	10	10
4-Nitrophenol	100-02-7	50	50	50
2,2'-Oxybis(1-Chloropropane)*	108-60-1	10	10	10
Pentachlorobenzene	608-93-5	10	10	10
Pentachloronitrobenzene	82-68-8	10	50	10
Pentachlorophenol	87-86-5	50	50	50
Phenanthrene	85-01-8	10	10	10
Phenol	108-95-2	10	10	10
Pyrene	129-00-0	10	10	10
Pyridine	110-86-1	20	20	20
1,2,4-Trichlorobenzene	120-82-1	10	10	10
2,4,5-Trichlorophenol	95-95-4	10	10	10
2,4,6-Trichlorophenol	88-06-2	10	10	10

*bis(2-chloro)isopropylether

Table 8-4-23
Dioxins/Furans by HRGC/HRMS EPA Method 1613B
Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (pg/L)	Soil RL [2] (pg/g)	Whole Sample RL (pg)
2,3,7,8-TCDD	1746-01-6	10	1.0	10
Total-TCDD	41903-57-5	10	1.0	10
1,2,3,7,8-PeCDD	40321-76-4	50	5.0	50
Total-PeCDD	36088-22-9	50	5.0	50
1,2,3,4,7,8-HxCDD	39227-28-6	50	5.0	50
1,2,3,6,7,8-HxCDD	57653-85-7	50	5.0	50
1,2,3,7,8,9-HxCDD	19408-74-3	50	5.0	50
Total-HxCDD	34465-46-8	50	5.0	50
1,2,3,4,6,7,8-HPCDD	35822-46-9	50	5.0	50
Total-HPCDD	37871-00-4	50	5.0	50
OCDD	3268-87-9	100	10	100
2,3,7,8-TCDF	51207-31-9	10	1.0	10
Total-TCDF	55722-27-5	10	1.0	10
1,2,3,7,8-PECDF	57117-41-6	50	5.0	50
2,3,4,7,8-PECDF	57117-31-4	50	5.0	50
Total-PECDF	30402-15-4	50	5.0	50
1,2,3,4,7,8-HXCDF	70648-26-9	50	5.0	50
1,2,3,6,7,8-HXCDF	57117-44-9	50	5.0	50
2,3,4,6,7,8-HXCDF	60851-34-5	50	5.0	50
1,2,3,7,8,9-HXCDF	72918-21-9	50	5.0	50
Total-HXCDF	55684-94-1	50	5.0	50
1,2,3,4,6,7,8-HPCDF	67562-39-4	50	5.0	50
1,2,3,4,7,8,9-HPCDF	55673-89-7	50	5.0	50
Total-HPCDF	38998-75-3	50	5.0	50
OCDF	39001-02-0	100	10	100

[1] The reporting limit is equivalent to the concentration of the lowest calibration standard, assuming all method-specific sample weights and volumes have been employed (i.e., 1 L for waters, 10 gr for soil, entire sample for whole sample RL, e.g., wipe, sampling module)
[2] Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

Table 8-4-24
Dioxins/Furans by HRGC/HRMS EPA Methods 8290, 23/8290 and 0023A/8290
Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (pg/L)	Soil/Tissue RL [2] (pg/g)	Whole Sample RL (pg)
2,3,7,8-TCDD	1746-01-6	10	1.0	10
Total-TCDD	41903-57-5	10	1.0	10
1,2,3,7,8-PeCDD	40321-76-4	50	5.0	50
Total-PeCDD	36088-22-9	50	5.0	50
1,2,3,4,7,8-HxCDD	39227-28-6	50	5.0	50
1,2,3,6,7,8-HxCDD	57653-85-7	50	5.0	50
1,2,3,7,8,9-HxCDD	19408-74-3	50	5.0	50
Total-HxCDD	34465-46-8	50	5.0	50
1,2,3,4,6,7,8-HPCDD	35822-46-9	50	5.0	50
Total-HPCDD	37871-00-4	50	5.0	50
OCDD	3268-87-9	100	10	100
2,3,7,8-TCDF	51207-31-9	10	1.0	10
Total-TCDF	55722-27-5	10	1.0	10
1,2,3,7,8-PECDF	57117-41-6	50	5.0	50
2,3,4,7,8-PECDF	57117-31-4	50	5.0	50
Total-PECDF	30402-15-4	50	5.0	50
1,2,3,4,7,8-HXCDF	70648-26-9	50	5.0	50
1,2,3,6,7,8-HXCDF	57117-44-9	50	5.0	50
2,3,4,6,7,8-HXCDF	60851-34-5	50	5.0	50
1,2,3,7,8,9-HXCDF	72918-21-9	50	5.0	50
Total-HXCDF	55684-94-1	50	5.0	50
1,2,3,4,6,7,8-HPCDF	67562-39-4	50	5.0	50
1,2,3,4,7,8,9-HPCDF	55673-89-7	50	5.0	50
Total-HPCDF	38998-75-3	50	5.0	50
OCDF	39001-02-0	100	10	100

[1] The reporting limit is equivalent to the concentration of the lowest calibration standard, assuming all method-specific sample weights and volumes have been employed (i.e., 1 L for waters, 10 gr for soil, entire sample for whole sample RL, e.g., wipe, sampling module)
[2] Soil reporting limits are based on wet weight of sample and will be higher when converted to a dry weight basis.

Table 8-4-25
PCBs by HRGC/HRMS Method 1668A
Estimated Minimum Levels

Parameter	Water ng/L	Solids ng/g	Tissues ng/g	Solid Wastes ng/g	100 uL Extract ng/mL
PCB 1	0.040	0.01	0.01	0.01	1.0
PCB 2	0.040	0.01	0.01	0.01	1.0
PCB 3	0.040	0.01	0.01	0.01	1.0
PCB 4	0.060	0.02	0.02	0.02	2.0
PCB 5	0.040	0.01	0.01	0.01	1.0
PCB 6	0.040	0.01	0.01	0.01	1.0
PCB 7	0.040	0.01	0.01	0.01	1.0
PCB 8	0.060	0.02	0.02	0.02	2.0
PCB 9	0.040	0.01	0.01	0.01	1.0
PCB 10	0.040	0.01	0.01	0.01	1.0
PCB 11	0.060	0.02	0.02	0.02	2.0
PCB 12/PCB 13	0.060	0.01	0.01	0.01	1.0
PCB 14	0.040	0.01	0.01	0.01	1.0
PCB 15	0.040	0.01	0.01	0.01	1.0
PCB 16	0.040	0.01	0.01	0.01	1.0
PCB 17	0.040	0.01	0.01	0.01	1.0
PCB 18/PCB 30	0.060	0.02	0.02	0.02	2.0
PCB 19	0.040	0.01	0.01	0.01	1.0
PCB 20/PCB 28	0.040	0.02	0.02	0.02	2.0
PCB 21/PCB33	0.040	0.01	0.01	0.01	1.0
PCB 22	0.040	0.01	0.01	0.01	1.0
PCB 23	0.040	0.01	0.01	0.01	1.0
PCB 24	0.040	0.01	0.01	0.01	1.0
PCB 25	0.040	0.01	0.01	0.01	1.0
PCB 26/PCB 29	0.040	0.01	0.01	0.01	1.0
PCB 27	0.040	0.01	0.01	0.01	1.0
PCB 31	0.040	0.02	0.02	0.02	2.0
PCB 32	0.040	0.01	0.01	0.01	1.0
PCB 34	0.040	0.01	0.01	0.01	1.0
PCB 35	0.040	0.01	0.01	0.01	1.0
PCB 36	0.040	0.01	0.01	0.01	1.0
PCB 37	0.040	0.01	0.01	0.01	1.0
PCB 38	0.040	0.01	0.01	0.01	1.0
PCB 39	0.040	0.01	0.01	0.01	1.0
PCB 40/PCB41/PCB 71	0.040	0.01	0.01	0.01	1.0
PCB 42	0.040	0.01	0.01	0.01	1.0
PCB 43/PCB 73	0.040	0.01	0.01	0.01	1.0
PCB 44/PCB 47/PCB 65	0.040	0.01	0.01	0.01	1.0
PCB 45/PCB 51	0.040	0.01	0.01	0.01	1.0
PCB 46	0.040	0.01	0.01	0.01	1.0
PCB 48	0.040	0.01	0.01	0.01	1.0
PCB 49/PCB 69	0.040	0.01	0.01	0.01	1.0
PCB 50/PCB 53	0.040	0.01	0.01	0.01	1.0
PCB 52	0.040	0.01	0.01	0.01	1.0
PCB 54	0.040	0.01	0.01	0.01	1.0
PCB 55	0.040	0.01	0.01	0.01	1.0
PCB 56	0.040	0.01	0.01	0.01	1.0
PCB 57	0.040	0.01	0.01	0.01	1.0
PCB 58	0.040	0.01	0.01	0.01	1.0
PCB 59/PCB 62/PCB 75	0.040	0.01	0.01	0.01	1.0
PCB 60	0.040	0.01	0.01	0.01	1.0
PCB 61/PCB 70/PCB 74/PCB 76	0.040	0.02	0.02	0.02	2.0
PCB 63	0.040	0.01	0.01	0.01	1.0
PCB 64	0.040	0.01	0.01	0.01	1.0
PCB 66	0.040	0.01	0.01	0.01	1.0
PCB 67	0.040	0.01	0.01	0.01	1.0
PCB 68	0.040	0.01	0.01	0.01	1.0
PCB 72	0.040	0.01	0.01	0.01	1.0

Table 8-4-25
PCBs by HRGC/HRMS Method 1668A
Estimated Minimum Levels, continued

Parameter	Water ng/L	Solids ng/g	Tissues ng/g	Solid Wastes ng/g	100 uL Extract ng/mL
PCB 77	0.040	0.01	0.01	0.01	1.0
PCB 78	0.040	0.01	0.01	0.01	1.0
PCB 79	0.040	0.01	0.01	0.01	1.0
PCB 80	0.040	0.01	0.01	0.01	1.0
PCB 81	0.040	0.01	0.01	0.01	1.0
PCB 82	0.040	0.01	0.01	0.01	1.0
PCB 83	0.040	0.01	0.01	0.01	1.0
PCB 84	0.040	0.01	0.01	0.01	1.0
PCB 85/PCB 116/PCB 117	0.040	0.01	0.01	0.01	1.0
PCB 86/PCB 87/PCB 97/PCB 109/PCB 119/PCB 125	0.040	0.01	0.01	0.01	1.0
PCB 88/PCB 91	0.040	0.01	0.01	0.01	1.0
PCB 89	0.040	0.01	0.01	0.01	1.0
PCB 90/PCB 101/PCB 113	0.040	0.01	0.01	0.01	1.0
PCB 92	0.040	0.01	0.01	0.01	1.0
PCB 93/PCB 100	0.040	0.01	0.01	0.01	1.0
PCB 94	0.040	0.01	0.01	0.01	1.0
PCB 95	0.040	0.01	0.01	0.01	1.0
PCB 96	0.040	0.01	0.01	0.01	1.0
PCB 98/PCB 102	0.040	0.01	0.01	0.01	1.0
PCB 99/PCB 112	0.040	0.01	0.01	0.01	1.0
PCB 103	0.040	0.01	0.01	0.01	1.0
PCB 104	0.040	0.01	0.01	0.01	1.0
PCB 105	0.040	0.01	0.01	0.01	1.0
PCB 106	0.040	0.01	0.01	0.01	1.0
PCB 107	0.040	0.01	0.01	0.01	1.0
PCB 108/PCB 124	0.040	0.01	0.01	0.01	1.0
PCB 110/PCB 115	0.040	0.01	0.01	0.01	1.0
PCB 111	0.040	0.01	0.01	0.01	1.0
PCB 114	0.040	0.01	0.01	0.01	1.0
PCB 118	0.040	0.01	0.01	0.01	1.0
PCB 120	0.040	0.01	0.01	0.01	1.0
PCB 121	0.040	0.01	0.01	0.01	1.0
PCB 122	0.040	0.01	0.01	0.01	1.0
PCB 123	0.040	0.01	0.01	0.01	1.0
PCB 126	0.040	0.01	0.01	0.01	1.0
PCB 127	0.040	0.01	0.01	0.01	1.0
PCB 128/PCB 166	0.040	0.01	0.01	0.01	1.0
PCB 129/PCB 138/PCB 163	0.040	0.01	0.01	0.01	1.0
PCB 130	0.040	0.01	0.01	0.01	1.0
PCB 131	0.040	0.01	0.01	0.01	1.0
PCB 132	0.040	0.01	0.01	0.01	1.0
PCB 133	0.040	0.01	0.01	0.01	1.0
PCB 134/PCB 143	0.040	0.01	0.01	0.01	1.0
PCB 135/PCB 151	0.040	0.01	0.01	0.01	1.0
PCB 136	0.040	0.01	0.01	0.01	1.0
PCB 137/PCB 164	0.040	0.01	0.01	0.01	1.0
PCB 139/PCB 140	0.040	0.01	0.01	0.01	1.0
PCB 141	0.040	0.01	0.01	0.01	1.0
PCB 142	0.040	0.01	0.01	0.01	1.0
PCB 144	0.040	0.01	0.01	0.01	1.0
PCB 145	0.040	0.01	0.01	0.01	1.0
PCB 146	0.040	0.01	0.01	0.01	1.0
PCB 147/PCB 149	0.040	0.01	0.01	0.01	1.0
PCB 148	0.040	0.01	0.01	0.01	1.0
PCB 150	0.040	0.01	0.01	0.01	1.0
PCB 152	0.040	0.01	0.01	0.01	1.0
PCB 153/PCB 168	0.040	0.01	0.01	0.01	1.0
PCB 154	0.040	0.01	0.01	0.01	1.0

Table 8-4-25
PCBs by HRGC/HRMS Method 1668A
Estimated Minimum Levels, continued

Parameter	Water ng/L	Solids ng/g	Tissues ng/g	Solid Wastes ng/g	100 uL Extract ng/mL
PCB 155	0.040	0.01	0.01	0.01	1.0
PCB 156/PCB 157	0.040	0.01	0.01	0.01	1.0
PCB 158	0.040	0.01	0.01	0.01	1.0
PCB 159	0.040	0.01	0.01	0.01	1.0
PCB 160	0.040	0.01	0.01	0.01	1.0
PCB 161	0.040	0.01	0.01	0.01	1.0
PCB 162	0.040	0.01	0.01	0.01	1.0
PCB 165	0.040	0.01	0.01	0.01	1.0
PCB 167	0.040	0.01	0.01	0.01	1.0
PCB 169	0.040	0.01	0.01	0.01	1.0
PCB 170	0.040	0.01	0.01	0.01	1.0
PCB 171/PCB 173	0.040	0.01	0.01	0.01	1.0
PCB 172	0.040	0.01	0.01	0.01	1.0
PCB 174	0.040	0.01	0.01	0.01	1.0
PCB 175	0.040	0.01	0.01	0.01	1.0
PCB 176	0.040	0.01	0.01	0.01	1.0
PCB 177	0.040	0.01	0.01	0.01	1.0
PCB 178	0.040	0.01	0.01	0.01	1.0
PCB 179	0.040	0.01	0.01	0.01	1.0
PCB 180/PCB 193	0.040	0.01	0.01	0.01	1.0
PCB 181	0.040	0.01	0.01	0.01	1.0
PCB 182	0.040	0.01	0.01	0.01	1.0
PCB 183/PCB 185	0.040	0.01	0.01	0.01	1.0
PCB 184	0.040	0.01	0.01	0.01	1.0
PCB 186	0.040	0.01	0.01	0.01	1.0
PCB 187	0.040	0.01	0.01	0.01	1.0
PCB 188	0.040	0.01	0.01	0.01	1.0
PCB 189	0.040	0.01	0.01	0.01	1.0
PCB 190	0.040	0.01	0.01	0.01	1.0
PCB 191	0.040	0.01	0.01	0.01	1.0
PCB 192	0.040	0.01	0.01	0.01	1.0
PCB 194	0.040	0.01	0.01	0.01	1.0
PCB 195	0.040	0.01	0.01	0.01	1.0
PCB 196	0.040	0.01	0.01	0.01	1.0
PCB 197/PCB 200	0.040	0.01	0.01	0.01	1.0
PCB 198/PCB 199	0.040	0.01	0.01	0.01	1.0
PCB 201	0.040	0.01	0.01	0.01	1.0
PCB 202	0.040	0.01	0.01	0.01	1.0
PCB 203	0.040	0.01	0.01	0.01	1.0
PCB 204	0.040	0.01	0.01	0.01	1.0
PCB 205	0.040	0.01	0.01	0.01	1.0
PCB 206	0.040	0.01	0.01	0.01	1.0
PCB 207	0.040	0.01	0.01	0.01	1.0
PCB 208	0.040	0.01	0.01	0.01	1.0
PCB 209	0.040	0.01	0.01	0.01	1.0

The estimated minimum level (EML) is defined as the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures. Deviations from the extraction amounts or final volumes listed results in corresponding changes in the actual sample ML. The EMLs provided are based on the mean plus 2 standard deviations for matrix-pooled historical blank data and calibration data obtained while performing EPA 1668A. The survey period was fourteen months, ending in February 2004.

Table 8-4-26
Polynuclear Aromatic Hydrocarbons by HRGC/LRMS
Reporting Limits (RLs)

Analyte	CAS Number	Water RL (ng/L)	Soil RL [(2)] (ng/g)	Biological RL (ng/g)	Air RL (ng/sample fraction)
Acenaphthene	83-32-9	10	1.0	1.0	20
Acenaphthylene	208-96-8	10	1.0	1.0	20
Anthracene	120-12-7	10	1.0	1.0	10
Benz[a]anthracene	56-55-3	10	1.0	1.0	10
Benzo[b]fluoranthene	205-99-2	10	1.0	1.0	10
Benzo[k]fluoranthene	207-08-9	10	1.0	1.0	10
Benzo[g.h,i]perylene	191-24-2	10	1.0	2.0	10
Benzo[a]pyrene	50-32-8	10	1.0	1.0	10
Benzo[e]pyrene	192-97-2	10	1.0	1.0	10
Biphenyl	92-52-4	10	1.0	1.0	20*
Chrysene	218-01-9	10	1.0	1.0	10
Dibenz[a,h]anthracene	53-70-3	10	1.0	1.0	10
2,6-Dimethylnaphthalene	581-42-0	10	2.0	2.0	20
Fluoranthene	206-44-0	10	1.0	1.0	10
Fluorene	86-73-7	10	1.0	1.0	10
Indeno[1,2,3-c,d]-pyrene	193-39-5	10	1.0	2.0	10
1-Methylnaphthalene	90-12-0	10	5.0	2.0	100
2-Methylnaphthalene	91-57-6	20	10	5.0	150
1-Methylphenanthrene	832-69-9	10	1.0	1.0	10
Naphthalene	91-20-3	50	20	10	400
Phenanthrene	85-01-8	10	1.0	1.0	20
Perylene	198-55-0	10	1.0	1.0	10
Pyrene	129-00-0	10	1.0	1.0	20
2,3,5-Trimethylnaphthalene	2245-38-7	10	2.0	1.0	10

* - The reporting limit listed for biphenyl cannot be supported for air train samples analyzed for both PAHs by this method and by EPA Method 8270 due to suspected interference from the 8270 surrogate spike

TABLE 8-5
Precision and Accuracy Measurements

Measurement	Definition
Accuracy	The degree of agreement of a measurement with an accepted reference or true value. The only true or known values in the laboratory are spiked samples. Expressed as laboratory control sample (LCS) percent recovery (% R): $$LCS\ \%\ Recovery = \frac{X}{t}\ x\ 100$$ where: X = observed concentration t = concentration of spike added Expressed as matrix spike/matrix spike duplicate (MS/MSD) sample percent recovery (% R): $$MS\ /\ MSD\ \%\ Recovery = \frac{X_s - X}{t}\ x\ 100$$ where: X_s = observed concentration in spiked sample X = observed concentration in unspiked sample t = concentration of spike added
Precision	The measure of analytical reproducibility of two values. Expressed as the relative percent difference (RPD) of two values. $$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$ where: X_1 = first observed concentration or recovery X_2 = second observed concentration or recovery Can also be expressed as the relative standard deviation (RSD). $$RSD = \left(\frac{s}{\bar{x}} \right) x\ 100$$ where: s = the standard deviation (see next page) $\bar{x}$ = the mean of the values.

TABLE 8-5
Precision and Accuracy Measurements
(Continued)

Measurement	Definition
Arithmetic mean	The average of a set of values. $\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$ where: $\bar{x}$ = the mean x_i = the ith data value n = number of data values
Standard Deviation	A measure of the random (probable) error associated with a single measurement within a data set. $s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$ where: s = sample standard deviation $\bar{x}$ = the mean x_i = the ith data value n = number of data values
Relative Standard Deviation	A measure of precision (relative dispersion). It is equal to the standard deviation (s) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.
ACCURACY	
Upper Control Limit (UCL)	$UCL = \bar{x} + 3s$
Lower Control Limit (LCL)	$LCL = \bar{x} - 3s$
PRECISION	
RPD	Zero to (mean RPD + 3s)

TABLE 8-6
Field Quality Control Samples

Type	Applicability		Accuracy and Precision	Introduced By
	Inorganic	Organic	Application	
Trip Blank (volatiles)	No	Yes	Accuracy	Whoever supplies containers to the field
Field Blank	Yes	Yes	Accuracy	Field Sampler
Rinsate Blank	Yes	Yes	Accuracy	Field Sampler
Collocated Sample	Yes	Yes	Precision	Field Sampler
Split Sample	Yes	Yes	Precision	Field Sampler
Field Duplicate	Yes	Yes	Precision	Field Sampler
Field Matrix Spike	Yes	Yes	Accuracy	Field Sampler

TABLE 8-7
Laboratory Quality Control Samples

Type	Frequency	Applicability		Accuracy and Precision Application	Introduced By
		Inorganic	Organic		
Analytical Spike	As specified in methods, or as needed	Yes	No	Accuracy	Analyst/ Prep
Duplicate	As specified in methods, or as needed	Yes	Yes	Precision	Analyst/ Prep
Instrument Blank	As specified methods, or as needed	Yes	Yes	Accuracy	Analyst
Interference Check Sample	As specified in methods	Yes	No	Accuracy	Analyst
Internal Standard	Each sample and standard	Yes	Yes Method Dependent	Both	Analyst/ Prep
Laboratory Control Sample	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike Duplicate	1 per each group of samples processed up to 20 samples.	Yes	Yes	Both	Analyst/ Prep
Method Blank	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Surrogate	All standards, method blanks, LCS, and samples.	No	Yes Method Dependent	Accuracy	Analyst/ Prep
Post Digestion Spike	At client request and 1 with each ICP air train batch, 1 with each Hg front-half batch	Yes	No	Accuracy	Analyst

TABLE 8-8
Laboratory Performance Quality Control Samples

Sample/Measurement	Purpose
Method Blanks	Demonstrates that the laboratory systems (*e.g.,* glassware cleaning procedures) and laboratory reagents used for the preparation and analysis of samples have not contributed to a false positive or negative measurement.
Instrument Blank	Demonstrates that the analytical system has not contributed to a false positive or negative measurement.
Laboratory Control Sample	Demonstrates the laboratory's ability to perform an analysis within the performance requirements of the method.

TABLE 8-9
Matrix Specific Quality Control Samples

Quality Control Sample	Purpose
Duplicate Samples	Estimates the ability of the laboratory to obtain precise measurements on a sample. This measure is dependent on the homogeneity of the sample being duplicated. Solid samples often portray poor sample homogeneity and therefore often have poor duplication with regards to the sample result.
Matrix Spike Sample	Estimates the ability of the laboratory to obtain accurate measurements on a sample. The measure is dependent on the bias a sample matrix may cause regarding a given analyte.
Matrix Spike Duplicate Sample	In addition to verifying the accuracy of the matrix spike sample, the matrix spike duplicate can be used with the matrix spike sample as a measure of precision by calculating the relative percent difference (RPD).

TABLE 8-10
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]	Method	Requirements[2]
Anions (Bromide, Chloride, Fluoride, Nitrite, Nitrate, Nitrite/Nitrate, ortho-Phosphate, Sulfate)	Water	100 mL	300.0	250 mL plastic or glass, Cool, 4°C, analyze ASAP after collection, within 28 days(Bromide, Chloride, Nitrate/Nitrite, Sulfate), 48 hours (Nitrate, Nitrite, ortho-phosphate)	9056	250 mL plastic or glass, Cool, 4°C, analyze ASAP after collection, within 28 days(Bromide, Chloride, Nitrate/Nitrite, Sulfate), 48 hours (Nitrate, Nitrite, ortho-phosphate)
	Solid	10 g	300.0	60 mL plastic or glass, Cool, 4°C, No preservative required, 28 days after leaching(Bromide, Chloride, Nitrate/Nitrite, Sulfate), 48 hours (Nitrate, Nitrite, ortho-phosphate) after leaching	9056	60 mL plastic or glass, Cool, 4°C, analyze ASAP after collection, within 28 days of leaching (Bromide, Chloride, Nitrate/Nitrite, Sulfate), 48 hours (Nitrate, Nitrite, ortho-phosphate)after leaching
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Cyanide (Amenable)	Water	250 mL	335.1	250 mL plastic or glass, NaOH to pH >12 0.6g ascorbic acid[4] Cool, 4°C, 14 days.	9012A	250 mL plastic or glass, NaOH to pH >12 0.6g ascorbic acid[4] Cool, 4°C, 14 days
Cyanide (Total)	Water	250 mL	335.4	250 mL plastic or glass, NaOH to pH >12 0.6g ascorbic acid[4] Cool, 4°C, 14 days	9012A	250 mL plastic or glass, NaOH to pH >12 0.6g ascorbic acid[4] Cool, 4°C, 14 days
	Solid	10g	--	Not Applicable	9012A	2 oz glass Teflon-lined lids, Cool, 4°C, 14 days
	Waste	10g	--	Not Applicable	9012A	4 oz glass Teflon-lined lids, Cool, 4°C, 14 days
Hardness (Total)	Water	50 mL	2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	Not Applicable

TABLE 8-10
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]	Method	Requirements[2]
Perchlorate	Water	250 ml	314.0	250 mL plastic or glass. Analyze within 28 days	--	--
	Solid	10 g	314.0	2 oz. plastic or glass. Analyze within 28 days of leaching	--	--
pH	Water	250 mL	150.1	250 mL plastic or glass. Analyze immediately. **This test should be performed in the field[5].**	9040B	250 mL plastic or glass. Analyze immediately. **This test should be performed in the field.[5]**
	Solid	20 g	---	Not Applicable	9045C	2 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.[5]
	Waste	20 g	---	Not Applicable	9045C	2 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.[5]
Metals (excludes Hg)	Water	100 mL	200.7	250 mL glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months	6010B	250 mL glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months
	Solid	10 g	200.7	2 oz glass or polyethylene container storage at 4 °C, 6 months	6010B	2 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	10 g	200.7	Not Applicable	6010B	2 oz glass or polyethylene container, storage at 4°C, 6 months

TABLE 8-10
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]	Method	Requirements[2]
Mercury (CVAA)	Water	100 mL	245.1	125 mL glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days	7470A	125 mL glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days
	Solid	10 g	245.5	2 oz glass or polyethylene container, Cool, 4°C, 28 days	7471A	2 oz glass or polyethylene container, Cool, 4°C, 28 days
	Waste	10 g	--	Not Applicable	7471A	2 oz glass or polyethylene container, Cool, 4°C, 28 days

TABLE 8-10
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]
Ash	Aqueous Waste Feed	10 g	ASTM D482	Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Organic Liquid Waste Feed	10 g		Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Soil, Sludge, Sediment, or Solid Waste Feed	10 g		Wide-mouth glass jar with Teflon™-lined closure (minimum 4 oz.). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
Density	Aqueous Waste Feed		ASTM D1963, D854	Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Organic Waste Feed			Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Soil, Sediment, Sludge or Other Solid Waste Feed			Wide-mouth glass jar with Teflon™-lined closure (minimum 4 oz.). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
Halogens	Aqueous Waste Feed	2 g	Methods 5050, ASTM Method E442	Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Organic Waste Feed	2 g		Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Soil, Sediment, Sludge or Other Solid Waste Feed	2 g		Wide-mouth glass jar with Teflon™-lined closure (minimum 4 oz.). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.

TABLE 8-10
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]
Heat of Combustion	Aqueous Waste Feed	2 g	D5865, D240	Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Organic Waste Feed	2 g		Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Soil, Sediment, Sludge or Other Solid Waste Feed	2 g		Wide-mouth glass jar with Teflon™-lined closure (minimum 4 oz.). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
Hexavalent Chromium	Stack gas samples	5 mL	0061/ 7199	Samples are collected in polypropylene containers. Samples must be stored at 4°C. The holding time is 14 days.
Viscosity	Aqueous Waste Feed		D445	Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Organic Waste Feed			Amber glass Boston round bottle with Teflon™-lined closure (minimum: 1 x 250 mL). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.
	Soil, Sediment, Sludge or Other Solid Waste Feed			Wide-mouth glass jar with Teflon™-lined closure (minimum 4 oz.). No preservation required. Recommended to cool to 4°C if high volatile content is suspected. No holding time specified.

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
(2) Holding times are calculated from date of collection.
(3) Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.
(4) Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.
(5) If not done in the field (ASAP) per the method and requested by client, it should be analyzed in lab within 48 hours.

TABLE 8-11
Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]	Method	Requirements[2]
Dioxins/Dibenzo -furans (HRGC/HRMS)	Water	1 L	1613B	1 liter amber glass w/fluoropolymer-lined screw cap. Add sodium thiosulfate if residual chlorine. If pH > 9, H_2SO_4 to pH 7-9. Cool, 0-4°C in the dark. Extraction , 1 year. Analysis, within 40 days of extraction.	8290	1 liter glass amber with Teflon®-lined lid, Cool, 4°C, Extraction, 30 days Analysis, 45 days from date of extraction
	Solid	10g	1613B	2 oz. amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, within 40 days of extraction.	8290	2 oz. glass amber wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 30 days Analysis, 45 days from date of extraction
	Waste	10g	1613B	2 oz. amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, within 40 days of extraction.	8290	2 oz. glass amber wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 30 days Analysis, 45 days from date of extraction

TABLE 8-11
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]
PCBS By HRGC/HRMS	Water	1 L	1668A	1 liter amber glass w/fluoropolymer-lined screw cap. Add sodium thiosulfate if residual chlorine. H_2SO_4 to pH 2-3. Cool, 0-4°C in the dark. Extraction , 1 year. Analysis, 1 year.
	Solid	10g	1668A	2 oz. amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, 1 year.
	Waste	10g	1668A	2 oz. amber wide, mouth, glass w/fluoropolymer-lined screw cap. Cool, <4°C in the dark until receipt in Lab, then <-10°C in the dark. Extraction , 1 year. Analysis, 1 year.
Nitroaromatics	Water	1L	8330 8332	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	2 g	8330 8332	2 oz. glass widemouth with Teflon®-lined lid Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	2 g	8330 8332	2 oz. glass widemouth with Teflon®-lined lid Cool, 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction
Pesticides/ PCBs	Water	1L	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	8081A 8082	4 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	1 g	8081A 8082	2 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction
Semivolatiles	Water	1L	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, within 40 days of extraction
	Solid	50 g	8270C	4 oz glass wide mouth with Teflon-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
	Waste	1 g	8270C	2 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days, Analysis, within 40 days of extraction

TABLE 8-11
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix[3]	Minimum Sample Size[1]	Method	Requirements[2]
Volatile Organics	Water	40 mL	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[4]
	Solid	5 g or 25 g	8260B	2 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C [5].
	Waste	5 g or 25 g	8260B	2 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C [5].

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

(2) Holding times are calculated from the date of collection.

(3) Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.

(4) For acrolein and acrylonitrile the pH should be adjusted to 4-5.

(5) Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to -12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

TABLE 8-12
Sample Containers, Preservatives, and Holding Times for TCLP[(1)] and SPLP[(2)]

Analytical Parameters	Matrix	Minimum Sample Size[(3)]	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

(1) TCLP = Toxicity Characteristic Leaching Procedure

(2) SPLP = Synthetic Precipitation Leaching Procedure

(3) Smaller sample size is adequate for solid samples or individual fractions. A combined volume of 32 oz. is recommended for semivolatiles and metals. A separate 4 oz. container should always be used for the volatile fraction. Volatile fractions should be stored with minimal headspace.

TABLE 8-13
Periodic Equipment Calibrations

Type of Equipment	Calibration Requirements
Balances	• Must be serviced and calibrated annually by an approved vendor. • Calibration must be checked daily or before use by analyst with weight(s) classified as Class 1 (formerly termed Class S) by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in the log. • All Class 1 weights must be certified by an outside vendor every three years. • All non-Class 1 weights must be checked annually against NIST Class 1 weights.
Thermometers	• Working glass mercury thermometers must be calibrated against a certified NIST thermometer at least annually as described in operation-specific SOPs. • Working non-mercury glass thermometers must be calibrated against a certified NIST thermometer quarterly as described in operation-specific SOPs. • The NIST thermometer must be recertified every three years.
Refrigerators/Freezers	• Thermometers must be immersed in a liquid such as mineral oil or glycol • Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: 4°C ± 2°C Freezer acceptance limits: - 10°to - 20°C
Ovens	• Temperature of units must be checked daily or before use. • Acceptance limits vary according to use as described in operation-specific SOPs and must be documented in the temperature log.
Micropipettors	• Calibrations are checked gravimetrically as required by the operation specific SOP at a quarterly frequency.
Syringes, Volumetric Glassware and Graduated Glassware	• All syringes and volumetric glassware are purchased as Class A items. • Class A items are certified by the manufacturer to be within ± 1% of the measured volume, therefore, calibration of these items by STL laboratories is not required. • All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents and sample volumes are within method tolerances.

TABLE 9-1
Proficiency Testing Programs

PT Sample Program Description	Analysis Performed	Frequency of Participation
Water Pollution Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	Trace Metals, Minerals, PCBs, Chlorinated Hydrocarbon, Pesticides, Volatile Halocarbons, Volatile Aromatics, Total CN	Semi-annual
Water Supply Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	pH, Sodium, Trace Metals, Cyanide, 2,3,7,8-TCDD.	Semi-annual
Hazardous Waste Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	Semivolatile Organics (BNA), Pesticides, Herbicides, Volatile Organics, Metals, Anions, PAH, Explosives	Semi-annual
Department of Energy Mixed Analyte Performance Evaluation Program (MAPEP)	Trace Metals, Semivolatile Organics	Annual
STL Corporate Double Blind	May include Volatile Organics, Metals, General Chemistry, Base/Neutral Acid Extractables, Pesticides/PCBs.	Annual

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SEVERN TRENT **STL**

LABORATORY QUALITY MANUAL

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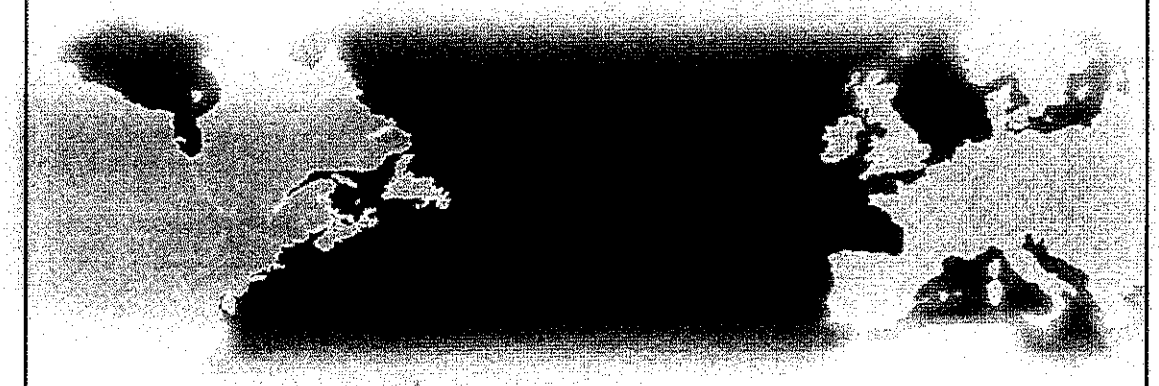
Appendix

List of Cited SOPs and Work Instructions 77

SEVERN TRENT **STL**

Vision

STL will be the recognized industry leader for environmental analysis.



Mission

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

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1.0 Introduction, Purpose, and Scope

1.1 STL Overview

STL Buffalo (STL) is a part of Severn Trent Laboratories, a major group of U.S. based companies. STL is a full-service environmental laboratory that provides quality comprehensive and integrated professional analytical services effectively and efficiently. A broad range of environmental testing services are offered that span a variety of matrices including aqueous, saline, solid, tissue and drinking water.

Associated with this activity are services to assure client requirements are known, communicated and satisfactorily addressed, and a deliverables package presenting the analytical results. The laboratory provides expert personnel for supervision, technical consultation, and project review for effective planning and implementation of analytical assignments.

STL operates under the regulations and guidelines of the following federal programs:

- Clean Water Act (CWA)
- National Pollution, Discharge, and Elimination System (NPDES)
- Occupational Safety and Health Administration (OSHA)
- Resource Conservation and Recovery Act (RCRA)
- Safe Drinking Water Act (SDWA)
- Toxic Substances Control Act (TSCA)

STL also provides services under various state and local municipal guidelines. A current table of analytical services and general service listing is presented on STL's website under the MySTL webpage or available from the laboratory. A current listing of STL Buffalo certifications (STLBuffCertList) is maintained by the laboratory on the company network directory. Copies of the actual certificates are available on the STL Buffalo intra-net site (BufNet).

1.2 Quality Assurance Policy

It is STL's policy to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Promote employee adherence to quality documentation and implementation of Corporate Policies and Procedures
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

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1.3 Management Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a quality system that is clear, effective, well communicated, and supported at all levels in the company.

Line organizations verify that specifications are achieved; QA organizations assist and provide oversight and verification of processes through planning, reviews, audits, and surveillances. The quality objectives are derived from this Laboratory Quality Manual (LQM), Standard Operating Procedures (SOPs) and Work Instructions.

1.4 Purpose

The purpose of the LQM is to describe STL's Quality System and to outline how that system enables all employees to meet the Quality Assurance (QA) policy. This LQM also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in this LQM.

1.5 Scope

This LQM is specific to STL Buffalo's quality systems and laboratory operations. All other STL locations have LQMs under the Corporate Quality Management Plan (QMP) or the Corporate QMP itself.

The laboratory is committed to ensuring that resources are available and deployed to meet client expectations. This includes gathering project information prior to sample receipt to ensure client expectations will be met with respect to:

- Sampling containers;
- Analytical methods employed;
- Accuracy and precision;
- Reporting limits;
- Personnel qualifications, training, and experience;
- Calibration and quality control measures employed;
- Regulatory requirements;
- Report contents;
- Supporting documentation, records and evidence; and
- Review of data

1.6 Servicing

Project Managers are the direct client contact and they ensure resources are available to meet project requirements. Although Project Managers do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project Managers provide a link between the client and laboratory resources.

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The laboratory has established procedures for performing and verifying that client servicing meets requirements. Typical services provided are:

- Sample Containers/Supplies – *Container Management: Process Operation/Bottle Order Set-Up (APM-BottleOrder-03)*
- Project QAP preparation – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*
- Regulatory advisory functions – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*
- Consulting – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*

Regulatory and advisory functions are addressed under the same procedures used for project planning.

2.0 References

The following references were used in preparation of this document and as the basis of the STL Quality System:

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, US EPA, Office of Environmental Information, EPA/240/B-01/004, March 2001.

EPA Requirements for Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, EPA/240,B-01/002 March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, EPA/240/B-01/003, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA Office of Environmental Information – Quality Staff, May 2000.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations with Implementation Guidance, EPA 2185, US EPA Office of Information Resources Management, August 1995.

National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA 600/R-04/003, US EPA Office of Research and Development, June 2003.

Quality Systems Manual for Environmental Laboratories, Department of Defense, Version 3.0, March 2005

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, December 1998.

Quality Systems for Analytical Services, U.S. Department of Energy, Rev. 1, April 2004.

This LQM was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards. Refer to Table 1 for a cross-section comparison of this LQM to the NELAC standards.

Table 1.

Correlation of QAPP Sections with NELAC 5.4.2.3 Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Laboratory Quality Manual Section
a. Quality policy statement, including objectives and commitments	1.2 Quality Assurance Policy 4.2.1 Objectives of the Quality System
b. Organization and management structure	4.1 Organization and Management
c. Relationship between management, technical operations, support services and the quality systems	4.1.2 Roles and Requirements 4.2 Quality System
d. Records retention procedures; document control procedures	4.3 Document Control 4.12.2 Record Retention
e. Job descriptions of key staff and references to job descriptions of other staff	4.1.2 Roles and Requirements
f. Identification of laboratory approved signatories	4.1 Organization and Management
g. Procedures for achieving traceability of measurements	5.5 Measurement Traceability
h. List of all test methods under which the laboratory performs its accredited testing	5.3.1 Method Selection
i. Mechanisms for assuring the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	4.4.2 Project-Specific Quality Planning
j. Reference to the calibration and/or verification test procedures used	5.4.3 Equipment Verification and Calibration 5.3.6.2 Data Review
k. Procedures for handling submitted samples	4.7.1 Sample Acceptance Policy 5.7 Sample Handling, Transport and Storage
l. Reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests	1.6 Servicing 4.1.1 Laboratory Facilities 5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
m. Reference to procedures for calibration, verification and maintenance of equipment	5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
n. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes	5.8.1 Proficiency Testing 5.8.2 Control Samples
o. Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	4.9 Control of Non-Conformances 4.10 Corrective Action 4.11 Preventive Action 5.8.6 Permitting Departures from Documented Procedures
p. Laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	4.4.2 Project-Specific Quality Planning 5.8.6 Permitting Departures from Documented Procedures
q. Procedures for dealing with complaints	4.8 Complaints

Table 1.

Correlation of QAPP Sections with NELAC 5.4.2.3 Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Laboratory Quality Manual Section
r. Procedures for protecting confidentiality and proprietary rights (including national security concerns)	4.7.2 Client Confidentiality and Proprietary Rights
s. Procedures for audits and data review	4.13 Internal Audits 4.14 External Audits 5.3.6 Data Reduction and Review
t. Process/procedures for establishing that personnel are adequately experienced in duties they are expected to carry out and are receiving any needed training	5.1.2 Training
u. Ethics policy statement developed by the laboratory and training personnel in their ethical & legal responsibilities	5.1.3 Ethics Policy
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3.0 Terms and Definitions

Accuracy: The degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: A systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: Environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of 1 to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (e.g., volatile organics, water), the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): A system of documentation demonstrating the physical possession and traceability of samples.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): Legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: A sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 of this LQM for a description of these conditions.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

Confirmation: Verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: Action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): Procedure to establish the ability to generate acceptable accuracy and precision.

Document Control: The act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed

ERA Sample: A control sample obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Equipment Blank (EB): A portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): Legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): Legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank (FB): A blank matrix brought to the field and exposed to field environmental conditions.

Field Duplicate (FD): Duplicate field-collected sample most commonly used to assess the accuracy of the field collection process.

Field of Testing (FOT): A field of proficiency testing is based on NELAC's categorization of accreditation based on program, matrix and analyte.

Good Laboratory Practices (GLP): Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: The maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: A blank matrix that is the same as the processed sample matrix (e.g. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody (COC): An unbroken trail of accountability that ensures the physical security of samples, data and records. Internal COC refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure; also referred to as Matrix Spike Blank (MSB); Environmental Resource Associate Sample (ERA).

Laboratory Quality Manual (LQM): A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): The minimum amount of a substance that an analytical process can reliably detect. Also referred to as the Method Detection Limit (MDL)

Matrix: The substrate of a test sample. Common matrix descriptions are defined in Table 2.

Table 2. Matrix Descriptions

Matrix	Description
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater, effluents, leachates and wastewaters.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge, ash, paint chips, filters, wipes or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined (i.e., drum liquid or oils).
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Matrix Duplicate (MD): Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): Field sample to which a known amount of target analyte(s) is added.

Matrix Spike Blank (MSB): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure; also referred to as Laboratory Control Sample (LCS).

Matrix Spike Duplicate (MSD): A replicate matrix spike.

Method Blank (MB): A blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: An estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: Refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: A sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: Belonging to a private person or company.

Quality Assurance (QA): An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): The overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control (QC) Sample: A control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quality Management Plan (QMP): A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Quantitation Limit (QL): The minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): Legislation under 42 U.S.C. 321 et seq. (1976).

Safe Drinking Water Act (SDWA): Legislation under 42 U.S.C. 300f et seq. (1974), Public Law 93-523.

Sampling and Analysis Plan (SAP): A formal document describing the detailed sampling and analysis procedures for a specific project.
Selectivity: The capability of a measurement system to respond to a target substance or constituent.

Sensitivity: The difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: A known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: A blank matrix stored for 1 to 2 weeks with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Systems Audit: A thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: Defined technical procedure for performing a test.

Toxic Substances Control Act (TSCA): Legislation under 15 U.S.C. 2601 et seq., (1976).

Traceability: The property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank (TB): A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Verification: Confirmation by examination and provision of evidence against specified requirements.

4.0 Management Requirements

The organizational chart of STL is presented in Figure 1. Corporate employees are located at various STL facilities as outlined in the organizational structure. The organizational chart of STL Buffalo is presented in Figure 2.

4.1 Organization and Management

The Laboratory Director and Quality Assurance Manager are responsible and have the signature authority for approving and implementing this plan. The Laboratory Director and/or his designee also have signatory authority for approval of work and release of reports. The following listing defines those employees that may act as report signatory designees for the Laboratory Director.

Technical Director
Operations Manager
Quality Manager
Customer Service Manager
Project Manager
Project Manager Assistant

Figure 1. STL Organization Chart

Figure 1. STL Organizational Chart

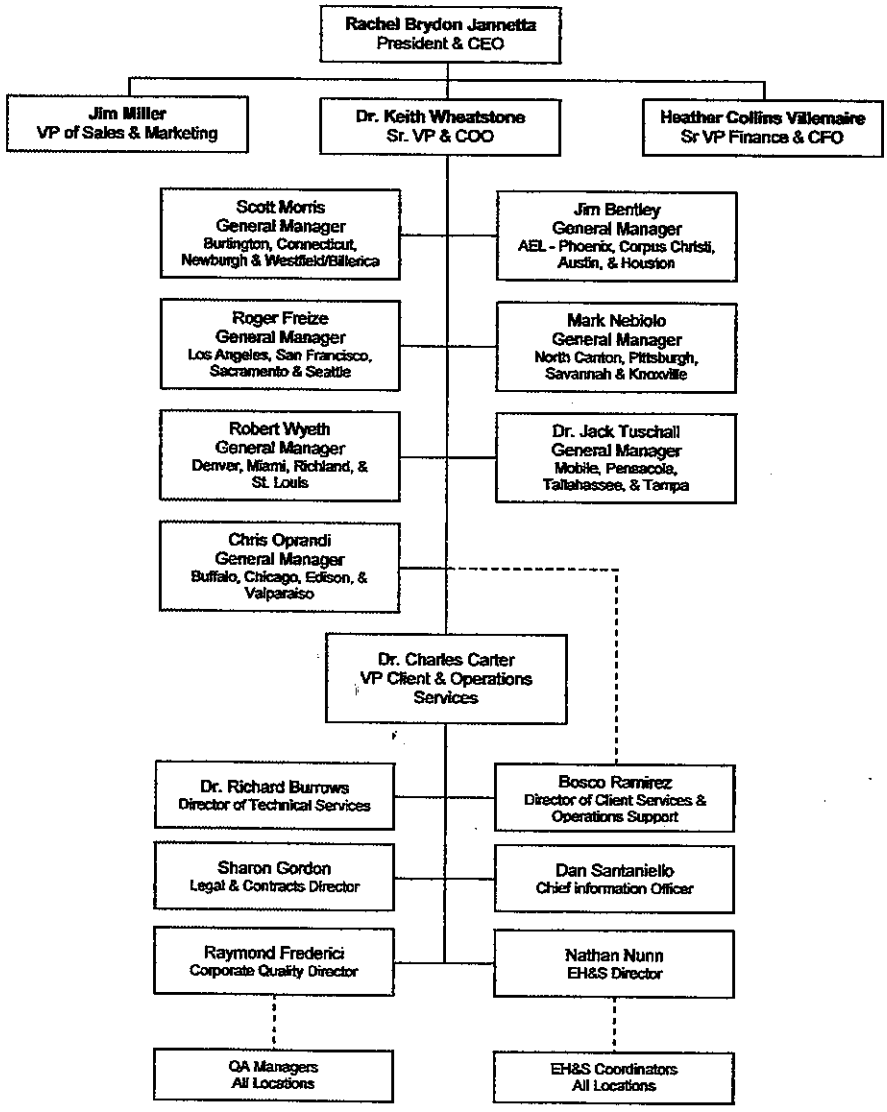
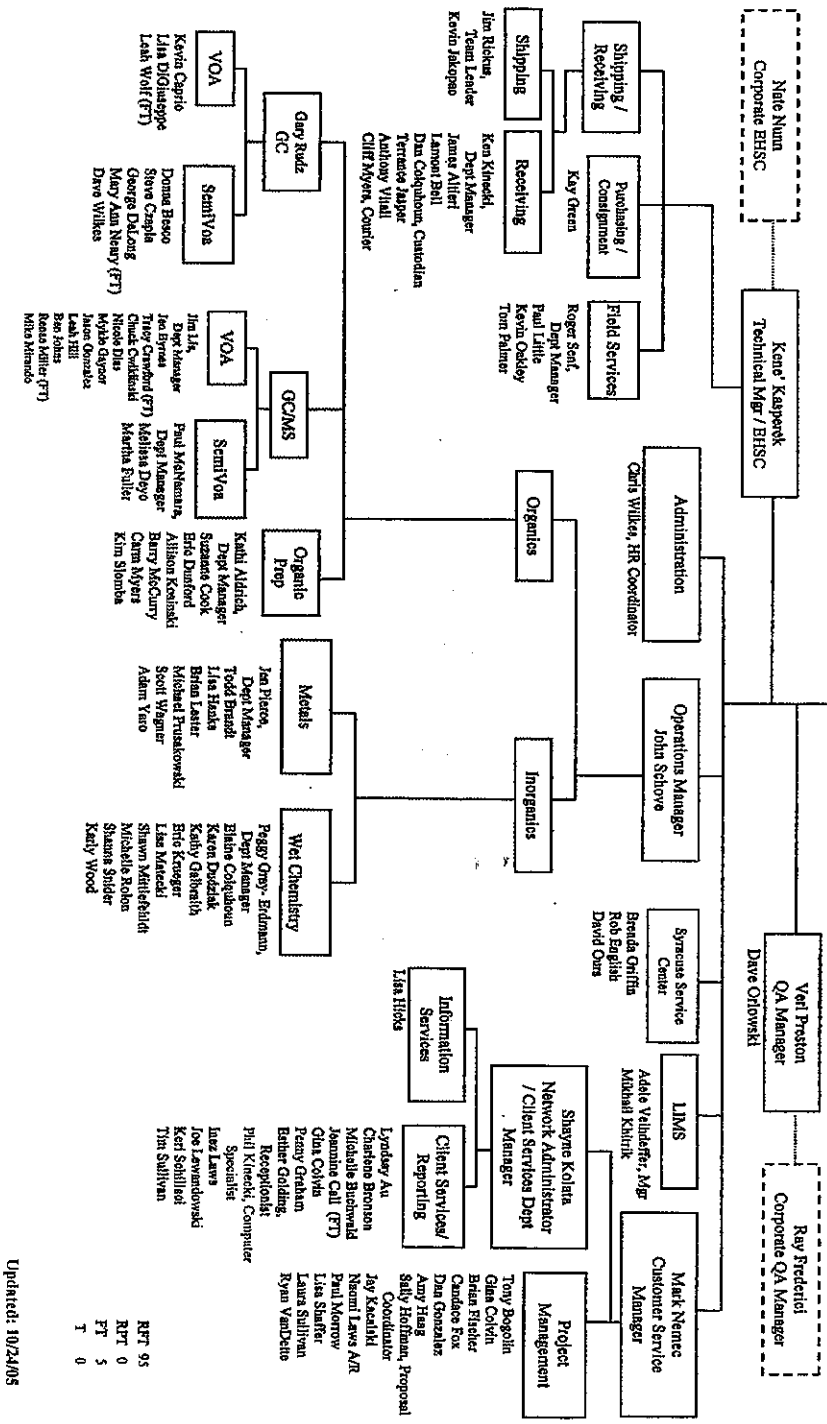


Figure 2. STL Buffalo Organizational Chart, November 2005



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4.1.1 Laboratory Facilities

The laboratory is located in Amherst, New York, which is approximately 10 miles from Buffalo, New York, and is staffed by 100 professionals. The laboratory is comprised of 28,000 square feet of state-of-the-art commercial laboratory and office space and houses both inorganic and organic operations. The facility is divided into separate work areas to facilitate sample throughput. These areas include the following:

- Sample receipt and refrigerated storage
- Bottle kit preparation
- Organic and TCLP sample preparation
- Metals digestion
- Wet chemistry laboratory
- Organic instrumentation laboratories
- Metals instrumentation laboratory

The main instrumentation laboratories are equipped with state-of-the-art instrumentation and sufficient duplicate equipment to provide back-up service for most major systems. A listing of laboratory equipment and instrumentation is referenced as STL Buffalo Capital Equipment List, (STLBuffEquipList). Table 3 is a summary of the major laboratory instruments.

Table 3. Major Equipment List

GC	GC/MS	ICP	ICP/MS	CVAA	HPLC	Auto Analyzer	IC	TOC	TOX
20	12	2	1	2	1	4	3	2	1

Each of the laboratory areas has separate heating, ventilation, and air conditioning systems. Non-destructive gas chromatographic detectors, and GC/MS rotary pumps are vented out of the instrumentation through charcoal filters.

4.1.2 Roles and Responsibilities

The specific duties and responsibilities of the Laboratory Director, Technical Director/Environmental Health & Safety Officer, Quality Assurance Manager, Operations Manager, Customer Service Manager/Project Managers, Laboratory Supervisors, Sample Management Coordination, Information Technology Manager, and Chemists/Technicians and Data Packaging Specialists are as follows.

In the absence of any one individual, the staff or assistant within each department is professionally skilled in the ability to administer the function of the administrator or support personnel. This will allow for the continuance of the day-to-day operations of the laboratory.

4.1.2.1 Laboratory Director

The ultimate responsibility for the generation of reliable laboratory data rests with the Laboratory Director, who is accountable to his General Manager and oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include allocation of personnel and resources, setting goals and objectives for both the business and employees, achieving the financial, business and quality objectives of STL. Furthermore, to see that all tasks performed in the laboratory are

conducted according to the requirements of this LQM, the Project Specifications and/or the appropriate QAPP; and to assure that the quality of service provided complies with the project's requirements.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

4.1.2.2 Quality Assurance Manager

The Quality Assurance Manager (QAM) has the full-time responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in this LQM. The QAM is responsible for:

- Ensures that the laboratory's quality system and LQM meet the requirements of the Corporate QMP.
- Ensures IDL/MDL studies are completed and documented
- Ensures method validation studies are completed and documented
- Periodically performs data package inspections
- Performs data authenticity audits on 100% of analysts and instruments
- Assist in the preparation, compilation, and submittal of quality assurance project plans
- Reviews program plans for consistency with organizational and contractual requirements and advises appropriate personnel of deficiencies
- Maintains QA records
- Maintains certifications and accreditations
- Initiates and oversees both internal and external audits; documents root cause investigations for all noted deficiencies; and ensures timely audit closure
- Maintains a corrective action process for internally identified issues and ensures timely closure
- Manages the laboratory's PT Program and performs/documents root cause investigations for all failures
- Monitors to ensure the documentation of training and method demonstration is current
- Facilitates SOP development and document control
- Submits monthly QA reports to management

The QAM shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QAM is available to any employee at the facility to resolve data quality or ethical issues. The QAM shall be independent of laboratory operations and has an indirect reporting relationship to the STL Corporate QA Director.

4.1.2.3 Technical Director

The Technical Director is responsible for assessing the construction and management of the facility design, maintaining environmental conditions, technical and financial evaluation of capital equipment and capital budgeting and asset valuation.

In addition, the Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers and clients, investigates technical issues identified by operations personnel or QA, and directs evaluation of new methods.

4.1.2.4 Operations Manager

The Operations Manager reports to the Laboratory Director and oversees the daily operations of the analytical laboratory, maintaining a working environment that encourages open, constructive problem solving and continuous improvement.

The Operations Manager is responsible for supervision of laboratory staff, setting goals and objectives for the laboratory, ensuring compliance with project/client requirements and ensuring on-time performance, supervises maintenance of equipment and scheduling of repairs. Responsibilities also include implementation of the quality system in the laboratory and ensuring timely compliance with audit and QA corrective actions.

In addition, the Operations Manager works with the Technical Director in evaluating technical equipment and assessing capital budget needs.

4.1.2.5 Customer Service Manager/Project Managers

The laboratory recognizes the importance of efficient project management. The laboratory Project Managers (PM) are responsible for preparing the LIMs project technical specifications which summarize QA/QC requirements for the project, maintaining the laboratory schedule, communicating technical requirements to the laboratory, and advising the Operations Manager, QA and Laboratory Supervisors of all variances. The laboratory Project Manager will provide technical guidance and the necessary laboratory-related information to the preparer of project-specific QAPPs and provide peer review of the final document to ensure accuracy of the laboratory information.

4.1.2.6 Laboratory Supervisors

The Laboratory Supervisors are as follows:

- Sample Management Supervisor
- Organic Preparation Supervisor
- GC Analysis Supervisor
- GCMS Volatiles Supervisor
- GCMS Semivolatiles Supervisor
- Metals Supervisor
- Wet Chemistry Supervisor

The Laboratory Supervisors serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Operations Manager in achieving section goals. The Laboratory Supervisors are responsible for ensuring that their personnel are adequately trained to perform analyses; that equipment and instrumentation under their control is calibrated and functioning properly; that system and performance audits are performed on an as-needed basis; provide input and review in the development and implementation of project-specific QA/QC requirements; and for providing the critical review of proposal and project work for programs as

directed by the Operations Manager and Laboratory Director. The Laboratory Supervisors coordinate these activities with the project management and quality assurance sections.

4.1.2.7 Sample Management Coordination

The Sample Custodian is designated as the Sample Management Coordinator for any work performed internally and responsible for the receipt and login of client samples. The sample custodian confirms the samples received against the Chain of Custody, transports the samples to the proper storage unit within the facility and tracks the disposal of client samples after the required holding time has expired.

4.1.2.8 Subcontract Sample Management Coordination

The Project Manager is designated as the Sample Management Coordinator for any work subcontracted under their management. The Project Manager verifies each subcontracting request to ensure that special client restrictions are not jeopardized (e.g., samples must be analyzed by the receiving affiliated or network laboratory and must maintain specific certification(s)). The Project Manager is also responsible for verifying the credentials; establishing the service agreement; ensuring data review; and invoicing of all laboratory subcontractors. The Project Manager discusses any deficiencies or anomalies with the subcontractor prior to reporting any data to the client.

4.1.2.9 Environmental Health and Safety Coordinator / Waste Management

The Health and Safety Coordinator is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Coordinator responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste and preparation of Safety related SOPs. The EHSC maintains overall EH&S program oversight, but may delegate specific day-to-day activities as necessary.

4.1.2.10 Information Technology Manager

The overall role of the Information Technology (IT) Manager is to enhance laboratory productivity through improved information access, flow, and security. For information to be of greatest value, it must be readily accessible and reliable. It is the responsibility of the IT Manager to provide software tools that allow quick and user friendly access to that information, while at the same time controlling access to that information to those that have the need and proper authority.

Information flow can be enhanced through automation. Automation is the minimization of human intervention in a process. Reduction in human intervention can result in significant error reductions and time savings. The IT Manager assists the laboratory in automation by providing hardware and software solutions to help minimize human intervention in data collection, processing, and storage.

The IT Manager is responsible for providing data security by controlling access, as mentioned above, and for providing for disaster recovery. Data stored on the central Laboratory Information Management System (LIMS) is the direct responsibility of the IT Manager. No fewer than two copies of all data should exist at any time so that lost or destroyed data can always be retrieved from an alternate source. These copies may consist of data within the system and on electronic storage media. Data stored electronically in other departments is the direct responsibility of those departments. However, the IT Manager is responsible for providing procedures and training to all laboratory operations, as appropriate, to assist in making backup copies of local data.

STL has established procedures for IT management:

- *Computer Systems Account and Naming Policy* – P-I-003
- *Computer Systems Password Policy* – P-I-004
- *Software Licensing Policy* – P-I-005
- *Virus Protection Policy* – P-I-006

4.1.2.11 Chemists / Technicians

Any effective laboratory quality assurance/quality control program depends on the entire organization, including management and every individual on the laboratory staff. Analysts and technicians must read and be familiar with the LQM, method SOPs and other essential standard operating procedures. They must know where SOPs are located and agree to adhere to them explicitly unless an error in the SOP is evident and they brought this to the attention of their supervisor or QA manager. They must receive ethics and data integrity training and sign an ethics agreement annually.

Analysts and technicians must ensure that their training records are up to date prior to performing a method without direct supervision. This includes maintaining their training file, filing demonstration of capability evidence and receiving supervisor approval.

The initial review for acceptability of analytical results rests with the analysts conducting the various tests. Observations made during the performance of an analytical method may indicate that the analytical system is not in control. Analysts must use quality control indicators to assure that the method is within acceptance limits, corrective action is taken or a non-conformance (job exception) report is documented/approved before reporting results.

4.1.2.12 Data Packaging Specialist

The Data Packaging Specialist is responsible for coordinating receipt of all data from the various service groups within the laboratory, reviewing data for compliance to laboratory QC criteria and/or criteria in the LIMs Project Profile Specification, and ensuring that data are reported in a timely manner and in the proper format.

4.2 Quality System

The quality system and quality objectives are driven by this LQM, SOPs and Work Instructions. Within these documents, the Laboratory Director and Quality Manager ensure that the quality policy is understood, implemented, and maintained at all levels of the organization. The development and implementation of appropriate accountabilities, duties, and authority by organizational positions are clearly delineated. Line organizations achieve and verify that specifications are achieved; the QAM provides oversight and verification of processes through planning, reviews, audits, and surveillances.

The Laboratory Director's leadership, support and direction ensure that the policies and procedures are implemented throughout the organization.

4.2.1 Objectives of the Quality System

The goal of the quality system is to ensure that business operations are conducted with the highest standards of professionalism and data integrity in the industry.

To achieve this goal, it is necessary to provide our clients with scientifically sound, well documented, regulatory compliant data, and to ensure that we provide the highest quality service available in the industry with uncompromising data integrity. A well-structured, organized and communicated quality system is essential in meeting this goal. The laboratory's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement.

This LQM, Work Instructions and the SOPs are the basis and outline for our quality and data integrity system and contain requirements and general guidelines under which the laboratory conducts operations. In addition, other documents may be used by the laboratory to clarify compliance with quality system or other client requirements. Within the LQM, SOP or Work Instruction numbers are noted in parenthetic text. These numbers refer to the laboratory procedure(s) associated with the subject item. A table listing these quality system policies and procedures is appended to this document.

The QA Manager is responsible for implementing and monitoring the Quality System. The QA Manager reports to the Laboratory Director on the performance of the quality system for review and continuous improvement. The QA Manager has sufficient authority, access to work areas, and organizational freedom (including sufficient independence from cost and schedule considerations) to:

- Initiate action to prevent the occurrence of any nonconformities related to product, process and quality system,
- Identify and record any problems affecting the product, process and quality system,
- Initiate, recommend, or provide solutions to problems through designated channels,
- Verify implementation of solutions, and
- Assure that further work is stopped or controlled until proper resolution of a non-conformance, deficiency, or unsatisfactory condition has occurred and the deficiency or unsatisfactory condition has been corrected.

The QA Manager identifies opportunities for continual improvement. When a situation arises where acceptable resolution of identified issues cannot be agreed upon at the laboratory, direct access to STL's Corporate QA Director is available. This provides laboratory QA personnel independence, where needed, to ensure that QA policies and procedures are enforced.

The QA Manager conducts annual training for all laboratory and administrative personnel to ensure their familiarity with the quality documentation and the implementation of the policies and procedures in their work.

4.3 Document Control

The laboratory maintains procedures to control documents and analytical data. Since an extensive quantity of data is generated and this is our primary product, document control is inherently segregated from data control, as described further in Sections 4.3.1 and 4.3.2.

4.3.1 Document Control Procedure

Organization, security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by information in the document header: Document Number, Revision Number, Effective Date, and Number of Pages. Document control may be achieved by either electronic or hardcopy distribution. Documents may be controlled for a specific time period after issuance. In this case the document will be marked with the date issue and expiration date.

Controlled documents are authorized and records of their distribution and archiving are maintained by the QA Department. Controlled status is defined as the continuous distribution of document updates where document marked as either "Controlled" or "Uncontrolled". Uncontrolled status is defined as the single distribution of the current SOP. Document updates are not distributed to people holding documents marked "uncontrolled". For tracking purposes, a control copy number is assigned to documents distributed with a controlled status. All copy numbers are written or typed in red to easily identify the SOP as a controlled copy.

4.3.1.1 Document Revision

Changes to documents occur when a procedural change warrants a revision of the document. After document revisions are authorized, all outdated versions are removed from use and disposed or segregated from the active/current document versions. A single copy of the archived document is retained for historical purposes. This archived version is clearly identified as an "Archived Copy".

SOPs are reviewed and/or updated on a 12 month basis, which is tracked by an established review schedule *(SOP Master Index)*. These reviews are conducted by the analyst, QA Manager, Department Supervisor, Laboratory Director or the Health and Safety Coordinator, all of whom may provide the approval signature for each SOP.

4.3.2 Data Control

All raw data, such as bound logbooks, instrument printouts, magnetic tapes, electronic data, as well as final reports, are retained for a minimum period of 5 years, unless otherwise specified by client or regulatory requirements. Such data may be maintained longer, as defined by client and project requirements. Specifics on the procedure of archiving records and client or project specific requirements are contained in the SOP, Record Storage and Retention,*(AGP-RecordStorage-56).*

Raw data and reports are documented and stored in a manner which are easily retrievable. The procedure for maintaining raw data records is briefly described below:

- Instrument print-outs for conventional inorganic parameters are filed by parameter and month. Inorganic Metals are filed by Instrument and Filename. Generally, current year documents are kept on file in the laboratory sections.
- All raw data, for example, instrument print-outs and logbooks, are maintained in a secured storage area or records are scanned and retained on electronic media.

- The computer information is backed up on tape daily, and stored in a secured and temperature/humidity controlled environment to maintain the integrity of the electronic information in the event of system failure. Copies of all back-up tapes are maintained in secured off-site locations.
- All copies of client final reports are maintained in hard copy format or electronically (e.g., Adobe Acrobat).

4.4 Request, Tender, and Contract Review

4.4.1 Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL's intent to provide both standard and customized environmental laboratory services to our clients. To ensure project success, technical staff members perform a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

All contracts entered into by the laboratory are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well as the ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this will be documented and discussed with the client prior to contract approval.

Any contract requirement or amendment to a contract communicated to STL verbally is documented and confirmed with the client in writing (e.g., letter, contract, e-mail, etc.). Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL. All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record.

4.4.2 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project (Project Information Requirements, APM-ProjInfo-20). QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the direct client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available

resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings *(APM-ProjInfo-20)* or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch (e.g., Job) as a reminder upon sample receipt and analytical processing.

Any changes that may occur within an active project is agreed upon between the client/regulatory agency and the Project Manager/laboratory. These changes (e.g., use of a non-standard method or modification of a method) must be documented prior to implementation. Documentation pertains to any document, e.g., letter, variance, contract addendum, which has been agreed to by both parties.

Such changes are also communicated to the laboratory through the management Production Meetings which are conducted two times per week. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the Project Manager or the individual laboratory section manager.

STL strongly encourages client visits to the laboratory and for formal/informal information sharing sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

4.4.3 Data Quality Objectives

Data quality objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation and during the development of QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into the measurement process of the laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The control samples and their applications, described in Section 5.8.2, are selected based on analytical method or client-specific requirements. Analytical QC samples for inorganic and organic analyses may include calibration blanks, instrument blanks, method blanks, laboratory control standards, calibration standards, matrix spikes, matrix duplicates and surrogate spikes.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

4.4.3.1 Precision

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from LCSs, MS, MSD, and MD.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

4.4.3.2 Accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCSs, MS and MSD.

Accuracy and Precision objectives employed by the laboratory are as defined in the CERCLA's Inorganic and Organic Statements of Work (SOW); statistically-derived control limits; or default limits as listed in each respective method SOP.

4.4.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

4.4.3.4 Completeness

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

4.4.3.5 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

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A measure of inter-laboratory comparability is obtained through the laboratory's participation in proficiency testing (PT) programs established with Water Supply (WS), Water Pollution (WP), Solid Waste (SW), and Underground Storage Tank (UST) programs. In addition, the laboratory employs the use of NIST or EPA traceable standards, when available, to provide an additional measure of assurance of the comparability of data.

Project representativeness and comparability are dependent upon the sampling plan on a project specific basis, and are therefore not covered in this LQM. Assessment of site and collection representativeness and comparability is performed by client or field engineer.

4.4.3.6 Additional DQOs

Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

For the performance of non-routine methods, e.g., client/contract requirement, MDLs or Method Validation Studies will be completed on an as needed basis. The turnaround time for such studies will be as determined by the client and Project Manager. Such studies will be reviewed and approved by the client and/or regulatory agency prior to project implementation.

Instrument Detection Limits

There are a number of ways to determine Instrument Detection Limit (IDL) sensitivity (e.g., signal-to-noise ratio, precision of the low-level standard, lowest calibration curve point or the IDL study defined within CLP). The method and means in which IDLs are determined are documented and maintained in the QA department for each individual instrument.

IDLs are periodically generated for each element by the metals laboratory based on project or program requirements (i.e., CLP is quarterly for each instrument). These limits are used to gauge instrument sensitivity without the introduction of preparation method variance.

Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory can report with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory reporting limits are further related and verified by the lowest point on a calibration curve. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory endeavors to keep reporting limits higher than the MDL. Wherever possible, reporting is limited to values approximately 2-5x the respective MDL to ensure confidence in the value reported.

Client specific requests for reporting below the routine laboratory reporting limit or approaching the IDL or MDL are special circumstances not to be confused with the previous statement. Data evaluated down to the MDL/IDL is qualified as estimated with a 'J' for organic analyses, a 'B' for inorganic analyses or with a comment in the report case narrative.

MDL studies are performed annually, and reporting limits are assessed. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated or the laboratory reporting

limit is reassessed. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to assure optimal performance or appropriate action is taken.

4.5 Subcontracting

Subcontracting is arranged with the consent of the client. Consent shall be requested in a timely manner and the client response shall be suitably prompt to ensure that it shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Proof of required certifications from the subcontract facility is maintained in the project records. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements (e.g., Technical Profile and LIMS Project Notes). STL may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting may also occur between STL facilities. Intra-company subcontracting within STL is arranged with the documented consent of the client or a QAPP specification. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

Project reports from both STL and external subcontractors are not altered and are included in their original form in the final project report provided by STL. This clearly identifies the data as being produced by a subcontractor facility. If subcontract data are incorporated into the laboratory's report (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples , as required in Section 5.9.4.

4.6 Purchasing Services and Supplies

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. The measurements for evaluation and selection of suppliers; the acceptance of supplies and services; and certificates of conformance are described in the procurement SOP *(Procurement of Laboratory Supplies and Services, APH-Supply-08).*

4.6.1 Solvent and Acid Lot Verification

Pre-purchase approval is performed for solvents and acids purchased in large quantities unless a certificate of conformance has been furnished. These may include acetone, ethyl ether, hexane, methylene chloride, nitric acid, hydrochloric acid, sulfuric acid, and hydrogen peroxide. Each lot of incoming supplies requiring pre-approval is checked against the previously approved lot number. If the lot number is not approved, the lot is refused. If the lot number is an approved lot number, it is accepted and documented. Solvents and acids are pre-tested in accordance with STLs Corporate *Testing Solvents and Acids* procedure (S-T-001) for all of the STL laboratories.

4.7 Service to the Client

4.7.1 Sample Acceptance Policy

Samples are considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservation.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples >6mm.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it is documented on the hardcopy COC, the LIMS Sample Receipt Checklist or on an Analytical Receipt Resolution Form (ARRF); and the client is contacted for instructions. If the client decides to proceed with the analysis, the project report will clearly indicate any of the above conditions and the resolution.

4.7.2 Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. Technical, business and proprietary information provided by a client and data/information generated by the laboratory are restricted for the use within the laboratory for purposes of accomplishing the project. Client information is not to be used on other projects or revealed except in conjunction with project work to anyone outside the laboratory without permission of the client.

STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client (*Client Confidentiality Section 6.9; APM-ProjInfo-20).*

4.8 Complaints

STL believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client's concerns captures "client knowledge" that helps to continually improve processes and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly. The investigation of the cause, resolution and authorization of corrective action is documented *[Data Quality Request (DQR); SOP AQA-DQR-65 or Corrective Action Notice (CAN); SOP AQA-CA-65)].*

Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a Data Quality Review request (DQR) or in a format specifically designed for that purpose (e.g., phone conversation record or e-mail). The Laboratory Director, CSM, Technical Director and/or QA Manager are informed of client complaints and assist in resolving the complaint.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. STL Buffalo uses an automated documentation and tracking mechanism for the DQR process which provides a system for trend analysis of repeat complaints. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA department is required to conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client, outlining the issue and response taken is strongly recommended as part of the overall action taken.

The number and nature of client complaints is reported by the QA Manager to the Corporate QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Quality Systems Management Review *(QMP, Section 4.15.2 and SOP AQA-Management Review-45).*

4.9 Control of Non-conformances

Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence on a Job Exception Report, also known as a non-conformance report *(AQA-CA-65)*

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Section Manager, Project Manager or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client is informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative.

4.10 Corrective Action

To consistently achieve technical and regulatory requirements, the laboratory data must be supported by an effective corrective action system. The system must be capable of isolating and rectifying both random and systematic errors. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

Mechanisms used to ensure problem definition include SOPs; internal and external audits and surveillances; and regular laboratory management meetings. When evaluation of performance against established criteria for good laboratory practices shows a condition that could adversely affect the quality of services provided, corrective action is initiated.

Any employee in STL can initiate a corrective action. The initial source of corrective action can also be external to STL (i.e., corrective action due to client complaint, regulatory audit, or PT(s)). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem affects a specific client project, the PM is informed immediately.

All corrective actions, whether immediate or long-term, will comprise the following steps to ensure a closed-loop corrective action process:

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign, and obtain commitment to, responsibility for implementing the corrective action.
- Implement the correction.
- Assess the effectiveness of the corrective action and verify that the corrective action has eliminated the problem.

4.10.1 Immediate Corrective Action

Immediate corrective actions to correct or repair non-conforming equipment and systems are generally initiated in response to adverse conditions identified through QC procedures. The analyst has relatively quick feedback that a problem exists, e.g., calibration does not meet or QC check samples exceed allowable criteria, and can take immediate action to repair the system.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. DQOs are evaluated against laboratory-established or against method or client specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. When the appropriate corrective action measures have been defined and the analytical system is determined to be "in-control" or the measures required to put the system "in-control" have been identified and scheduled, the problem and resolution or planned action is documented in the

appropriate logbook or Job Exception Report. Data generated by an analytical system that is determined to be out-of-control must never be released without approval of the Section Manager, QA Manager, Laboratory Director and client notification.

When an acceptable resolution cannot be met or data quality is negatively affected, the analyst will notify their Section Manager and initiate a Job Exception. If a Job Exception is required, it is routed for proper authorizations and direction. Proper authorization and direction is given by the Project Manager and/or QA Manager. Based upon the circumstances and judgment of the Project Manager, in conjunction with the QA Manager, the client will be notified of the situation.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written Job Exception Report and appropriate corrective action (e.g., reanalysis) is taken and documented.

A Job Exception documents analytical problems at the bench level. This form allows for the documentation of the out-of-control situation, actions undertaken to correct the problem and a return-to-control status. All Job Exceptions are signed/dated by the respective laboratory section manager.

The QA Manager has the authority to stop the analysis, e.g., failure to meet method or project requirements, and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without appropriate documentation leading to the QA Manager's approval and sign-off.

4.10.2 Long-term Corrective Action

Long-term corrective action is generally initiated due to QA issues, which are most often identified during internal and external audits (Sections 4.13 & 4.14). Typically, a deeper investigation into the root cause of the nonconformance is warranted, and the problem may take much longer to identify and resolve. Staff training, method revision, replacement of equipment, and LIMS reprogramming are examples of long-term corrective action.

4.10.3 Responsibility and Closure

The Section Manager is responsible for correcting out-of-control situations, placing highest priority on this endeavor. Associated corrective actions, once verified for effectiveness, are incorporated into standard practices. Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved. Section Managers are accountable to the Operations Manager to ensure final acceptable resolution is achieved and documented appropriately.

The QA Manager also may implement a special audit (Section 4.13). The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure QA policies and procedures are enforced.

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4.11 Preventative Action

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity which can be initiated by clients, employees, business providers, and affiliates. The QA section has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Preventive action opportunities may be identified from information obtained through activities related to but not limited to the corrective action process, performance evaluation program, internal audits, management review, and/or market trends, industry trends and competitive comparisons.

Established standard practices for preventive action are included in the Corrective and Preventive Action SOP *(AQA-CA-35)*; the Data Quality Request SOP *(AQA-DQR-65)* and the Quality System Management Review SOP *(AQA-Management Review-45)*. These procedures describe the information sources used to detect, analyze, and eliminate potential causes of nonconformities and to ensure effective implementation of solutions.

4.12 Records

4.12.1 Record Types

Record types are described in Table 4.

4.12.2 Record Retention

Data reports are filed electronically as .pdf files by job number. Hardcopy COC files are maintained and are filed with the original Job File in job number order.

Laboratory data, project management files, QA records (e.g., PT scores/corrective actions; MDLs/IDLs, statistical analysis, QAPPs, etc..), Human Resources information, etc.., are compiled by date order. The same procedure is followed both in current and archived hardcopy storage.

Upon archiving, a record is made in the Archive Logbook and a number is assigned for each storage box of records. This logbook documents the contents (description and dates) of each storage box. Records are maintained for the periods defined in Tables 5 and 6. On an annual basis, the storage boxes are reviewed and those records subject to disposal are purged.

Table 5 outlines the laboratory's standard record retention time. For raw data and project records, record retention is calculated from the date the project report is issued. For other records, such as Controlled Documents, QC, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 6 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 4. STL Record Types

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records

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Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records
See Section 3. Terms and Definitions	LQMs/ QAPPs	Audits/ Responses	COC Documentation	Accounting
	QMP (Corporate)	Certifications	Contracts and Amendments	Corporate Safety Manual, Permits, Disposal Records
	SOPs	Job Exceptions / DQRs	Correspondence	Employee Handbook
		Logbooks*	QAPP	Personnel files, Employee Signature & Initials, Training Records
		Method & Software Validation, Verification	SAP	
		Standards Certificates	Telephone Logbooks	Technical and Administrative Policies
	Work Instructions	MDL/IDL/IDC Studies	E-mails	
		PTs	Electronic Data Report	
		Statistical Evaluations		

*Examples of Logbook types: Maintenance, Instrument, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, and Balance Calibration.

Table 5. STL Record Retention

Record Type		Archival Requirement *
Raw Data	All* (Electronic Data Reports (.pdf & EDD)	5 Years from completion
Controlled Documents	All*	5 Years from document retirement date
QC	All*	5 Years from archival
Project	All*	5 Years from project completion
Administrative	Personnel/Training	Indefinitely
	Accounting	10 years

* Exceptions listed in Table 6.

4.12.3 Programs with Longer Retention Requirements

Some regulatory programs and clients have longer record retention requirements than the laboratory's standard record retention time. These are detailed in Table 6 with their retention requirements and client-specific requirements are listed in the Record Retention and Storage SOP (*AQA-RecordStorage-56*). In these cases, the longer retention requirement is implemented and

noted in the archive. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 6. Special Record Retention Requirements

Program	Retention Requirement
NY Potable Water NYCRR Part 55-2	10 years
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA – 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement
Louisiana – All environmental data	10 years

4.12.4 Archives and Record Transfer

Archives are indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented.

STL ensures that all records are maintained as required by the regulatory guidelines and per this LQM upon facility location change or ownership transfer. Upon facility location change, all archives are retained by STL in accordance with this LQM. Upon ownership transfer, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. Any further record retention requirements will be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established.

In the event that the laboratory is closed, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. All records will then be transferred to STL's corporate record storage location. All boxes and contents will be appropriately labeled with the dates of destruction (Refer to Tables 5 and 6) and managed in accordance their policies.

4.13 Internal Audits

Quality assurance audits and surveillances are conducted to assess the performance of laboratory systems in meeting technical, regulatory and client requirements; and to evaluate the operational details of the QA program *(Systems Audits; S-Q-002)*. They provide a means for management to be apprised of, and to respond to, a potential problem before it actually impacts the laboratory operations. They also are a mechanism for ensuring closure of corrective actions resulting from external audits.

4.13.1 Audit Types and Frequency

A number of types of audits are performed at STL. These audit types and frequency are categorized in Table 7.

Table 7. Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data	QA Department or Designee	**Data Report Review:** As necessary to ensure an effective secondary review process
		Analyst Data Audits: 100% of all analysts annually
		Electronic Data Audits: 100% of all organic instruments
Special	QA Department or Designee	As Needed

4.13.2 Systems Audits

Systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager. Systems audits cover all departments of the facility, both operational and support. The review consists of laboratory systems, procedures, documentation and issues noted in external audits.

The audit report is issued by the QA Manager within 21 calendar days of the audit. The audit report is addressed to the Operations Manager and Department Supervisors and copied to the Corporate Quality Director and the Laboratory Director.

Written audit responses are required within 30 calendar days of the audit report issue. A maximum of one calendar month is given to address any recommended corrective actions. The audit response is directed to all individuals copied on the audit report. Where a corrective action may require longer than a calendar month to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

4.13.3 Data Audits

Data audits are focused to assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria.

The QA Department provides feedback and/or corrections and revisions to project reports where necessary. Records of the data audits are kept, and the frequency of data audits is included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of

differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client (Section 4.8). The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for permanent corrective action.

The frequency of data auditing may also be dependent upon specific clients and regulatory programs. All active laboratory logbooks and QC files are subject to periodic audits/ surveillances by the QA personnel.

4.13.3.1 Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from the operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. The QA Manager will report the percentage of analysts reviewed (for the year) in the monthly QA report and should average about 8% per month.

4.13.3.2 Electronic Data Audits

Electronic data audits are performed on 100% of all organic instruments by the QA department or a designee independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The QA manager will report the percentage of instruments reviewed (for the year) in the monthly QA report and should average about 8% of instruments per month. Electronic data audits include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6.1.

4.13.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, proficiency testing results, data audits, systems audits, validation comments, or regulatory audits. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

4.14 External Audits

STL is routinely audited by clients and external regulatory authorities – both government and non-government. Whether the audit is scheduled or unannounced, full cooperation with the audit team is provided by the laboratory and administrative staff. STL recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15 Management Reviews

4.15.1 QA Reports to Management

A monthly QA report is prepared by the QA Manager and forwarded to the Laboratory Director and Corporate Quality Director. The reports include statistical results that are used to assess the effectiveness of the quality system. The required information for the monthly report is shown in Figure 3.

4.15.2 Quality Systems Management Review

A quality systems management review is performed at least annually by the Laboratory Director and QA Manager *(SOP AQA-Management Review-45)*. This review ensures that the laboratory's quality system is adequate to satisfy the laboratory's policies and practices, government requirements, certification, accreditation, approval requirements, and client expectations. Quality systems management reviews are accomplished through the evaluation and revision of this LQM, monthly quality assurance reporting and goal setting.

Management reviews of specific quality system elements may be performed through continuous improvement activities, monthly QA reports, process changes, SOP revisions, and/or audit reports/responses. Documentation of these reviews are not required unless it is inherent in the review mechanism (e.g., approval signatures on SOP revisions).

4.15.3 Monthly QA Report and Metrics

By the 3rd day of the month, the QA manager prepares a monthly QA report. The report is sent to the Laboratory Director, General Manager and Corporate Quality Director. The report contains a narrative summary and metrics spreadsheet. At a minimum, the report content contains the items listed below (Figure 3). During the course of the year, the Laboratory Director, General Manager or Corporate Quality Director may request that additional information be added to the report.

Figure 3. Monthly QA Report Format

1	Audits
	Internal System Audits External System Audits
2	Revised Reports / Client Feedback
	Revised Reports Client Complaints Client Compliments
3	Certification Changes
	Changes Losses / Revocations
4	Proficiency Testing
	Study participation and scores Combined PT scores Repeat failures
5	SOP Status
	Report the percentage of SOPs that have been revised or reviewed within the last 24 months.
6	Miscellaneous QA and Operational Issues
	Narrative outlining improvements, regulatory

	compliance issues and general concerns.
Appended	Metrics Spreadsheet
	Summarize metrics in template provided by the Corporate Quality Director

5.0 Technical Requirements

5.1 Personnel

5.1.1 General

STL management believes that its highly qualified and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry. The staff consists of professionals and support personnel that include the following positions:

- General Manager
- Laboratory Director
- Technical Director
- QA Manager
- Human Resource Manager
- Customer Service Manager
- Operations Manager
- QA Specialist
- Health & Safety Coordinator / Waste Management
- Project Manager
- Information Technology Manager
- Network Administrator
- Department Supervisor
- Analyst
- Sample Custodian
- Technician
- Data Reporting Specialist

In order to ensure that employees have sufficient education and experience to perform a particular task, job descriptions are developed for all personnel. Job Descriptions are located on the STL Intranet Site's Human Resources web-page:

http://stlnet.stl-inc.com/Corporate/HR/JobDescriptions/JobDescrip_index.htm.

5.1.2 Training

STL is committed to furthering the professional and technical development of employees at all levels. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for STL employees are outlined in Job Descriptions.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency.

The QA section in conjunction with the Human Resources section are responsible for maintaining documentation of these activities.

Each laboratory section is required to maintain documentation associated with analytical training (e.g., training records, IDOCs, CDOCs, and controlled documents). The QA department maintains documentation of method proficiency (e.g., IDMPs, MDLs, MDLVs, PT Sample Tracking, QC Control Limits/Data). This information is available to managers and staff for planning and evaluation.

The following evidence items are maintained in the employees technical training file for each technical employee:

- An Ethics Agreement signed by each staff member (renewed each year).
- A Confidentiality Agreement signed by each staff member (renewed each year).
- Initial Demonstration of Capability (IDOC)
- The employee has read and understood the latest version of the laboratory's quality documentation.
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- Annual evidence of continued DOC that may include successful analysis of a blind sample on the specific test method; a similar test method; an annual DOC; or four successive and acceptable LCSs.
- Documentation of external training courses attended
- All training regarding QA policies and procedures

The Human Resource department maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Table 8. STL Employee Minimum Training Requirements

Specialty	Experience
General Chemistry and Instrumentation	Six months
Gas Chromatography	One year
Atomic Absorption	One year
Mass Spectrometry	One year
Spectra Interpretation	Two years

Required Training	Time Frame [1]	Employee Type
Environmental Health & Safety	Month 1	All
Ethics	Month 1	All
Data Integrity	Month 1	Technical and PMs
Ethics Refresher	Annually	All
Quality Assurance	Quarter 1	All
Initial Demonstration of Capability	Prior to unsupervised method	Technical

(IDOC)	performance	

[1] From the date of initial employment unless otherwise indicated.

The quality assurance training includes an overview of regulatory programs and program goals, discussions about data integrity and data misrepresentation and an overview of laboratory quality control procedures and purposes.

When an analyst has not met these training requirements, they can perform a task under the supervision of a qualified analyst, peer reviewer or department supervisor, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

Technical training is accomplished by the Operations Manager, Department Supervisor or a senior analyst to ensure method comprehension. All new personnel are required to demonstrate competency in performing a particular method by successfully completing an Initial Demonstration of Capability. IDOCs are performed by the analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the IDOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. A IDOC Certification Statement is recorded and maintained in the employee's training file. Tabulated results summary and raw data are completed and signed by the analyst and section manager with the proper entries made onto the analysts training record. The data are submitted to the QA department for approval and entry into the master IDOC spreadsheet and filing. Figure 4 shows an example of a IDOC Certification Statement.

On an annual basis, each analyst's method capabilities must be evaluated. The requirement that a CDOC (Continued Demonstration of Capability) be completed for each method currently being analyzed must be presented for approval to QA in the same format as the IDOC discussed above.

Further details of the laboratory's training program are described in the SOP related to Laboratory Personnel Training *(AQA-TRAIN-10)*.

5.1.3 Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times; STL has established an Ethics Policy (P-L-006) and an Ethics Agreement (Figure 5). Each employee signs the Ethics Agreement, signifying agreed compliance with its stated purpose. The ethics agreement is required to be re-signed on an annual basis.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize the Company's ability to do work on Government contracts, and for that reason, the Company has a Zero Tolerance approach to such violations.

Ethics is also a major component of STL's quality and data integrity systems. Each employee is trained in ethics within two weeks of hire and quality training within three months of hire. Annually, ethics refresher training will be provided. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the Corporate Quality Director.

Figure 4. Demonstration of Capability Certification Statement

SEVERN TRENT STL

DOC Cert. Statement
Revision 6
October 12, 2005

SEVERN TRENT LABORATORIES - BUFFALO

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee: _____ Page _____ of _____

Method Number: _____ Date: _____

Parameters or Analytes: _____

Initial Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

Trained By: _____

Date training began: _____ Date training completed: _____

Continued Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

I CERTIFY that I have read and understand the SOP identified above. I have also submitted data associated with the demonstration of capability.

_____ _____
Employee Signature Date

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

John Schove _____ _____
Operations Manager Signature Date

Verl Preston _____ _____
Quality Assurance Manager Signature Date

Figure 5. STL Ethics Agreement

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identification, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by Company Policy;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees; and
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of senior management, up to and including the President of STL.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contact or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE:_____ Date: _____

Supervisor/Trainer:_____ Date:_____

5.2 Facilities

The laboratory is a secure facility with controlled and documented access. Access is controlled by keyless entry access cards, locked doors, and a staffed reception area. All visitors sign in and are escorted by STL personnel while at the facility. The laboratory is locked at all times.

The facility is designed for efficient, automated high-quality operations. The laboratory is equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facility, such as hood flow, are routinely monitored and documented.

The facility is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. STL also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc..

5.3 Test Methods

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Routine analytical services are performed using standard EPA-approved methodologies. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices.

5.3.1 Method Selection

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager in the LIMs technical profile. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology.

Most of the test methods performed at STL originate from test methods published by a regulatory agency such as the US EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods. A listing of methods in which the laboratory is capable of performing is listed in the laboratory's Master Methods Index and Preservation Table *(SOP ASR-Psrv-07).*

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.

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-98-003, February 1999.

Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88-039, December 1988, Revised July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992.

Statement of Work for Inorganics Analysis, ILM04.2, ILM05.2 and ILM05.3 USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM04.2 (with OLM04.3 update) and OLC02.1, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

New York State Department of Environmental Conservation, Analytical Services Protocol, NYSDEC ASP.

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Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and establishes an implementation schedule. As such, the laboratory strives to perform only the latest versions of each approved method.

5.3.2 SOPs

STL maintains a Master Index of SOPs *(SOP Master Index)* for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe function and processes not related to a analytical testing (e.g., administrative procedures).

Method SOPs contain the following information, but not necessarily in the order listed:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Identification of Test Method
2. Applicable Matrix
3. Scope and Application, including test analytes
4. Summary of the Test Method
5. Reporting Limits
6. Definitions
7. Interferences
8. Safety
9. Equipment and Supplies
10. Reagents and Standards
11. Sample Collection, Preservation and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Calculations
16. Method Performance
17. Data Assessment and Acceptance Criteria for Quality Control Measures
18. Corrective Actions for Out-of-Control Data
19. Contingencies for Handling Out-of-Control or Unacceptable Data
20. Waste Management/Pollution Prevention
21. References
22. Tables, Diagrams, Flowcharts and Validation Data
23. Changes From Previous Revision

Process SOPs contain the following information, but not necessarily in the order listed.

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Scope
2. Summary
3. Definitions
4. Responsibilities
5. Procedure
6. References
7. Tables, Diagrams, and Flowcharts
8. Changes from Previous Revision

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP Master Index, and records of controlled distribution. SOPs, at a minimum, undergo annual review (12 months). Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

Figure 6. Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories, Inc. (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

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SOP Interim Change Form

The SOP Interim Change Form is used for implementation, documentation, and authorization of changes to SOPs *(Procedure for Writing, Reviewing and Revising SOPs, AQA-SOP-55)*. Immediate changes in SOPs may be necessary to accommodate improvements; to implement acceptable changes in practices; or to correct potential errors in the existing version. The reason for the change will be identified and a detailed description of the procedure change will be presented. Since this form will become part of the referenced SOP, until such time that the SOP is updated, it must be legible and comprehensible. The Interim Change Form must provide an exact description and identify the affected sections.

Once this form is completed and changes are authorized, it becomes an official part of the SOP for which it revises, and is subject to all document control and records management policies.

5.3.3 Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4 Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome.

It is the responsibility of the Operations Manager to present to the QA Manager all applicable method validation studies for review and approval. The documented approval by the Operations Manager, Department Supervisor and QA Manager must be applied to all applicable validation records before the method is released for use. Method verification may require some, but not all, of the activities described in Section 5.3.5.

5.3.5 Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed methods. While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity
Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determines MDLs are described in Section 4.4.3.6 and the corporate procedure for *MDL Policy, (S-Q-003).*

Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)
An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system.

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When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data are to be reported in this region, they must be done so with a qualification that denotes the semi-quantitative nature of the result.

Determination of Interferences
A determination that the method is free from interferences in a blank matrix is performed.

Determination of Range
Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Demonstration of Capability
DOCs are performed prior to method performance.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

Documentation of Method
The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

Continued Demonstration of Method Performance
Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS and Method Blanks.

5.3.6 Data Reduction and Review

Analytical data are entered/downloaded directly into LIMS or recorded on pre-formatted bench sheets that are paginated and bound into laboratory logbooks. These logbooks are issued and controlled by the laboratory's QA Section. A unique document control code is assigned to each book to assure that chronological record keeping is maintained. Analytical data may also be electronically stored as a secure .pdf file.

Analytical data are referenced to a unique sample identification number for internal tracking and reporting. Both LIMS entries and logbook pages contain the following information, as applicable: analytical method, analyst, date, associated sample numbers, standard concentrations, instrument settings, and raw data. Entries are in chronological order and maintained so as to enable reconstruction of the analytical sequence.

The analyst is responsible for entering / recording all appropriate information, and for signing and dating all logbook entries daily. All entries and logbook pages are reviewed for completeness by a supervisor, peer reviewer or the analyst themselves. Data review checklists document the analytical review of the LIMS entries, logbook and associated QC indicators. Copies of instrument outputs

(chromatograms, mass spectra, etc.) are maintained on file or electronically with the analyst's signature/initials and date.

5.3.6.1 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data are reduced by the analyst and updated to the LIMs. Both the data entry and raw data are then verified by the department supervisor or alternate analyst. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the data and manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the STL Corporate SOP entitled *Acceptable Manual Integration Practices (S-Q-004).*

Copies of all raw data and the calculations used to generate the final results, such as bound logbooks, are retained on file for a minimum of 5 years or as otherwise requested by the client/project.

Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

5.3.6.2 Data Review

All data, regardless of regulatory program or level of reporting, are subject to a thorough review process. The individual analyst continually reviews the quality of the data through calibration checks, quality control sample results and performance evaluation samples. Data review is initiated by the analyst during, immediately following, and after the completed analysis.

All levels of the review are documented on Data Review Checklists that are specific to each laboratory section *(Technical Data Review; AGP-DataReview-21).*

Primary Review

The primary review is often referred to as a "bench-level" review. In most cases, the analyst who generates the data (e.g., logs in, prepares and/or analyzes the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

Once an analysis is complete, the primary reviewer ensures, where applicable, that:

- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.

- Manual integrations are appropriate.
- Data flags to indicate manual integrations are recorded.
- Manual integrations are authorized by a date and signature or initials of primary analyst.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- COC procedures have been followed.
- Primary review is documented by date and initials/signature of primary analyst.
- All unused portions of hardbound logbooks are 'Z'ed out; corrections are made with a single line drawn through the error and are dated and initialed

Any anomalous results and/or non-conformances noted during the Primary Review are documented on the Data Review Checklist and on a Job Exception ; and are communicated to the Supervisor and the Project Manager for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 4.9. Case narrative comments are generated by the primary reviewer for any unresolved anomalous results or non-conformances.

Secondary Review

The secondary review is also a complete technical review of a data and is performed by the Supervisor, peer analyst or data specialist. The secondary review is documented on the same Data Review Checklist as the primary review.

The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms
- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature (hardcopy or electronic) of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions
- Review and approve case narrative comments

If problems are found during the secondary review, which are documented on the data review checklist, the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

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Completeness Review
The completeness review includes the review of the case narrative which outlines anomalous data and non-compliances using project narrative notes, Job Exceptions and DQRs generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?

Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

The laboratory Department Supervisor, Data Reporting personnel and the Project Manager contribute to the completeness review.

5.3.7 Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability
Access to the laboratory's LIMS system that collects, analyzes, and processes raw instrumental data, and those that manage and report data is both controlled and recorded. System users are granted access levels that are commensurate with their training and responsibilities.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. The system has the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability (e.g., Enviroquant, Chemstation, TotalChrom).

Verification
All the LIMS software programs have been verified prior to use and prior to the implementation of any version upgrades. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The verification of LIMS software programs are conducted by the Information Technology Manager with the assistance of the QA Manager, Operations Manager and the Department Supervisors. The IT Manager documents the approval of the program verifications. All records of the verification are retained as QC records.

Validation

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Software validation involves documentation of the verification of final calculated results. Software validation is performed by the QA manager on all in house programs. Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

The QA manager must retain documentation of the validation process as defined above. The designated LIMS methods administrator at the laboratory has the responsibility to validate any LIMS methods, calculations or criteria codes prior to use for sample analysis.

Auditing
STLs LIMS System Managers continually review the control, security, and tracking of IT systems and software.

Version Control
The laboratory maintains copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of 5 years from its retirement date. The associated hardware, required to operate the software, is also retained for the same time period.

5.4 Equipment

5.4.1 Equipment Operation

STL is committed to routinely updating and automating instrumentation. The laboratory maintains state of the art instrumentation to perform the analyses within the QC specifications of the test methods. The laboratory maintains an Equipment List *(STLBuffEquipList)* that documents the following information:

- Identity
- Date In Service
- Manufacturer's Name, Model Number, Serial Number
- Current Location

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks.

5.4.2 Equipment Maintenance

STL employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded.

Any item of equipment or instrumentation that has been subjected to overloading or mishandling, provides suspected results, has been shown by verification or otherwise to be defective, is new or

not been used for an extended period of time, is taken out of service and tagged as "OUT-OF-SERVICE", *(AGP-OutofService-65)*

Any instrumentation that is brought back on-line must have MDLs and DOCs performed and have acceptance within prescribed criteria; or calibrated by a certified agency (e.g., balances or Class S weights) and tagged as being within calibration specifications; and proven to provide consistent measurements (e.g., refrigerators, eppendorf pipettes, ovens).

The return to analytical control following instrument repair is documented in the maintenance logbook. Notation of the date and maintenance activity is recorded each time service procedures are performed. Maintenance logbooks are retained as QA records.

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory. Table 9 lists STL's major equipment and the suggested maintenance procedures.

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Change dryer tube	As Needed
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP & ICP/MS	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check re-circulator levels	Monthly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Change pump oil	Monthly
	Change Cones	As required
	Change printer cartridge	As required
	Replace pump tubing	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Annually
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Agilent GC/MS	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed power wires and insulation	As Required
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples and solvents	Daily
	Change autosampler rotor/stator	As required
Balances	Class "S" traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	At least Annually

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Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Conductivity Meter	0.01 M KCl calibration Conductivity cell cleaning	Weekly As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Check conductivity Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Daily Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Weekly As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

5.4.3 Equipment Verification and Calibration

All equipment is calibrated prior to use (Initial Calibration) to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. All sample measurements are made within the calibrated range of the instrument and in compliance with method requirements. The calibration data, which includes instrument conditions and standard concentrations, is documented in pre-formatted instrument injection logs or within LIMS itself. The preparation of all reference materials used for calibration is documented in standards preparation logbooks in accordance with *SOP AGP-STD-14 (Standards Traceability and Preparation Logbooks).*

Once an instrument is calibrated, ongoing instrument calibration is demonstrated (Continuing Calibration) at the appropriate frequency as defined in the test method. Refer to the STL Corporate Policy *Selection of Calibration Points (P-T-001),* for guidance on using calibration data. Any instrument that is deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

5.4.3.1 Instrument Calibration

Specific instrument calibration procedures for various instruments are summarized further in this section, and detailed in the respective analytical methods. Typically, more than one analytical method is available for an analysis. These various methods and other program requirements (e.g., U.S. EPA CLP, AFCEE, USACE, QAPPs, contracts, etc.) may specify different calibration requirements. Therefore, calibration details as specified in the respective laboratory SOPs, Technical Profiles, QAPP, program requirements, and contracts supersede the general instrument calibration procedures are described further in Table 10. Complete details are provided in each method SOP.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Metals (ICAP)	Initial Calibration	Following a period of time sufficient to warm up the instrument, the ICP is calibrated prior to each analytical run or minimally every 24 hours. Calibration standards are prepared from reliable reference materials and contain all metals for which analyses are being conducted. Working calibration standards are prepared fresh daily. Prior to an analytical run, the instrument is calibrated using appropriate standards. An Initial Calibration Verification (ICV) standard is analyzed immediately after standardization, followed by an Initial Calibration Blank (ICB). The ICV is from a source other than that used for initial calibration and the ICB must be free of target analytes at and above the value to be reported or appropriate corrective action must be taken. ICP Interference Check Samples (ICSAB) are analyzed at the frequency described in each method SOP.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a Continuing Calibration Verification (CCV) standard and a Continuing Calibration Blank (CCB). The response of the CCV must be within the SOP-specified criteria (e.g., ± 10% recovery of the true value). The CCB must be free of target analytes at or above the value to be reported or appropriate corrective action must be taken. If any ICVs/CCVs or blanks exceed their acceptance criteria, appropriate corrective action must be taken.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Inorganic Colorimetric Methods	Initial Calibration	A full initial standard calibration curve will be prepared for all colorimetric analyses. Working standards to define this curve will include a minimum of five (5) concentrations which cover the anticipated range of measurement, plus a calibration blank. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit as defined in Section 8.6 or a level suitable for meeting specific program requirements. The requirement for an acceptable initial calibration is described in the analytical SOP. If the criteria are not met, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, is entered into the laboratory notebook, or associated instrument printouts, and retained with the sample data. If the initial curve is not analyzed that day, a daily calibration verification must be analyzed. This daily calibration will at a minimum consist of a blank and a mid-range standard. Results must be within SOP-specified criteria. If not, reanalysis of the standards may be done once to verify the readings; otherwise, a new curve will be developed.
Inorganic Colorimetric Methods (cont'd.)		For procedures that require pretreatment steps, a minimum of one standard shall be prepared with the pretreatment. If the pre-treated standard is within SOP-specified criteria, the curve will be used. If the pre-treated sample is not within the criteria, the reason will be determined. If it is determined that the difference between the curves is inherent in the procedure, the curve will be based on the standards prepared and carried through the pretreatment. An ICV will be analyzed immediately after the standardization, followed by an ICB. The ICV must be from a source other than that used for initial calibration. The ICV must be within SOP-specified criteria and the ICB must be free of target analytes or appropriate corrective action must be taken.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a CCB and a CCV. If any ICVs/CCVs or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are evaluated for acceptability or reanalyzed. (If the CCV is elevated and the samples are ND, the data are deemed acceptable.)
Ion Chromatography	Initial Calibration	The ion chromatograph will be calibrated every three months or sooner if calibration verification can not be achieved. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the anticipated range of measurements. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit. If SOP-specified calibration criteria cannot be achieved, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, will be archived with sample raw data.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration Verification	A calibration verification standard and blank will be analyzed each day prior to sample analysis, throughout the sequence at a frequency of 10% and at the end of the analysis shift. The response calculated as a percent recovery of the standard must meet SOP or program-specific criteria. The response of the blank must be less than the concentration to be reported for samples analyzed.
GC/MS	All GC/MS instrumentation is calibrated to set specifications prior to sample analysis. These specifications vary depending on the requirements of the analytical program and the designated analytical method.	
	Tuning and Mass Calibration	Mass spectrometers are calibrated with perfluorotributylamine (FC-43) or perfluorophenanthrene (FC- 5311) as required to ensure correct mass assignment. In addition, at the beginning of the daily work shift, the GC/MS system must be tuned with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and 4-bromofluorobenzene (BFB) for volatiles analysis, and calibrated to target compounds. Autotunes are run with PFTBA(perfluorotributylamine),which is encased in a vial inside the mass spec. DFTPP and BFB are run daily (12 hours where appropriate) for SVOA and VOA respectively.
GC/MS (cont'd.)	Tuning and Mass Calibration	Laboratory work using SW-846 protocols, defines the work shift as a 12-hour period initiated by the injection of DFTPP or BFB. For drinking water programs (500 series methods), a 12-hour work shift is specified in the method for calibration frequency. For wastewater programs (600 series methods), the tune expires when the day's analytical sequence is complete; however, no time limit is given for the length of the daily GC/MS work shift; therefore a maximum of 24 hours for 624 and 625 is used. Ion abundances will be within the windows dictated by the specific program requirements.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Initial Calibration	After an instrument has been tuned, initial calibration curves (generally 3-5 points) are generated for the compounds of interest. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards. Instrument response to these target compounds are evaluated against SOP-specified criteria. Linearity is verified by evaluating the response factors (RF) for the initial calibration standards against SOP-specified criteria. Once an acceptable calibration is obtained, samples may be analyzed up until the expiration of the tune. At that time, the instrument must be re-tuned prior to further analysis. After acceptable tuning, a continuing calibration standard may be analyzed in lieu of a full multi-point calibration if the SOP-specified criteria are met. The majority of compounds analyzed for GC/MS comprise EPA's Target Compound List (TCL) or Priority Pollutant List (PPL). For add-on compounds not on the current TCL or PPL, initial calibration may be performed using a single point calibration of the additional compound(s), unless prior arrangements are made for a full three-to-five point calibration. Calibration data, to include linearity verification, will be maintained in the laboratory's records of instrument calibrations. 3 to 5 point curves for all GCMS analytes, special list. The only case where single point standards are used is for quantitation of PCBs other than Arochlor 1016 or 1260.
	Continuing Calibration	During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations, as defined in the specific SOPs. If criteria cannot be met, appropriate corrective action must be taken.
GC and HPLC	Gas chromatographs and high performance liquid chromatographs will be calibrated prior to use as described in analytical SOP or program requirements. Calibration standard mixtures will be prepared from appropriate reference materials and will contain analytes appropriate for the method of analysis or program requirements	
GC and HPLC (cont'd.)	Initial Calibration	Initial calibration will include three or more calibration standards covering the anticipated range of measurement. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards.

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Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration	The response of the instrument will be verified for each analysis sequence by evaluation of a daily calibration verification standard at a mid-range concentration. In order to demonstrate that the initial calibration curve is still valid, the calibration check standard must be within SOP or program-specified acceptance criteria for the compounds of interest or the instrument must be recalibrated. For multi-analyte methods, this check standard may contain a representative number of target analytes rather than the full list of target compounds. Optionally, initial calibration (e.g., the full range of concentration levels) can be performed at the beginning of the analysis sequence. Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard every ten samples or 12 hour sequence (depending on the method protocol), including external QC. If the SOP or program-specified calibration criteria are not met for the compounds of interest, appropriate corrective action must be taken.

5.5 Measurement Traceability

5.5.1 General

Traceability of measurements is assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard is subject to ongoing certifications of accuracy.

At a minimum, these include procedures for checking specifications for balances, thermometers, temperature, De-ionized (DI) and Reverse Osmosis (RO) water systems, automatic/eppendorf pipettes and other volumetric measuring devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards [with the exception of class A glassware (including glass microliter syringes that have a certificate of accuracy)].

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balances are calibrated on each day of use. All thermometers and temperature monitoring devices are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use

Laboratory DI and Elga water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use

Procedures for maintenance and record keeping of support equipment are defined in SOP *Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devices and Reagent Water (AGP-SupportEquip-02)*

5.5.2 Reference Standards

The receipt of all reference standards is documented in the departmental Chemical History Logbook. Standards are obtained from commercial vendors and sources may vary depending upon the availability of mixes and solutions from vendors. Each production unit is responsible to ensure, when available, that all standards are traceable to EPA, NIST or A2LA and are accompanied by a Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis.

The receipt of each dry chemical, purchased stock solution or reference material to be used as a standard is assigned a unique ID number. The chemical name, manufacturer, lot number, date received, expiration date, date opened and initials of the analyst who opened the chemical are documented. The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory labels all standard and QC materials with expiration dates.

The preparation of all daughter solutions, whether a single or multiple-component stock, intermediate, or working standard solution, is documented in a standard solution preparation logbook. This documentation references the Standard ID of the respective parent solution(s) used in its preparation, providing a solid trail back to the solution or chemical received from the vendor. These records include the standard name, final volume, matrix, final concentration, analyst initials, prep date and expiration date. A daughter solution should not have an expiration date which post-dates any of the parent solutions used in its preparation.

References standards are labeled with a unique Standard Identification Number, date received, and the expiration date. All documentation received with the reference standard or documentation of standard purity is retained as a QC record and references the Standard Identification Number. All efforts are made to purchase standards that are $\geq$ 97.0% purity. If this is not possible, the purity is used in performing standards calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate QC criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an ICV or LCS is used as the second source confirmation.

Storage conditions, such as shelf life, ambient or chilled, controlled or restricted access, wet or desiccated, etc., are in conformance with the specifications set in the associated method, the program requirements, or the manufacturer's recommendation, as appropriate.

5.5.3 Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be, at a minimum, the purity required in the test method. The date

of reagent receipt and the expiration date as well as the date of reagent preparation (where applicable) are documented in the standards preparation logbooks.

5.6 Sampling

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7 Sample Handling, Transport, and Storage

5.7.1 General

COC can be established either when bottles are sent to the field, or at the time of sampling. STL can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory. Complete details for sample container preparation are contained within *Sample Container Preparation and Shipment SOP (ASR-Bottle-03)*. A summary of sample receipt is as follows with complete details available within the *Receipt of Analytical Samples SOP (ASR-Receipt-05).*

Samples are received at the laboratory by the designated sample custodians and a unique LIMS job number is assigned. The following information is recorded for each sample shipment:

- Client/Project Name.
- Date and Time of Laboratory Receipt.
- Laboratory Job Number
- Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{\circ}C$ (for samples with a temperature requirement of $4^{\circ}C$, a cooler temperature of just above the water freezing temperature to $6^{\circ}C$ is acceptable); sample receipt is considered "compromised" and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 is documented in an Analytical Receipt Resolution Form (ARRF) and brought to the immediate attention of the Project Manager for resolution with the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor are repackaged, iced, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Refrigerated storage coolers are maintained at $4 \pm 2^{\circ}C$ and the temperatures are monitored daily.

All samples are stored according to the requirements outlined in the test method, and in a manner such that they are not subject to cross contamination or contamination from their environment.

Access to the laboratory is restricted to laboratory personnel or escorted guests as described in Section 5.2. Therefore, once sample possession is relinquished to the laboratory, the sample is in a designated secure area (e.g., the laboratory facility) accessible only to authorized personnel. Locked storage coolers are available for protocol that require internal COC procedures.

5.7.2 Sample Identification and Traceability

The sample custodian organizes the sample containers, COCs, and all pertinent information associated with the samples. The sample identity is verified against all associated sample information. Any inconsistencies are documented via an ARRF and forwarded to the Project Manager for resolution with the client prior to identifying the sample(s) into LIMS.

Each sample container is assigned a unique Sample Identification Number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label.

All unused portions of samples, including empty sample containers, are returned to the secure sample control area, unless it has been documented that the container was disposed.

5.7.3 Sub-Sampling

Taking a representative sub-sample from a container containing a soil or solid matrix is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation.

After thoroughly mixing the sample within the sample container or transfer to a suitable plastic bag, a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight. Any non-homogenous looking material is avoided and noted as such within the sample preparation record.

5.7.4 Sample Preparation

Sample preparation procedures vary for each matrix and analytical method are as referenced in the laboratory SOPs.

5.7.5 Sample Disposal

Samples are retained in STL storage facilities for 30 days after the project report is sent unless prior written arrangements have been made with the client. Samples may be held longer or returned to the client per written request. Unused portions of samples are disposed of in accordance with federal, state and local regulations. The laboratory removes or defaces sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). Complete details on the disposal of samples, digestates, and extracts is available within the *Sample Disposal SOP (ASR-DISP-33) and Hazardous Waste Management SOP (AWM-HazMg-01).*

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5.8 Assuring the Quality of Test Results

5.8.1 Proficiency Testing

The laboratory analyzes Proficiency Test (PT) samples as required for accreditation and as outlined in NELAC. The laboratory participates in the PT program semi-annually for each PT field of testing for which it is accredited, according to the NELAC PT field of testing published guidelines. This includes drinking water, wastewater and solid/soil matrices.

The laboratory also participates in various client PT programs, when submitted.

PT samples are handled and tested in the same manner (procedural, equipment, staff) as environmental samples. Results of PT samples are distributed to the laboratory line management for review and action, if required. Any required response to deficiencies are submitted to the QA department for review and are filed with the PT study records. PT test sample data are archived using the requirements for project and raw data record retention.

5.8.1.1 Double Blind Performance Evaluation

The laboratory participates in an annual double blind performance evaluation study. An external vendor is contracted to submit double blind samples to the laboratory. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Corporate Quality Director and to the laboratory. This is administered as a double blind program in order to assess all facets of the laboratory's operations.

5.8.2 Control Samples

Control samples (e.g., QC indicators) are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Control samples must be uniquely identified and correlated to unique batches. Control samples further evaluate data based upon (1) Method Performance, which entails both the preparation and measurement steps; and (2) Matrix Effects, which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Control sample types and typical frequency of their application are outlined Sections 5.8.2.1 through 5.8.2.5 and Tables 11 through 15. Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method and regulatory program control samples are as listed in Sections 7 and 8 typically of each method SOP.

5.8.2.1 Method Performance Control Samples: Preparation Batch

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment.

Control samples are added to each prep batch to monitor method performance (Table 11) and are processed through the entire analytical procedure with investigative/field samples.

Table 11. Preparation Batch Control Samples

Control Sample Type	Details	
Method Blank (MB)	Use	Monitors for potential contamination introduced during the sample preparation and analytical processes.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method.
	Description	Organics: Laboratory pure water for water samples or a purified solid matrix for soil or solid samples (when available or when requested); solid matrices commonly include sodium sulfate, vendor or agency supplied soil or solid, or purchased sand; these solids may require purification at the laboratory prior to use. Inorganics: Laboratory pure water for both water and soil or sediment samples. Volume/weights are selected to approximately equal the typical sample volume/weight used in sample preparation; and final results in a soil/solid batch may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison to actual field samples.
Laboratory Control Sample (LCS)	Use	Measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method. For multi-analyte methods, the LCS may consist of surrogates in the blank matrix, and or a representative selection of target analytes/internal standards.
	Description	Prepared from a reference source of known concentration and processed through the preparation and analysis steps concurrently with the field samples. Aqueous LCS's may be processed for solid matrices unless a solid LCS is requested; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the actual field samples.
Known QC Sample	Use	Comply with regulatory requirements; check the accuracy of an analytical procedure; troubleshoot method performance problems; verify an analyst in training's ability to accurately perform a method; to verify the return-to-control after method performance problems; and may also be used as an LCS.
	Typical Frequency [1]	As defined by the client or QAPP.
Known QC Sample (cont'd.)	Description	Obtained from outside suppliers or agencies; generally require preparation from concentrated materials by dilution into a standard matrix; contain known analytes or compounds; acceptance limits are provided by the vendor.

[1] Denotes an STL required frequency.

Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. However, a field blank should not be selected for matrix QC, as it does not

provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

5.8.2.2 Method Performance Control Samples: Matrix

Matrix control samples include sample duplicates (MD), sample matrix spikes (MS), and sample surrogate spikes. These control samples help monitor for potential physical and chemical effects which may interfere with the precision and/or accuracy of the selected analytical method. Since interferences can enhance or mask the presence of target analytes, matrix control samples measure the degree of interference and are used to assist in the interpretation of the analytical results. The laboratory avoids performing matrix QC on known field blank samples, such as trip blanks and rinsates, since these samples are not indicative of the sample matrix.

Table 12. Matrix Control Samples

Control Sample Type	Details	
Matrix Duplicate (MD)	Use	Monitors the effect of site matrix on the precision of the method; and of the reproducibility of laboratory preparation and measurement techniques. **Note:** Precision may also be affected by the degree of homogeneity of the sample, particularly in the case of non-aqueous samples or aqueous samples with particulates. Sample homogeneity and matrix effect should be considered when field samples are used to assess reproducibility. **Note:** A field duplicate, when received, measures Representativeness of sampling and the effect of the site matrix upon precision.
Matrix Duplicate (MD) (cont'd.)	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP [2].
	Description	Performed by analyzing two aliquots of the same field sample independently; analyzed for each associated sample matrix (e.g., when requested by the client or the analytical method).
Matrix Spike (MS)	Use	Measures the effect of site sample matrix on the accuracy of the method.
Matrix Spike (MS) (cont'd.)	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP.
	Description	Aliquot of a field sample which is spiked with the analytes or compounds of interest; analyzed for each associated sample matrix (when requested by the client or analytical method). The determination of MS percent recovery (% R) requires an analysis of a fortified sample and a non-fortified sample under the same procedural conditions (e.g., sample volumes, dilutions, procedural conditions, etc.). The concentration determined in the non-fortified sample is subtracted from the fortified sample concentration before determining the %R. The degree of homogeneity of the sample, particularly in the case on non-aqueous samples or samples with particulates, may affect the ability to obtain representative recoveries.
Matrix	Use	Measures effect of site sample matrix on precision of method.

Table 12. Matrix Control Samples

Control Sample Type	Details	
Spike Duplicate (MSD)	Typical Frequency [1]	1 per 20 samples per matrix, when requested by the client or the analytical method, or per SAP/QAPP [2].
	Description	Alternative to sample duplicate. Generally, inorganic protocols specify an MD/MS and organic protocols specify an MS/MSD.
Surrogate Spike	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Every QC and analytical sample.
	Description	Compounds similar to the target analytes in structure, composition and chromatography, but not typically found in the environment, are added to each QC and analytical sample, prior to preparation (e.g., extraction). If the surrogates in an analytical batch do not all conform to established control limits, the pattern of conformance in investigative and control samples is examined to determine the presence of matrix interference or the need for corrective action.
Internal Standards	Use	Monitor the qualitative aspect of organic and inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] Denotes an STL required frequency.
[2] Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

5.8.2.3 Matrix QC Frequencies

The frequency of matrix QC indicators depends on regulatory program compliance, a project's data quality objectives, or a client's requirements. The following frequency will be applied to samples when the regulatory programs are known and it does not conflict with project or client requirements.

Table 13. EPA Program Requirements

Program	Description [1]
SDWA	MD performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent.
CWA	MS (GC methods) and MD is performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent. For GC/MS Methods, MS is performed at a 5% frequency or 1 per preparation batch of ≤20 samples, whichever is more frequent.
RCRA	MS/MSD or MS/MD is performed at a rate of 5% per client (independent of the preparation batch). For clients submitting less than 10 samples, the method matrix QC requirement may be satisfied by another client's sample within the same prep batch unless the paperwork indicates a client requirement for matrix QC.
U.S. EPA CLP	MS/MSD or MS/MD is performed at a rate of 5% or 1 set per Sample Delivery Group (SDG) per matrix, independent of the prep batch. Samples are processed in simultaneous or continuous batches.

[1] MS, MSD and MD may not be applicable to some analytical protocols because of the nature of the sample or protocol.

5.8.2.4 Method Performance Control Samples: Instrument Measurement

Control samples are used to ensure that optimum instrument performance is achieved. These samples help ensure that the proper identification and quantitation of target compounds or analytes are achieved. The instrument control samples appropriate to each analytical technique are described in laboratory SOPs for each respective method. A brief description of these checks is included in Table 14.

Table 14. Instrument Performance Control Samples

Control Sample Type		Description
		Inorganics
ICV	Use	Calibration standard of known concentration prepared from a source other than that used for the calibration standards.
	Sequence	Analyzed after the standard curve to confirm calibration.
ICB	Use	Blank water or solvent; confirms the calibration and assures that any potential contamination is less than the reporting limit.
	Sequence	Analyzed immediately after the ICV.
ICP Interference Check Samples (ICSA/ICSB)	Use	Verifies the absence of spectral interferences.
	Sequence	Analyzed consecutively at the beginning of each eight hour analytical sequence, after the ICV/ICB, and again at an eight hour frequency following a CCV/CCB. When CLP protocols are followed, the ICSA/B will be analyzed with the analytical sequence, before the final CCV/CCB.
Reporting Limit Verification	Use	Verifies linearity near the reporting limit for CLP metals analyses. (Note: CRI is at a level 2X the CRDL; CRA is near the CRDL).
Standard (CRA and CRI)	Sequence	Performed only when analyzing CLP Samples or as specified by the client or program. Analyzed after the ICB. The CRI is also analyzed at the end of the eight hour analytical sequence, prior to analysis of the final CCV/CCB.
CCV	Use	Confirm that the instrument performance has not significantly changed during the analytical sequence; to verify stable calibration throughout the sequence; and/or to demonstrate that instrument response did not drift over a period of non-use. May be made from a source other than that used for the standard curve, however if the ICV is 2nd source, the CCV may be same source.
	Sequence	Analyzed at 10% or every two hours, whichever is more frequent; also analyzed at the end of the analytical sequence.
CCB	Use	Water blank used to confirm that the baseline has not drifted and to monitor for contamination at the reporting limit.
	Sequence	Analyzed at a rate of 10% for inorganics and at a rate of 1 per 10 readings/injections or every two hours, whichever is more frequent, for CLP metals; also analyzed at the end of the analytical sequence.
ICP Metals Linear Range	Use	Verify linearity and document the upper limit of the calibration range for each element.

Table 14. Instrument Performance Control Samples

Control Sample Type	Description	
Analysis Standard (LRS)	Sequence	Performed quarterly with a blank and a minimum of five standard concentrations to cover the anticipated range of measurement; one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient $\geq$ 0.995 in order to consider the responses linear over that range.
ICP Inter-Element Correction (IEC)	Use	Correction factors for spectral interference (particularly due to Al, Ca, Fe, and Mg).
	Sequence	Determined at least annually for all wavelengths used for each analyte reported by ICP; or any time the ICP is adjusted in any way that may affect the IECs.
	Organics	
GC/MS Tuning & Performance	Use	Ensures correct mass assignment and is monitored through response to target compounds during initial and continuing calibration, with minimum response criteria for specified system performance check compounds (SPCCs), and linearity is verified by evaluating the response factors (RF) for calibration check compounds (CCCs).
	Sequence	Tuned at the beginning of the daily work shift. Throughout the analysis, blanks, internal standard areas, surrogates, chromatographic baseline, resolution of peaks, and overall quality of the chromatography are used collectively to monitor instrument performance.
GC & HPLC Instrument Performance	Use	Monitored through retention time shift evaluation, linearity checks, and degradation checks of selected target compounds (e.g., for Endrin or DDT as appropriate).
GC & HPLC Instrument Performance	Sequence	Continuing calibration verification (e.g., blanks, shifts in chromatographic baseline or retention times, resolution of peaks, and overall quality of the chromatography) throughout the analytical sequence is accomplished through analysis of calibration check standards.

5.8.2.5 Method Performance Control Samples: Analysis Batch

Matrix specific control samples are used to assess the precision and accuracy of the method as applied to the specific sample matrix. These indicators provide information on sample matrix effects that is independent of the efficiency of the preparatory technique. The method performance control samples appropriate to each analytical technique are identified in the respective method. A brief description of these checks is included in Table 15.

These control samples are performed to provide a tool for evaluating how well the method performed for the respective matrix. These values are used by the client to assess the validity of a reported result within the context of the project's data quality objectives. For matrix specific QC results falling outside laboratory control limits which are attributed to matrix affects, no systematic corrective action is taken.

Table 15. Analysis Batch Performance Control Samples

Control Sample Type	Description	
ICP Serial Dilution	Use	5X Dilution of a field sample (performed at the instrument) to check for possible physical and/or chemical interferences.
	Sequence	5% of field samples or 1 per ≤20 samples per batch.
Method of Standard Addition (MSA)	Use	When specified by the analytical protocol or by client request.
	Sequence	When specified by the analytical protocol or by client request.

5.8.3 Statistical Control Limits and Charts

Statistical control limits and control charts are used to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

Establishment of Limits

The purpose of using statistical control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate control samples in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:

Accuracy

As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and shall be investigated.

Roughly 95% of points should fall within 2s of the mean. The values +2s and -2s are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

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Precision

Precision is used to indicate matrix variability so that appropriate decisions can be made by the client when repeated analyses vary significantly. The coefficient of variation, expressed as a percentage (e.g., the %RSD) for the data set used to calculate accuracy control limits defines the control limit for precision. Duplicate analyses of the QC samples, such as duplicates or MS/MSD, should have an RPD less than or equal to this established precision control limit to be considered free of matrix interferences.

The laboratory calculates statistical control limits on an annual basis, or more frequently if change have been made to the analytical process which affects the chemistry of the method. Such limits are available on a project or QAPP-specific basis.

5.8.4 Calibration

Calibration protocols are method-specific, are briefly described in Table 10 and are defined in the Sections 6 & 7 of the method SOPs.

5.8.5 Glassware Cleaning

All glassware is thoroughly cleaned prior to use to ensure that sample integrity is not affected from artifacts caused by contaminated glassware.

A summary of general cleaning procedures follows with details provided in the *Laboratory Glassware Cleaning* SOP *(AGP-Glass-04):*

General laboratory glassware is cleaned with a low- or non-phosphate detergent, followed by thorough rinsing with tap water and deionized water.

Volumetric flasks and pipettes used for inorganics (method dependent), test tubes and caps used for micro-COD procedures, phosphate glassware, and metals-related glassware include an acid-washing step.

BOD glassware, includes use of EPA approved disposable plastic bottles or cleaning with a nitric or sulfuric acid and/or a NOCHROMIX-washing step.

Organic glassware includes a solvent-wash.

5.8.6 Permitting Departures from Documented Procedure

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure is documented in a Job Exception and reported in the case narrative. In most cases, these departures can be made with the approval of the Department Supervisor, Project Manager and the client. Issues of serious concern, as determined by the Operations Manager, Department Supervisor or Project Manager, will be brought to the attention of the Laboratory Director and/or QA Manager. In some instances, it is appropriate to inform the client before permitting a departure. The Project Manager, in consultation with the QA Manager, will make the determination as to the degree of notification required by the client.

On rare occasions, special analytical techniques will be requested for research, project specific requirements, or client needs. In these instances, SOPs may not be available, however, the analyst will thoroughly record the analytical steps and observations within a bound preformatted logbook.

5.8.7 Development of QC Criteria, Non-Specified in Method/Regulation

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must examine the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of STL's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc.).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory to test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, Accuracy $\pm$25%, and RSD of <30%. The laboratory may opt to develop a method that meets these criteria and document through the Method Blank results, MDL study, and LCS results that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

5.9 Project Reports

The SOP for data package assembly and reporting formats is *ARP-Report-125* and a summary of this procedure follows.

Analytical reports comprise final results (uncorrected for blanks and recoveries unless specified), methods of analysis, levels of reporting, surrogate recovery data, and method blank data. In addition, special analytical problems will be noted in the case narratives. The number of significant figures reported are consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed.

Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter, mg/L). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per kilograms, ug/kg). Reporting limits take into account all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified by program requirements (e.g.,IRPMS reports).

A client report is generated with various steps of approval prior to printing of the final version. If any analytical anomalies were encountered during the analyses, e.g., an out-of-control matrix duplicate, it is documented in a case narrative. The case narrative is prepared by the respective operating unit, project manager, or other designated personnel and inserted in the final report.

The final report forms are printed, data packages are organized, a glossary of flags and acronyms is added, and reports are paginated.

5.9.1 General

The criteria described in Section 5.9.2 apply to all Project Reports that are generated under NELAC requirements. The criteria described in Section 5.9.3 and 5.9.4 apply to all Project Reports.

5.9.2 Project Report Content

- Title
- Laboratory name, address, telephone number, contact person
- Unique Laboratory Project Number
- Name and Address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Methods
- Report Paginated

The following are required where applicable to the specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight
- Whole Effluent Toxicity: Statistical package used
- If holding time $\leq$ 48 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging or narrative comment where results are reported below the quantitation limit.

5.9.3 Project Narrative

A Project Narrative and/or Cover Letter is included with each project report and, at a minimum, includes an explanation of any and all of the following occurrences:

- Listing of any subcontracted analyses and subcontractor location
- Non-conformances

- "Compromised" sample receipt (see Section 4.7.1)
- Method Deviations
- QC criteria failures
- Any authorized SOP deviations, non-conformances and QC failures must be covered in the case narrative, cover letter or within the report.

Project Release

The Project Manager or his designee authorizes the release of the project report with a signature.

Where amendments to project reports are required after issue, these are documented in the form of a DQR (refer to Section 4.8) and can be in the form of a separate document and/or electronic data deliverable resubmittal. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report will be kept intact and the revisions and cover letter included in the project files.

5.9.4 Subcontractor Test Results

Subcontracted data are clearly identified as such, and the name, address, and telephone number for the laboratory performing the test is included in the project report. Subcontracted results from laboratories external to STL are not reported on STL report forms or STL letterhead. Test results from more than one STL facility are clearly identified with the name of the STL facility that performed the testing, address, and telephone number for that facility. Data from subcontractors' reports may be added to an STL electronic deliverable.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- The name, address, and telephone number of the facility are provided.
- Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- The intra-company subcontractor's original report, including the chain of custody is retained by the originating laboratory.
- Proof of certification is retained by the originating laboratory.
- All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5 Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL's services. STL offers a variety of EDD formats. EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process in Section 4.4.1. Once the laboratory has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

EDDs are subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory demonstrates that it can

routinely generate that EDD without errors. Any revisions to the EDD format are reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

5.9.6 Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. Regardless of the level of reporting, all projects undergo the levels of review as described in Section 5.3.6.

Appendix. List of Cited SOPs and Work Instructions

Cited Section No(s).	Status	Description	Document No.
1.1	Buff	Certification Listing	STLBuffCertList
1.6 5.7.1	Buff	Container Management: Process Operation/Bottle Order Set-Up	APM-Bottle Order-03
1.6 4.4.2	Buff	Project Management: Project Planning Process/Project Information Requirements	APM-ProjInfo-20
4.1.1 5.4.1	Buff	Capital Equipment Listing	STLBuffEquipList
4.1.2.9	Buff	Computer System Account and Naming Policy Computer System Password Policy Software Licensing Policy Virus Protection Policy	P-I-003 P-I-004 P-I-005 P-I-006
4.3.1.1 5.3.2	Buff	SOP Master Index	STLBuff_SOPIndex
4.3.2 4.12.3	Buff	Data Management: Record Storage and Retention	AGP-RecordStorage-56
4.4.2	Buff	Project Kick-Off Meetings	APM-ProjInfo-20
4.6	Buff	Procurement of Laboratory Supplies and Services	APH-Supply-08
4.6.1	STL	Testing Solvents and Acids	S-T-001
4.7.2	Buff	Client Confidentiality	APM-ProjInfo-20
4.8	Buff	Data Quality Request	AQA-DQR-65
4.8	Buff	Preventative or Corrective Action	AQA-CA-35
4.8 4.11	Buff	Job Exception Report (Non-conformance Report)	AQA-CA-35
4.11	Buff	Quality Systems Management Review	AQA-Management Review-45
4.11	Buff	Preventive Action Measures	AQA-CA-35
4.13	STL	Systems Audits	S-Q-002
5.1.2	Buff	Laboratory Personnel Training	AQA-Train-10
5.1.3	STL	Ethics Policy	P-L-006
5.3.1	Buff	Methods Capabilities & Index	ASR-Prsv-07
5.3.2	Buff	SOP Interim Change	AQA-SOP-55
5.3.5	STL	MDL Policy	S-Q-003
5.3.6.1	STL	Acceptable Manual Integration Practices	S-Q-004

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Appendix. List of Cited SOPs and Work Instructions

Cited Section No(s).	Status	Description	Document No.
5.3.6.2	Buff	Data Review Checklists / Technical Data Review GC Extractables / HPLC GC Volatiles GC/MS: Volatiles and Semi-Volatiles Metals Wet Chemistry	AGP-DataReview-21
5.4.2	Buff	Instrument and Equipment Out-of-Service Tagging	AGP-OutofService-65
5.4.3	STL	Selection of Calibration Points	P-T-001
5.4.3	Buff	Standards Traceability and Preparation	AGP-STD-14
5.5.1	Buff	Balance Calibration, Care and Use	AGP-SuppEquip-02
5.5.1	Buff	Thermometer Calibrations	AGP-SuppEquip-02
5.5.1	Buff	Water Quality	AGP-SuppEquip-02
5.7.1	Buff	Sample Receipt: Handling and Processing	ASR-Receipt-05
5.7.1	Buff	Sample Container Preparation and Shipment	ASR-Bottle-03
5.7.5	Buff	Laboratory Waste Disposal Procedures	ASR-Disp-33 and AWM-Haz.Mg-01
5.8.5	Buff	Glassware Cleaning Procedures	AGP-Glass-04
5.9 5.9.6	Buff	Data Management: Reporting	ARP-Report-125

Attachment U

Laboratory Standard Operating Procedures for Analytic Methods

STL Buffalo

LABORATORY STANDARD OPERATING PROCEDURES

Once printed, this is considered an uncontrolled document

SOP No.	Revision No.	Effective Date	Page
AWC-CYANIDE-8	7	May 31, 2006	1 of 23

TITLE: TOTAL CYANIDE 335.4 / 9012A / 335.1

SUPERCEDES: Revision 6

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Christopher Spencer, Laboratory Director		5/31/06
Verl D. Preston, Quality Manager		5/31/06
Peggy Gray-Erdmann, Department Manager		5/31/06

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1.0 IDENTIFICATION OF TEST METHODS

1.1 Methods 335.1, 335.4 and 9010B / 9012A

2.0 APPLICABLE MATRIX

2.1 Applicable matrices are drinking, surface, saline waters, sediments and other solids including oils, domestic and industrial.

3.0 REPORTING LIMIT

3.1 Reporting Limit (aqueous) = 0.01 mg/L

3.2 Reporting Limit (solids) = 10.0 mg/kg

3.3 Method Detection Limit is determined annually and kept on file with the QA department.

4.0 SCOPE AND APPLICATION

4.1 The colorimetric procedure for aqueous samples is used for concentrations below 0.5 mg/L and quantitation is accurate to 0.01 mg/L. Levels of cyanide can be detected to 0.005 mg/L

TITLE: TOTAL CYANIDE 335.4 / 9012A / 335.1

SUPERCEDES: Revision 6

4.1.1 For samples with concentrations higher than 0.5 mg/L, a dilution must be performed.

4.2 The colorimetric procedure for soil, oil, and other solid samples is used for concentrations below 50mg/kg and provides accurate quantitation down to 10.0 mg/kg.

4.2.1 For samples with concentrations higher than 50mg/kg, a dilution must be performed.

5.0 SUMMARY OF THE TEST METHOD

5.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then analyzed colorimetrically.

5.2 In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the samples and the standards.

6.0 DEFINITIONS

6.1 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

7.0 INTERFERENCES

7.1 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation nitrate and nitrite will form nitrous acid which will react with some organic compounds to form oximes. These compounds formed will decompose under test conditions to generate HCN. The interference of nitrate and nitrite is eliminated by pretreatment of all samples and standards with sulfamic acid.

7.2 Sulfides adversely affect the colorimetric procedure. Samples that contain hydrogen sulfide, metal sulfides or other compounds that may produce hydrogen sulfide during the distillation should be eliminated.

7.2.1 Steps to remove sulfide interference should only be used when the potential interferences are confirmed to be present since the treatment can affect cyanide recoveries.

7.3 The presence of surfactants may cause samples to foam during refluxing. If this occurs, the addition of an agent such as DOW CORNING 544 antifoam agent will prevent the foam from collecting in the condenser tube.

7.4 Oxidizing agents, such as chlorine, decompose most cyanide containing samples. Therefore, each sample is tested for chlorine using potassium iodide starch test papers.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Potassium Cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Chloramine T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.

Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Potassium	Poison Corrosive	5 Mg/M3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1 Reflux-distillation apparatus: Enviro Midi-Distillation system. Includes heat block with a variable timer, built in cold finger condensers with flowmeter and Teflon valves for controlling the vacuum for the scrubbers.

9.1.1 20 glass tubes

9.1.2 10 absorber impingers with fitted ends

9.1.3 10 reflux impingers

9.2 4 culture tube racks

9.2.1 16X120 mm disposable culture tubes

9.3 5 - 125 mL Erlenmeyer flasks

9.4 Filter pump

9.5 Analytical balance

9.6 Vortex mixer

9.7 Miscellaneous laboratory glassware (volumetric flasks, graduated cylinders, etc.).

9.8 Eppendorf pipettes

9.9 Lead acetate test paper

9.10 Potassium iodide – starch test paper

9.11 Lachat Quikchem 8000: flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

9.11.1 Sampler
9.11.2 Multi-channel proportioning pump
9.11.3 Reaction unit or manifold
9.11.4 Colorimetric detector (570nm) (similar to a spectrophotometer)
9.11.5 Data system

10.0 REAGENTS AND STANDARDS

10.1 VWR 10 N Sodium Hydroxide solution

10.2 VWR 0.25 N Sodium Hydroxide solution

10.3 VWR Magnesium Chloride solution

10.4 Sulfamic Acid
10.4.1 50.0 g Sulfamic Acid to 500 ml DiH2O.

10.5 50% Sulfuric Acid
10.5.1 250mL deionized H2O + 250mL Concentrated sulfuric acid

10.6 Cadmium Carbonate (Powder)

10.7 Ascorbic acid

10.8 Methyl red indicator

10.8.1 Dissolve 0.1g methyl red sodium salt into 100 mL of Deionized water.

10.9 Stock cyanide solution 1mL = 1 mg CN

10.9.1 Purchased pre-made, from two separate sources (1 source for the Intermediate SRM, 1 source for the Intermediate STD)

10.10 Phosphate Buffer, 0.71m

10.10.1 Dissolve 97.0g potassium phosphate, monobasic, anhydrous into 1L of deionized water. Prepare biannually.

10.11 Chloramine-T solution

10.11.1 Dissolve 2.0g Chloramine-T into 250mLs of DI water. This solution must be made daily.

10.12 Pyridine-Barbituric acid reagent

10.12.1 In a fume hood place 15g of Barbituric acid into a 1L volumetric flask, and add just enough deionized water to wash the sides of the flask and wet the barbituric acid. Add 75 mLs of pyridine while stirring until barbituric acid dissolves. Add 15 mLs of concentrated HCl, mix, and cool to room temperature. Dilute to 1L with deionized water and mix. Refrigerate and store in dark bottle. Prepare weekly.

10.13 Intermediate Cyanide SRM/STD: 50 mL/L

10.13.1 2.5mL of the 1000ppm Standard is added to 50mL deionized water

10.14 Deionized (Reagent) Water

10.15 Carrier: 0.25N NaOH purchased pre-made.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 The sample should be collected in plastic or glass bottles 125mL or larger size. All bottles must be thoroughly cleansed and rinsed to remove soluble material. STL Buffalo purchases pre-cleaned bottles that come with a certificate of analysis.

11.2 Samples must be preserved with 2 mLs of 10 N NaOH per Liter of sample (pH>12) at the time of collection.

11.3 Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator/cooler to maintain temperature at 4±2° C. Distillation and analysis for cyanide must be completed within 14 days after sample collection for methods 9010/9012, 335.1, 335.2 and 335.4.

TITLE: TOTAL CYANIDE 335.4 / 9012A / 335.1

SUPERCEDES: Revision 6

12.0 QUALITY CONTROL

12.1 All QC samples must be in the established acceptable range set by Severn Trent Laboratories.

12.2 All blanks must read less than the STL Buffalo quantitation limit.

12.2.1 Quantitation limits are 0.01 mg/L for aqueous samples and 10.0 ug/g for soils and other solids.

12.2.2 All blanks associated with USACE samples should be less than 1/2 the STL Buffalo quantitation limit for Cyanide.

12.3 Duplicates and spikes are distilled and analyzed every twenty samples or less. Acceptance limits for spikes and duplicates are statistically derived and updated annually. Current limits are maintained in the laboratory LIMs system. If a duplicate or a spike falls outside the acceptance limits, the data should be reviewed to determine cause. Re-analysis may be necessary if unacceptable values are due to lab error.

12.4 If sample specific spikes and duplicates have not been requested by a client, you must choose one for the batch. If there is not enough volume to perform a duplicate and spike on any given sample in the group, an MSB and MSBD may be used. Be sure to include all information pertinent to the analysis, such as spike levels, date of analysis, analyst's initials, weights and volumes of samples in the Cyanide logbook. Also, be sure to include any out of the ordinary observations, comments or any problems that may occur.

13.0 CALIBRATION AND STANDARDIZATION

13.1 A calibration curve is made by preparing a series of standards and a blank, using suitable volumes of the Intermediate Calibration Standard

13.2.1 The Intermediate Calibration Standard: 2.5ml stock 1000ppm STD into 50 ml of deionized water.

13.2.2 The calibration curve is to be distilled if using a complex cyanide standard.

13.2.3 Calibration Curve is prepared using the following:

Curve Concentration	Intermediate STD into 100ml of 0.25m NaOH
0 mg/L	50ml of 0.25m NaOH
0.005 mg/L	0.005 ml Intermediate STD into 50ml of 0.25m NaOH
0.01 mg/L	0.01 ml Intermediate STD into 50ml of 0.25m NaOH
0.05 mg/L	0.05 ml Intermediate STD into 50ml of 0.25m NaOH
0.10 mg/L	0.1 ml Intermediate STD into 50ml of 0.25m NaOH
0.20 mg/L	0.2 ml Intermediate STD into 50ml of 0.25m NaOH
0.30 mg/L	0.3 ml Intermediate STD into 50ml of 0.25m NaOH
0.40 mg/L	0.4 ml Intermediate STD into 50ml of 0.25m NaOH
0.50 mg/L	0.5 ml Intermediate STD into 50ml of0.25m NaOH

13.2.4 A calibration curve containing these nine points is analyzed monthly.

13.2.5 Acceptance criteria for the calibration curve is a correlation coefficient (R-value) $\geq$0.995. If the R-value is less than 0.995, the calibration standards must be remade and a new curve analyzed

14.0 PROCEDURE

14.1 Sample Pretreatment

14.1.1 Oxidizing agents, such as chlorine, decompose most cyanides. Test by placing a drop of sample on a strip of potassium iodide (KI) starch paper previously moistened with acetate buffer solution, pH 4. A bluish color indicates the need for treatment. Add L-ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper, then add an additional 0.006g of ascorbic acid for each 100 mL of sample volume.

14.1.2 Oxidized products of sulfide convert CN^- to SCN^- rapidly, especially at high pH. Test for S_2^- by placing a drop of sample on lead acetate paper previously moistened with acetic acid buffer solution, at a pH of 4. Darkening of the paper indicates presence of S_2^-. Add lead acetate, or if the S_2^- concentration is too high, add powdered lead carbonate [$Pb(CO_3)2$] to avoid significantly reducing pH. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate paper. Filter sample before raising pH for stabilization. When particulate, metal cyanide complexes are suspected, filter solution before removing S_2^-. Reconstitute sample by returning filtered particulates to the sample bottle after the S_2^- removal. Homogenize particulates before analysis.

14.1.3 Aldehydes convert cyanide to cyanohydrin. Longer contact times between cyanide and the aldehyde and the higher ratios of aldehyde to cyanide both result

in increasing losses of cyanide that are not reversible during analysis. If the presence of aldehydes is suspected, stabilize with NaOH at time of collection and add 2ml 3.5% ethylene diamine solution per 100 ml of sample.

14.2 Cyanide amenable to chlorination

14.2.1 When determining cyanide amenable to chlorination part of the sample is first chlorinated to decompose the cyanides. Both the chlorinated and untreated samples are then subjected to distillation. The difference between the CN^- concentrations found in the two samples is expressed as cyanides amenable to chlorination.

14.2.2 To prepare amenable portion of sample, pour 50ml of sample into a 250ml Erlenmeyer flask completely wrapped in aluminum foil. To this add $Ca(OCl)_2$ solution dropwise while agitating and maintaining a pH of 11-12 (adding NaOH if necessary.) Test for proper chlorination by placing a drop of solution on a strip of KI-starch paper. Cover flasks with aluminum wrapped watch glass and maintain residual chlorine for 1 hour while agitating. After 1 hour remove any residual chlorine by dropwise addition of $NaAsO_2$ solution or H_2O_2, followed by 4 drops of $Na_2S_2O_3$ solution. Test with KI-starch paper after each addition, until there is no color change. Distill as outlined in section 14.7.1

14.3 Standards, blanks, and spikes:

14.3.3 Add 0.4 mL of the 50 mg/L SRM, dilute to 50 mL with deionized water for a final concentration of 0.4ppm. This is the Initial Calibration Verification (ICV) All further Continuing Calibration Verifications (CCV) are made by adding 0.25mL of the 50mg/L SRM to 50mL deionized water for a final concentration of 0.25 mg/L.

14.3.4 Add 50 mLs of deionized water to one test tube. This is the preparation blank

14.3.5 Add 0.25 mL of a purchased ERA QC Check STD, dilute to 50 mL with deionized water. This is the ERA high level QC STD.

14.3.6 If a soil sample is being distilled, a purchased soil ERA QC Check STD must also be added to the run. To do this, add approximately 0.5g of a solid ERA and dilute to 50 mL with deionized water.

14.3.6.1 Do not use Soil ERA check standards for USACE projects.

14.3.7 Add 50 mLs of each sample to the appropriate test tube.

14.2.7.1 For aqueous samples: Test tubes are certified 50ml. Pour sample from bottle into tube, stopping when meniscus reads 50ml.

14.2.7.2 For soil samples: Weigh 0.5 ± 0.1 grams of sample. Record weight in distillation log. Transfer soil sample and bring to a final volume of 50 ml with deionized water in distillation tube.

14.3.8 For sample spikes, add 0.1mL of the 50mg/L SRM and dilute to 50 mL with your sample (This is a 0.1mg/L spike for aqueous samples).

14.4 Place one reflux impinger in each sample tube.

14.4.3 Place the tube containing the sample into the heat block holes, making sure the gas outlet is facing the rear of the heating unit.

14.5 Add 50 mLs of 0.25N NaOH into a clean tube, place one absorber impinger in each of the tubes.

14.5.3 Place the tube containing 0.25N NaOH in the front of the block. Connect the absorber impinger gas inlet tube to the reflux impinger gas outlet tube. Connect the impinger vacuum tube to the valve tube.

14.6 Place one cold finger condenser into each reflux impinger, making sure that the water ports are facing the rear of the block.

14.6.3 Turn on the cooling water and adjust pressure so that it reads ~35.

14.7 Turn on the vacuum unit and adjust each valve to give a flow of ~3 bubbles per second in each reflux tube.

14.8 Total and Amenable Cyanide samples:

14.7.1. Through the air inlet of the reflux impinger add 2.0ml of Sulfamic acid (wait 3 minutes), SLOWLY, dropwise 5.0 mL of 1:1 sulfuric acid (rinse the air inlet), 2.0 mLs of magnesium chloride solution and a final rinse with DI water.

14.8. Free Acid Dissociable Cyanide samples:

14.8.1 Do not add sulfamic acid, sulfuric acid and magnesium chloride solution. Through the air inlet of the reflux impinger add 2.0 ml of acetate buffer and 2.0ml of zinc acetate solution. Add 2-3 drops of methyl red indicator. If the solution does not turn pink add glacial acetic acid dropwise until pink color persists.

14.9. Turn unit on using the red rocker switch. Allow samples to reflux for 75 minutes. This

allows for 15 minutes heat up time and 60 minutes of refluxing time.

14.10. Remove the absorber impinger from the tube and blow out excess liquid with bulb. Repeat for all samples.

14.11. System Start-up Procedure

14.11.1. Prepare reagent and standards as described in section 10.0.

14.11.2. Set up manifold as shown in section 22.

14.11.3. Input data system parameters as in section 22.

14.11.4. Place samples and/or standards in autosampler (section 22.4). Input the information required by the data system, such as concentration, replicates and QC scheme.

14.11.5. Calibrate the instrument by injecting the standards.

14.12 System Notes

14.12.1 Allow 15 minutes for heating unit to warm up to 60°C.

14.12.2 If sample concentrations are greater than 0.5 mg/l, the distilled sample should be diluted with 0.25M sodium hydroxide (NaOH) diluent. Do not dilute distilled samples or standards with Di Water.

14.12.3 For analyzing distilled samples, prepare a standard curve by plotting the peak area of distilled standards against concentration values. Compute concentrations by comparing sample peak area with standard curve.

15.0 CALCULATIONS

15.1 Sample results are calculated from the calibration curve by using linear regression.

15.2 In the case of any dilution, the results have the dilution factor automatically calculated.

15.3 For liquid samples, the result is expressed as mg/l.

15.4 For solid samples, the result is expressed as mg/kg on a dry basis.

15.4.1 To convert the mg/l result obtained from the calibration curve to mg/kg use the following equation:

mg/kg (wet) = [mg/l X final vol. of leached sample] / grams sample used

mg/kg (dry) = mg/kg (wet) / decimal dry weight

15.5 Percent Recovery for Analyses Involving Spikes:

$$\%\,\text{Recovery} = \left[\frac{(SSR - SR)}{SA}\right] \times 100$$

where:

SSR = spiked sample result
SR = sample result
SA = spike added

15.6 Relative Percent Difference (RPD):

$$RPD = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \times 100$$

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

15.7 Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

where:

a = area counts for analyte to be measured
m = slope
x = concentration
b = intercept

and

$$m = \frac{\sum x_i a_i}{\sum x_i^2}$$

$$b = Y_{ave} - bx_{ave}$$

15.8 Percent Recovery for LCS:

$$\%\,\text{Recovery (LCS)} = 100\left(\frac{E}{C}\right)$$

where:

E = obtained (experimental) value C = true value

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16.0 METHOD PERFORMANCE

16.1. Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See STL SOP S-Q-003, "Method Detection Limit Studies," current revision, for further guidance. Current STL Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

16.2. A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

16.2.1. This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

16.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

16.2.3. Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

16.2.4. Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.3. Training Qualifications

16.4. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.5. The following analyst validation information is maintained for this method in the laboratory QA files.

16.6. The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

16.7. The analyst must read and understand this SOP.

16.8. The analyst must read and understand the Method used as reference for this SOP.

16.9. The analyst must complete a DOC or successfully analyze PT samples annually.

16.10. The analyst must complete the STL Quality Assurance Training.

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17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 ICAL: calibration factor >0.990, must be analyzed at a minimum of once every three months.

17.2 ICV (second source)/ LCS: Obtained values must be within ±10% of true value.

17.3 Method Blank:

17.3.1 Detected concentrations < PQL or

17.3.2 Detected concentrations < 10X amount in associated sample

17.3.3 All blanks associated with DOD QSM and AFCEE must be less than half the reporting limit.

17.4 Matrix Spike: Acceptance limits for sample spike recovery are based on the historical data and are statistically derived annually. They are maintained in the LIMs system.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 ICAL: Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required. Please refer to STL Corporate Policy for information on the proper selection of calibration points.

18.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

18.3 LCS / CCV: Reanalyze the LCS /CCV.

18.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

18.3.2 If 2nd analysis is unacceptable, analyze a new ICAL.

18.3.3 Method Blank: Reanalyze all samples associated with an unacceptable method blank unless:

18.3.3.1 Detected concentrations < PQL or

18.3.3.2 Detected concentrations < 10X amount in associated sample

18.3.4 Matrix Spike: Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

18.3.4.1 LCS recovery is acceptable

18.3.4.2 Recoveries in both MS and MSD are consistent (%RSD<30)

18.3.4.3 If LCS is unacceptable – re-analysis is required.

18.3.4.4 If recoveries in MS/MSD are different (e.g.: one high, one low) further evaluation should be made. Matrix interference can not be assumed in this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final

decision process prior to releasing data.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

19.1.1. Holding times exceeded

19.1.2. Insufficient sample volume for reanalysis

19.1.3. In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

20.0 WASTE MANAGEMENT/POLLUTION PREVENTION

20.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

20.2 Waste Streams Produced by the Method from the instrument that contains pyridine has a pH >7 and is disposed of in the "D" waste container along with all other wastes having a pH > 7.0.

20.3 Reagents produced using pyridine are acidic and disposed of in the "A" waste container along with all other wastes having a pH < 7.0.

21.0 REFERENCES

21.1 USEPA Methods for the Determination of Inorganic Substances in Environmental Samples, Method 335.4, EPA/600/R-93/100, Revised August 1993

21.2 Method 9010B, "Total and Amenable Cyanide: Distillation," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Update III, December 1996.

21.3 Method 9012A, "Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)," Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Third Edition, Update III, December 1996.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Cyanide Manifold Diagram

22.2 Data System Parameters for QC8000

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22.3 Analytical Sequence
22.4 Analytical Batch
22.5 Distillation Batch
22.6 Wet Chemistry Batch Summary

23.0 CHANGES FROM PREVIOUS REVISION

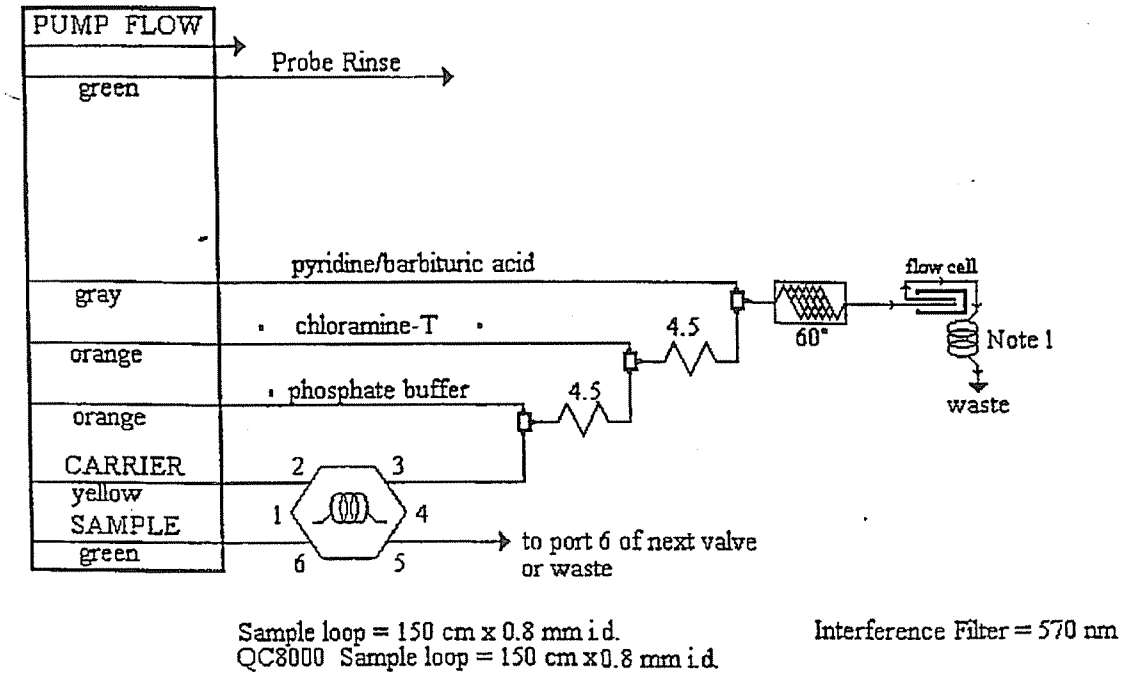
23.1 Updated section 15.0, 16.0, 17.0, 18.0 and 19.0.
23.2 Updated Section 13.2.3 to reflect curve is made from NaOH not deionized water.

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22.1 Cyanide Manifold Diagram



Carrier is 0.25 M sodium hydroxide solution. All manifold tubing is 0.8mm (0.030 in) i.d. This is 5.2 uL/cm. 4.5 is 70 cm tubing on a 4.5 cm coil support.

APPARATUS: An injection valve, a 10mm path length flow cell, and a colorimetric detector module are required. The  shows 650 cm tubing wrapped around the heater block at the specified temperature.

Note 1: 2 meter back pressure loop, 0.52 mm i.d.

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22.2 Data System Parameters for QC 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample Throughput: 80 samples/h, 45 s/sample
Pump speed: 35
Cycle period: 45

Analyte Data:

Concentration Units: mg CN-/L
Peak Base Width: 39s
% Width Tolerance: 100
Threshold: 25000
Inject to Peak Start: 24 s
Chemistry: Direct

Calibration Data:

Levels	1	2	3	4	5	6	7	8	9
Concentration mg/L	0.500	0.400	0.300	0.200	0.100	0.050	0.010	0.005	0.000

Calibration fit Type: 1^{ST} Order Polynomial
Calibration Rep. Handling: Replace
Weighting method: No
Concentration Scaling: None
Force through Zero: No

Sampler Timing:
Min. Probe in wash Period: 14 s
Probe in Sample Period: 20 s

Valve Timing:
Load Time: 0.0 s
Load Period: 20 S
Inject Period: 25 s

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22.3 Analytical Run Sequence

Cyanide Non CLP sequence
LCS/CCV
ICB/CCB
ERA QC Check STD (soil/water)
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
CCV
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate
Sample spike
CCV
CCB

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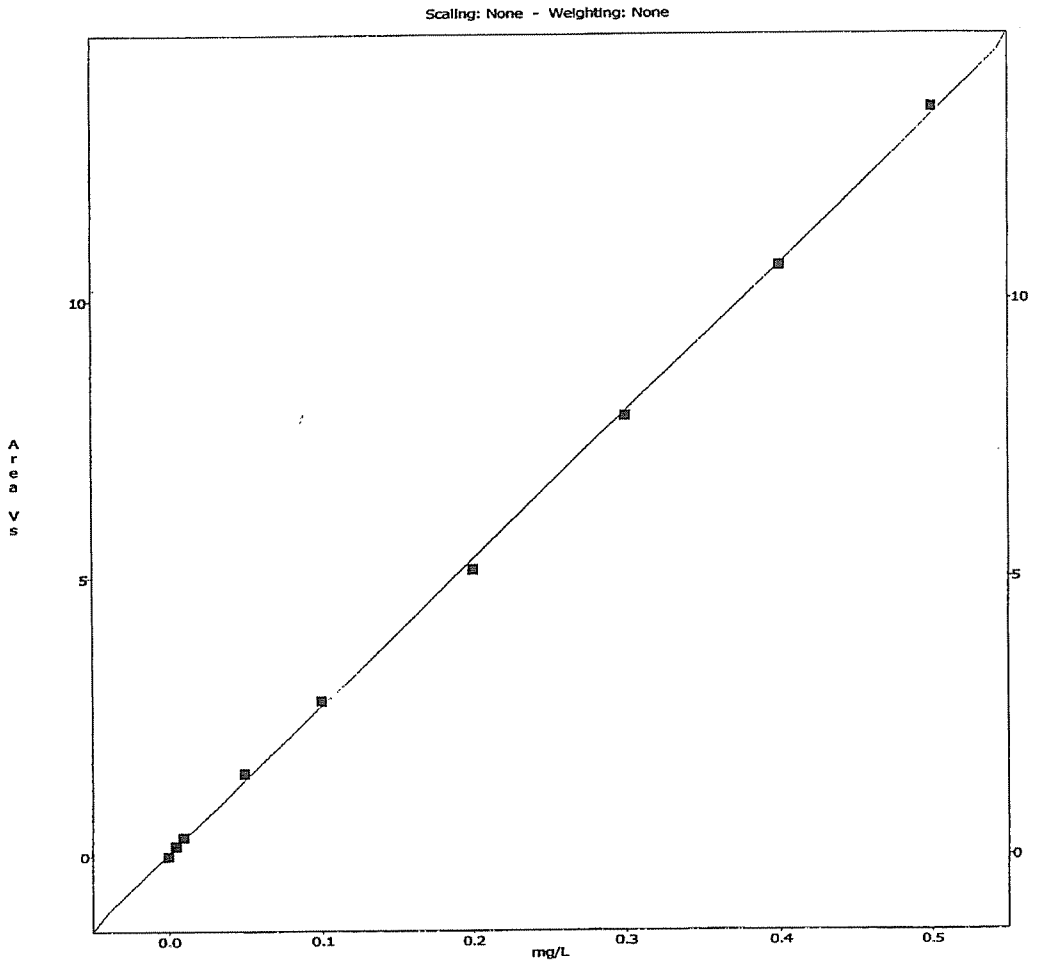
22.4 Analytical Batch Run

CYANIDE AB009975

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	13421440	0.500	13421440					0.0	0.0	-1.0
2	10603059	0.400	10603059					0.0	0.0	0.4
3	7901005	0.300	7901005					0.0	0.0	1.2
4	5143232	0.200	5143232					0.0	0.0	3.8
5	2782003	0.100	2782003					0.0	0.0	-3.3
6	1487514	0.050	1487514					0.0	0.0	-8.9
7	341542	0.010	341542					0.0	0.0	-12.1
8	185050	0.005	185050					0.0	0.0	-6.1
9	0	0.000	0					0.0	0.0	

curve 9/3

1st Order Poly
Conc = 3.774e-008 Area - 1.681e-003
r = 0.9998



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STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

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STL-BUFFALO - Chloride
OPERATOR: Lisa
ACQ. TIME: Sep 4, 2003 17:34:11
DATA FILENAME: C:\OMNION\DATA\090403D.FDT
METHOD FILENAME: C:\OMNION\METHODS\CL.MET

Start 1734
End 1752
Method 335.4

Multi-Channel Table
Type: Calibration Standards
Channel Range: 1 to 8 -- Cup Range: 1 to 50

Cup	Sample ID	# of Reps	(uv-s)

NO SAMPLE INFO FOR THE SELECTED CUP RANGE

Method - Ch. 1 0 A3809975

METHOD DESCRIPTION:
Created: Mar 29, 2000 21:55:50
Modified: Sep 4, 2003 16:44:54

ANALYTE DATA:
Analyte Name:
Concentration Units: mg/L
Chemistry: Direct
Inject to Peak Start (s): 8.0

JS 9/4/03

Multi-Channel Table
Type: Unknowns
Channel Range: 1 to 8 -- Cup Range: 1 to 27

Cup	Sample ID	Sampling Time	# of Reps	(mg/L) cyanide	Auto Dil Factor	
	.250					
1	~~lcs@.400ppm~~	17:34:15	1	0.2596	1.00	104%
2	icb	17:37:48	1	-0.0049	1.00	<.005
3	era P090502	17:38:42	1	0.3444	1.00	88% @ .392ppm
4	a3834904	17:39:35	1	-0.0049	1.00	
5	a3834904dup	17:40:28	1	-0.0049	1.00	
6	a3835101	17:41:21	1	0.0096	1.00	
7	a3835101spk	17:42:14	1	0.0844	1.00	75%
8	a3835401	17:43:07	1	-0.0049	1.00	
9	a3835402	17:43:59	1	-0.0049	1.00	
10	a3835403	17:44:52	1	-0.0049	1.00	
11	a3835404	17:45:44	1	-0.0049	1.00	
12	a3840401	17:46:36	1	-0.0049	1.00	
13	ccv@.25ppm	17:47:28	1	0.2575	1.00	103%
14	ccv	17:48:20	1	-0.0049	1.00	<.005
15	a3837001	17:49:12	1	-0.0049	1.00	
16	a3837002	17:50:05	1	-0.0049	1.00	
17	a3838301	17:51:00	1	-0.0049	1.00	
25	ccv	17:51:52	1	0.2613	1.00	105%
26	ccb	17:52:44	1	-0.0049	1.00	<.005

Solutions
4-126-E
A-33-G
4-114-D
4-117-B

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TITLE: TOTAL CYANIDE 335.4 / 9012A / 335.1

SUPERCEDES: Revision 6

22.5 Distillation Batch Log

STL BUFFALO

Cyanide Log- Distillation and Colorimetric

Prep Date	Read Date	Analyst	Job #	Sample I.D.	Dist. Flask #	Spec Flask #	Sample Vol/Wt (mg/l)	Dilution	ABS	Curve Conc. (ug)	Final Conc. mg/l or ug/g	True Value Spike Amount (ppm)	Comments
9/4	9/4	MSB	ICV	0.400 250ppm	1								
			ICB	Blank	2								
			ERA	P091602	3								
			8349	4	4								
			↓	4 dup	5								
			8351	1	6								
			↓	1 Spike	7								
			8354	1	8								
				2	9								
				3	10								
				4	11								
			↓	5	12								
			8389	1	13								
			8402	1	14								335.2
			8404	1	15								335.2
			8370	1	16								
			↓	2	17								
			8363	1	18								

Start Time 0900 Reviewed By [illegible] Solutions _____

End Time _____ Date 9/4 Spec N/A

H2S ✓ Cl2 ✓

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SUPERCEDES: Revision 6

22.6 Wet Chemistry Batch Summary

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ METHOD_____ BATCH _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____

WC Summary Rev 2 / 2-2005

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES
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TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl Preston, Quality Manager	Verl D Preston	8/30/05
Christopher A. Spencer, Laboratory Director		8/30/05
P. McNamara, Supervisor	For P. McNamara	08/30/05

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1.0 IDENTIFICATION OF TEST METHODS

1.1 Method 8270C, SW846, 3RD Edition, update III.

2.0 APPLICABLE MATRIX

2.1 Waters, soils, sludges and TCLP.

3.0 REPORTING LIMIT

3.1 See Table 1 for a listing of laboratory quantitation limits.

4.0 SCOPE AND APPLICATION:

4.1 The analytical method is utilized for the analysis of water, air sampling media, sediment and soil from hazardous waste sites for the organic compounds listed in Table 1. Table 1 includes CAS numbers and estimated quantitation limits for each analyte. Typical sample size should be 30 grams for soils and 1 liter for waters. The method begins with the extraction of the sample aliquot either by sonication (soils) or separatory funnel extraction (waters), into 1:1 methylene chloride/ acetone mixture. The extraction volume is then concentrated to 1.0ml final volume for waters and soils. The extracts are prepared for analysis with the addition of internal standard to each vial. One microliter of each extract is then directly injected into a gas chromatograph and the compounds are separated by mass using a capillary column and analyzed using a mass spectrometer. A summary of the analysis procedure is provided in Attachment A.

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5.0 SUMMARY OF TEST METHOD

5.1 See Scope and Application.

5.0 DEFINITIONS

6.1 All definitions are in parenthesis. Additional definitions scan be found in the STL Buffalo Laboratory Quality Manual (LQM).

7.0 INTERFERENCES

7.1 Some of the possible interferences that arise during GCMS Semivolatile analysis include, but are not limited to:

1. Glassware contamination
2. Matrix interference
3. Aldol condensation
4. System air leaks
5. Injection port/liner contamination
6. Warped filament, and/or dirty source and rods
7. APIX analytes Methapyrilene and Phentermine split at all concentrations and require manual integration in calibration standard.

7.2 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

7.3 See section 1.4 and 3.0 of method 8270c for other interferences, with the exception that there is no carryover in direct injection GCMS.

8.0 SAFETY:

All STL employees are required to comply with STL' s Corporate Safety Manual, which has been prepared in accordance with OSHA requirements. This includes wearing the proper laboratory clothing such as lab coat, gloves and safety glasses.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Chemicals that have been classified as carcinogens or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

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Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

8.1 Analysts are expected to use caution and common sense while working in a laboratory environment. Each employee is required to read the companies' Corporate Safety Manual. All of the samples to be analyzed have the potential to contain hazardous substances. Most standards also contain hazardous chemicals and many do contain known carcinogens. Employees must use protective equipment when handling standards, samples and extracts including gloves, lab coats and safety glasses. It is the analyst's responsibility to read and familiarize themselves with the MSDS of each chemical and/or reagent involved in this method.

8.2 Samples, standards and/or extracts should never be opened or transferred outside of a fume hood.

8.3 Waste disposal is all C waste with the exception of some acids used in the cleaning of equipment which is disposed of in AN waste.

8.4 Spills should be cleaned up promptly and waste should be disposed of as per the Chemical Hygiene Plan.

8.5 There is also the danger of burns while doing repair or maintenance on a gas chromatograph. One must use caution while working on or near the injection port or transfer line.

8.6 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES:

9.1 Calibrated micro syringes 10, 25, 50, 100, 500, 1,000 microliter.

9.2 2ml amber vials and caps.

9.3 Disposable pipets and pipet bulbs.

9.4 Volumetric flasks.

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9.5 Gas Chromatograph/Mass Spectrometer (GC/MS) System

9.5.1 Gas Chromatograph -
- Hewlett Packard 6890
- Carrier gas Helium UPC grade or equivalent

9.5.2 Gas Chromatography Column
- Analysis: ZB-5 or ZB-5MS (Crossbond 5% diphenyl-95% dimethyl polysiloxane) 30 meter 0.25mm ID.25 or 0.50 µmdf, or an equivalent alternative.

9.5.3 Deactivated Guard Column installed between injection port and chromatograghic column with deactivated union.

9.5.4. Mass Spectrometer
- HP5973 and HP5973 inert
- Tuning compound PFTBA
- Scan Range 35-500 AMU/second

9.5.5 Data System
- HP Chemstation
- Teknivent and HP enviroquant software

10.0 REAGENTS AND STANDARDS:

10.1 Methylene Chloride – high purity

10.2 Standards:

10.2.1 Stock Standards
Calibration Mix #1 1000µg/ml
Calibration Mix #2 2000µg/ml
Benzidines Mix 2000 µg/ml
N-Nitrosodiphenylamines 5000 µg/ml
OLM Mix 2000 µg/ml
Benzoic Acid 2000 µg/ml
BN/AP Surrogate Mix 4000 µg/ml
DFTPP mix 50µg/ml or equivalent
Internal Standard Mix 2.0 mg/ml

All Certificates of Analysis received from the manufacturer are maintained in a laboratory notebook. Stock standards are prepared every twelve months or sooner, if necessary.

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10.2.2 Initial and Continuing Calibration Solutions
8270 Stock Solution

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc.	Final Conc. In Samples
Calibration Mix # 1	$MECL_2$	1000 ng/ul	400µl	2000ul	200 ng/ul	200 ug/L
Calibration Mix # 4	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
Benzidines Mix	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
N-Nitrosodiphenylamine Mix	$MECL_2$	5000 ng/ul	80µl	2000ul	200 ng/ul	200 ug/L
BN/AP Mix	$MECL_2$	4000 ng/ul	100µl	2000ul	200 ng/ul	200 ug/L
OLM Mix	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
Benzoic Acid	$MECL_2$	2000 ng/ul	400µl	2000ul	200 ng/ul	400 ug/L

10.2.3 Working Standards

10.2.3.1. Surrogate Standard Spiking Solution (A00001-AIMS® Code)
Surrogate Standard Spiking solution is prepared that contains nitrobenzene-d5, terphenyl1-d14, 2-fluorobiphenyl, and 1,2-dichlorobenzene-d4 at a concentration of 100µg/ml; phenol-d5, 2,4,6-tribromophenol, 2-fluorophenol and 2-chlorophenol-d4 at a concentration of 150µg/ml. Surrogate standards are added to all samples and calibration solutions. Additional surrogates may be added at the laboratory's discretion.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. In Samples
Semivolatile Acid Surrogate Phenol-d5 2,4,6-Tribromophenol 2-Fluorophenol 2-Chlorophenol-d4	MEOH	10,000ng/ul	1,500ul	100,000ul	150ug/L
Semivolatile B/N Surrogate Nitrobenzene-d5 Terphenyl-d14 2-Fluorobiphenyl 1,2-Dichlorobenzene-d4	MEOH	5000ng/ul	2,000ul	100,000ul	100ug/L

10.2.3.2. Matrix Spiking Solution (11 compound) (A00055-AIMS® Code)
The 11 compound matrix spiking solution consists of the following:

Bases/Neutrals	Acids
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	

a. Using the Intermediate Acid and BN Standards, the Matrix Spike solution is prepared that contains each of the base-neutral compounds above at 100µg/ml in methanol and the acid compounds at 100µg/ml in methanol.

Standard	Solvent	Stock Conc.	Initial Wt/Vol	Final Vol.	Final Conc. in Solution	Final Conc. In Aqueous Samples
Acid Matrix Spike Intermediate	MeOH	10000ng/ul	5000uls	500mls	100 ug/ml	100 µg/L
BN Matrix Spike Intermediate	MeOH	5000ng/ul	10000uls	500mls	100 ug/ml	100 ug/L

10.2.3.3 Matrix Spiking Solution (all compound) (A00193- AIMS® Code)

The all compound matrix spiking solution contains each of the following SVOA target analytes at 100µg/ml in methanol. Additional compounds may be included in the spike mixture if required for a specific project.

Ancenaphthene	Dibenzo(a,h)anthracene	Indeno(1,2,3-cd)pyrene
Acenaphthylene	Dibenzofuran	Isophorone
Anthracene	di-n-butyl phthalate	2-Methylnaphthalene
Benzo(a)anthracene	1,2-Dichlorobenzene	2-Methylphenol
Benzo(b)fluoranthene	1,3-Dichlorobenzene	4-Methylphenol
Benzo(k)fluoranthene	1,4-Dichlorobenzene	Naphthalene
Benzo(ghi)perylene	3,3'Dichlorobenzidine	2-Nitroaniline
Benzo(a)pyrene	2,4-Dichlorophenol	3- Nitroaniline
Benzoic acid	Diethyl phthalate	4- Nitroaniline
Benzyl alcohol	2,4-Dimethylphenol	Nitrobenzene

Bis(2-chloroethoxy)methane	Dimethyl phthalate	2-Nitrophenol
Bis(2-chloroethyl)ether	4,6-Dinitro-2-methylphenol	4-Nitrophenol
2,2'-oxybix(1-Chloropropane)	2,4-Dinitrophenol	N-nitrosodiphenylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-Di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	Pentachlorophenol
Butyl benzyl phthalate	Di-n-octyl phthalate	Phenanthrene
2-Chloroaniline	Fluoranthene	Phenol
4-Chloro-3-methylphenol	Fluorene	Pyrene
2-Chloronaphthalene	Hexachlorobenzene	1,2,4-Trichlorobenzene
2-Chlorophenol	Hexachlorobutadiene	2,4,5-Trichlorophenol
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	2,4,6-Trichlorophenol
Chrysene	Hexachloroethane	

10.2.3.4. Instrument Performance Check Solution (DFTPP)

A solution of Decafluorotriphenylphosphine (DFTPP) at a concentration of 50 ng/µL in methylene chloride is prepared such that a 1µL injection will result in a final concentration of 50ng on column. The instrument performance check solution also contains Benzidine, Pentachlorophenol and 4,4-DDT for use in evaluating chromatographic performance.

DFTPP Check Solution

Standard	Solvent	Final Conc.
DFTPP	$MECL_2$	50 µg/ml
Benzidine	$MECL_2$	50 µg/ml
Pentachlorophenol	$MECl_2$	50 µg/ml
4,4'-DDT	$MECL_2$	50 µg/ml

10.2.3.5. Initial and Continuing Calibration Solutions

Calibration standards are prepared at a minimum of five concentration levels (10, 50, 80, 120, and 160 total ng per 1µL). Each calibration standard should contain each compound of interest and each surrogate.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc,	Final Conc. In Samples
50ppm SSTD 050 Internal Standard Custom Mix	$MECL_2$	200 ng/ul 2000ng/ul 500ng/ul	250µl 20µl 60ul	1000ul	50 ng/ul 40ng/ul 80ng/ul	50ug/l
10ppm SSTD 010 Internal Standard Custom Mix	$MECL_2$	200 ng/ul 2000 ng/ul 500 ng/ul	50µl 20µl 80ul	1000ul	10ng/ul 40ng/ul 50ng/ul	10ug/l
80ppm SSTD 080 Internal Standard Custom Mix	$MECL_2$	200 ng/ul 2000 ng/ul 500 ng/ul	400µl 20µl 40ul	1000ul	80 ng/ul 40ng/ul 100ng/ul	80ug/l
120ppm SSTD 120 Internal Standard	$MECL_2$	200 ng/ul 2000 ng/ul	300µl 10µl	500ul	120 ng/ul 40ng/ul	120ug/l
160ppm SSTD 160 Internal Standard	$MECL_2$	200 ng/ul 2000 ng/ul	400µl 10µl	500ul	160 ng/ul 40ng/ul	160ug/l

10.2.3.6. Internal Standard Solution

Internal Standard solution is prepared at a concentration to maintain a 40ng level per 1µl. The Internal Standard solution contains the following compounds at a concentration of 40ng/µl, Acenaphthene-d10, Chrysene-d12, 1,4-Dichlorobenzene-d4, Napthalene-d8, Perylene-d12 and Phenanthrene-d10.

10.2.3.7. Storage of Standard Solutions

Stock, secondary dilution, and working standards are stored at 4°C or less in teflon-lined crimp-cap amber bottles or vials. These standards are prepared every twelve months or sooner, if necessary.

The continuing calibration standard (50ng) is stored at 4°C or less in teflon-lined crimp-cap amber vials. This standard may be prepared weekly, but may be continued to be used if no degradation and/or evaporation has occurred.

Samples, sample extracts and standards are stored in separate refrigerators.

10.2.3.8 Expiration of Standard Solutions.

Before any analyst uses a standard solution he or she must consult the "Standards Chemical History Log" to determine when or if the standard has expired.

11.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

11.1 Water samples may be collected in 1L (or more) amber glass containers with teflon-lined, screw-caps.

11.2 Soil/Sediment Samples may be collected in glass containers fitted with teflon-lined screw-caps or closed end tubes.

11.3 All samples are stored at 4 C (+/-2C) from the time of collection until extraction

11.4 Aqueous samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.

11.5 Soil samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

12.0 QUALITY CONTROL

12.1 Method Blanks - A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate/clean sand for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.1 A method blank must be prepared once for the following, whichever is more frequent:

- Each prep batch
- Each 20 samples in a batch, including matrix spikes/matrix spike duplicates, that are of a similar matrix (water, soil/sediment or similar concentration (soil/sediment only), or
- Whenever samples are extracted by the same procedure (separatory funnel extraction or sonication).

12.1.2 For semivolatile analysis, a method blank for water samples consists of 1 L volume of

reagent water spiked with 1.0mL of the surrogate spiking solution. For medium or low level soil/sediment samples, a method blank consists of 1g or 30g of sodium sulfate/clean sand spiked with 1.0mL of the surrogate spiking solution, respectively. Extract, concentrate, cleanup and analyze the blank according to procedures for water and soil samples.

12.1.3 Acceptance Criteria – levels of target analytes in the method blank must be less than the required reporting limit or less than one-tenth the concentration of the respective analyte in the associated samples. For USACE all target analytes must be less than one half of the MRL (Method Reporting Limit) and common laboratory contaminants must be less than the MRL. The MRL is set at either the MDL or the MDL Check.

12.1.4 Corrective Actions for Method Blank Analyses - If the acceptance criteria for method blank analysis is not met, the analytical system may be assumed to be out of control. The following corrective actions may be taken:

- If contamination is the problem, then the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvent, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in the GC/MS be eliminated. Samples associated with the contaminated blank must be re-extracted and re-analyzed.

- If surrogate recoveries in the method blank do not meet the acceptance criteria, first reanalyze the method blank. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, re-extract and re-analyze the blank and all associated samples OR the samples may be reported as estimated, and noted in the case narrative.

- If the method blank does not meet internal standard response requirements, check calculations, the internal standard spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard responses meet their acceptance criteria. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples. If the instrument malfunctioned, correct the instrument problem and reanalyze the method blank. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the blank

12.2 Matrix Spike Blank/Matrix Spike/Matrix Spike Duplicate(MSB/MS/MSD)

12.2.1 A matrix spike blank, matrix spike and matrix spike duplicate are analyzed to evaluate the analytical system and the effects of sample matrix on the methods used for semivolatile analysis.

12.2.2 The matrix spike blank, matrix spike, and matrix spike duplicate are spiked with the compounds of interest (at concentrations noted in the standard preparation section).

12.2.3 A matrix spike blank, matrix spike and matrix spike duplicate are extracted and analyzed for every batch of 20 samples of a similar matrix. Matrix spike and matrix spike duplicates are not performed for field QC samples such as rinsates, or field/trip blanks

12.2.4 If insufficient sample amount is received to perform matrix spike and matrix spike duplicate analysis, duplicate matrix spike blanks may be processed.

12.2.5 Dilutions
Dilutions of MS/MSD samples are performed only if the unspiked sample requires a dilution in order to maintain any target compound concentrations in the upper half of the calibration. MS/MSD samples will not be diluted to get spiked or non-spiked compounds below the highest calibration standard.

12.2.6 Calculations for MS/MSD
The concentrations of spiked compounds are determined using equations described for sample analysis. After determining the compound concentrations, the percent recovery is calculated using Equation 1.

Equation 1

$$\text{Matrix Spike Recovery} = \frac{\text{SSR - SR}}{\text{SA}} x100$$

Where,
SSR= Spike Sample Result
SR = Sample Result
SA = Spike Added

The relative percent difference between the matrix spike and matrix spike duplicate is calculated using Equation 2.

Equation 2

$$\text{RPD} = \frac{[\text{MSR - MSDR}]}{1/2\,(\text{MSR + MSDR})} \times 100$$

Where,
RPD = Relative Percent Difference
MSR = Matrix Spike Recovery
MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value

12.2.7. Technical Acceptance Criteria for MS/MSD
The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for matrix spike and matrix spike duplicate analysis also.

The matrix spike recovery limits are based on historical data and are updated annually.

The matrix spike recovery limits are advisory. If the recovery limits are not met, no further corrective action will be necessary. However, frequent occurrences of this nature should be investigated.

Re-extraction and re-analysis of the matrix spike and matrix spike duplicate may be necessary if, in the technical judgement of the analyst and/or supervisors, an error was made during the extraction procedure

12.3 Technical Acceptance Criteria for MSB:
The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for the matrix spike blank analysis also.

The matrix spike blank recovery limits are based on historical data and are updated annually.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analysis must be included with the report.

12.4 Surrogate Recoveries
The surrogate compound concentrations are determined using calculations found in Section 9. The recoveries are then determined using Equation 3

Equation 3

$$\%\ Recovery = \frac{Concentration\ (\vee\ amount)\ found}{Concentration\ (\vee\ amount)\ spiked}$$

Recovery limits for surrogate compounds are based on historical data and are updated annually.

12.5 QC Acceptance Criteria for AFCEE or USACE projects are provided in Attachments B and C

respectively.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Instrument Operating Conditions

- Gas Chromatograph; The following are recommended GC conditions that may vary slightly depending on the compound list and the column film thickness.

Initial Temperature: 40-50°C
Initial Hold Time: 3 minutes (hold time may vary to ensure proper chromatographic separation).
Temperature program 40-50°C to 70°C at 20°C/min to 195 at
16°C/min to 325 at 30°C/min
Final Temperature: 325°C
Final Hold Time: As necessary for TCL compound identification
Injector Temperature: 250°C
Source Temperature: 230°C
Transfer Line Temperature: 310°C
Injector: splitless
Front Inlet Pressure: 7.00 psi
Purge Flow: 15.0 mL/min
Purge Time: 0.50 min
Total flow: 19.2 mL/min
Injection Volume: 1µl
Carrier Gas: Helium
Carrier Flow: 36 cm/sec

- Mass Spectrometer

Electron Energy: 70 volts (nominal)
Mass Range: 35 to 500 amu
Scan Time: Not to exceed 1 second per scan

13.2 Instrument Performance Check

The GC/MS system is tuned using Perfluorotributylamine (PFTBA) such that an injection of 50ng of DFTPP will meet the abundance criteria listed in Table 2.

Prior to the analysis of standards or samples, the mass calibration and resolution of the GC/MS system is verified by the analysis of DFTPP. This analysis will verify the proper tuning of the system for 12 hours. After 12 hours, the instrument performance must be verified before standard and sample analysis may continue.

The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions.

Breakdown of 4,4'-DDT into 4,4'-DDD and 4,4'-DDE may be used to assess GC column performance and injection port inertness and must be less than 20%.

The compounds Benzidine and Pentachlorophenol should be present and at their normal responses for this concentration. Peak tailing should not be visible (PCP tailing factor <5 and Benzidine <3). If responses are poor and excessive peak tailing is present, corrective actions for the GC/MS instrument performance check solution may be required.

All subsequent standards and samples must be acquired under the same GC/MS tuning conditions that were used for the analysis of the instrument performance check solution.

13.2.1 Technical Acceptance Criteria for the GC/MS Instrument Performance Check (DFTPP) is listed in Table 2.

13.2.2 Corrective Actions for the GC/MS Instrument Performance Check
If any of the acceptance criteria are not met, the DFTPP should be re-injected to insure that the injection made was not a cause for failure. If, after reinjection, acceptance criteria has not been met, one or more of the following corrective actions may be taken:

1. Retune the GC/MS
2. Clean the source; replace parts, etc...
3. Cut the column at the injector end
4. Replace the column
5. Replace the septum in the injector
6. Replace the injector liner
7. Clean injection port with $MeCl_2$
8. Change injection port seal
9. An instrument service call may be placed.

13.3 Initial Calibration
After the instrument performance check criteria has been met and prior to the analysis of samples, the GC/MS system is calibrated at a minimum of five concentration levels in order to establish instrument sensitivity and linearity.

The initial calibration should be performed when major instrument maintenance has been performed or if continuing calibration criteria cannot be met.

Major instrument maintenance may consist of source cleaning, column changing, or quadrapole rod adjustment. Preventative maintenance such as septum changes, injector liner changes or column cutting may not require an initial calibration to be performed.

13.3.1 Procedure

Five calibration standards are prepared which contain all target and surrogate compounds. A 20µl aliquot of internal standard solution is added to a 1mL aliquot of each calibration standard solution. The resulting concentration of internal standards is 40ng. A 1µl injection would result in a final concentration of 40ng on column. The internal standards used are given in Table 3.

The relative response factors (RRF) for each target and surrogate compound is determined using equation 4. The characteristic ions for a given compound are listed in Tables 3 and 6. Internal standard assignments are listed in Table 4.

Equation 4

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,
A_x = Area of the characteristic ion for the compound to be measured (see Table 4)
A_{is} = Area of the characteristic ion for specific internal standard (see Table 3)
C_{is} = Amount of the internal standard injected (ng)
C_x = Amount of the compound to be measured injected (ng)

The mean relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of the RRF values for the initial calibration using the following equation:

Equation 5

$$\%RDS = \frac{Standard\ Deviation}{Mean} x\ 100$$

Where,

$$Standard\ Deviation = \sqrt{\frac{\sum_{i+1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

x_i = each individual value used to calculate the mean

x = the mean of n values
n = the total number of values

13.3.2 Acceptance Criteria for Initial Calibration

The average response factor (RRF) for each System Performance Check Compound (listed in Table 5) must be greater than or equal to the compound's minimum acceptable relative response factor of 0.050.

The %RSD over the initial calibration range for relative response factor for each Calibration Check (Table 5) compound %RSD must be less than or equal to the 30%.

The %RSD over the initial calibration range for the relative response factor for all other compounds must be less than or equal to 15%.

OR

The mean %RSD for all compounds must be less than or equal to 15% .

OR

A least squares regression correlation coefficient of greater than 0.990 for all compounds greater than 15% RSD.

OR

A non-linear coefficient of determination of greater than 0.990 for all compounds greater than 15% RSD. For a 2nd order non-linear regression, 6 calibration points must be used and for a 3rd order non-linear regression, 7 calibration points must be used.

13.3.3. Corrective Actions for Initial Calibration

If any of the acceptance criteria for initial calibration are not met, it may be necessary to reanalyze one or more of the calibration standards. If after reanalysis, the acceptance criteria have not been met, it may be necessary to take further corrective actions.

The following corrective actions may be taken if the acceptance criteria for initial calibration cannot be met.

1. Prepare fresh standards and reanalyze the initial calibration.
2. Replace the septum on the injector
3. Replace the injector liner

4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Clean the source
7. An instrument service call may be placed

The acceptance criteria must be met before sample analysis may proceed.

13.4 Initial Calibration Verification

To verify the accuracy of the initial calibration, a standard is obtained from a source different from the calibration standards.

Immediately following analysis of an acceptable initial calibration curve, a 80ng/µl aliquot of this independent standard is injected.

Recoveries of all compounds shall fall within ±25% of the expected value, however, recoveries of up to 40% are allowable for up to four compounds.

13.5 Continuing Calibration

If there is no time left in the 12-hour time period after initial calibration, the instrument performance check may be analyzed and a 50ng/1µl standard may be analyzed to verify the calibration of the instrument.

The continuing calibration check must be analyzed once every 12-hour time period of operation. This check must be analyzed prior to the analysis of samples for a given 12-hour time period.

13.5.1 Procedure for Continuing Calibration

The 50ng/µl standard is used for the continuing calibration. The relative response factor is calculated using procedures described for initial calibration.

If quantitation is performed using response factor, calculate the percent difference between the mean relative response factor from the most recent initial calibration and the continuing calibration relative response factor for each semivolatile target and surrogate compound using Equation 6.

Equation 6

$$\%\ Difference_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} x\ 100$$

Where,

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria
RRF_c = Relative response factor from continuing calibration standard

If quantitation is performed using a least squares regression or a non-linear model, calculate the concentration of all analytes and surrogates in the continuing calibration as described in section 8.3.2 of this SOP. Calculate the percent drift using Equation 7.

Equation 7:

$$\%Drift = \frac{Conc_E - Conc_A}{Conc_E} x100$$

Where:
$Conc_E$ = Expected Concentration
$Conc_A$ = Actual Concentration

13.5.2 Acceptance Criteria for Continuing Calibration

The relative response factor (RRF) for each System Performance Check Compound must be greater than or equal 0.050.

The RRF of percent drift for Calibration Check Compounds must be less than 20%. The RRF percent difference or percent drift for all other compounds must be within ±25%, with up to four compounds within ±40%D. For expanded list and additional compounds not on the EPA TCL list a percent drift of 40% is allowed. Any analyte may have an elevated response >40%D if it is not detected in the associated samples.

Internal Standard retention times and responses are evaluated after acquisition of the continuing calibration check. If the retention time of any internal standard shifts by more than 30 seconds or the response of any internal standard is outside of the-50%to +100% range, the system shall be inspected and corrected as needed. The CCV will be reanalyzed after inspection. If the problem is not resolved, a new initial calibration must be performed.

13.5.3 Corrective Actions for Continuing Calibration

If any of the technical acceptance criteria for continuing calibration are not met, it may be necessary to reanalyze the continuing calibration standard. If after reanalysis the acceptance criteria cannot be met, further corrective actions may be required.

The following corrective actions may be taken if the acceptance criteria for continuing

calibration cannot be met.

1. Replace the septum on the injector
2. Replace the injector liner
3. Replace injection port seal
4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Prepare fresh standards
7. Reanalyze the initial calibration

13.6 Calibration acceptance criteria for AFCEE and USACE projects are provided in Attachments B and C respectively.

14.0 PROCEDURE:

14.1 Sample extracts shall be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, continuing calibration and second source calibration verification requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

Internal standard solution is added to each sample extract. 20µL of internal standard solution is added to each accurately measured 1.0mL of water sample extract. For soil/sediment samples and water samples subjected to GPC, 10µL of internal standard solution is added to each accurately measured 0.5mL of sample extract. This will result in a concentration of 40ng/µL of each internal standard.

Necessary dilutions are made prior to adding internal standard solution. The internal standard solution must be added so that the concentration of each internal standard is 40ng/µL.

14.2. Dilutions

Dilutions of sample extracts are required if any target compound exceeds the initial calibration range.
The dilution chosen should keep the response of the largest target compound within the calibration range.

14.3. Qualitative Identification

14.3.1 Target Compounds
Target compound identification is done by comparing the sample mass spectrum to that of the standard. The following criteria must be satisfied in order to verify identifications. Elution of the sample analyte within GC relative retention time unit window established

from the 12-hour calibration standard.

Correspondence of the sample analyte and calibration standard component mass spectra.

To establish correspondence of the GC relative retention time (RRT), the sample component RRT must compare with ±0.06 RRT units of that of the standard RRT. If samples are analyzed within the same 12-hour period as the initial calibration, the 50ng standard is used to verify relative retention times.

To establish correspondence of the sample component mass spectra to that of the standard, the following criteria must be met:

- All ions present in the standard mass spectrum at a relative intensity greater than 10.0 percent (most abundant ion in the spectrum equals 100.0 percent) must be present in the sample spectrum.

- The relative intensities of ions specified in the paragraph above must agree within ±20.0 percent between the standard and sample spectrum. (Example: For an ion with an abundance of 50.0 percent in the standard spectrum, the corresponding sample ion abundance must be between 30.0 and 70.0 percent).

- Ions greater than 10.0 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. When target compounds are below contract required quantitation limits (CRQL) but the spectrum meets the identification criteria, report the concentration with a "J".

If a compound does not meet all of the above criteria, but in the technical judgement of the mass spectral interpretation specialist the identification is correct, the compound will be identified. Documentation of such by the specialist on the raw data is required.

14.3.2 Non-Target Compounds

A library search may be executed for non-target sample components for the purpose of tentative identification. For this purpose, the NIST/EPA/NIH mass spectral library is used to identify non-target compounds of greatest apparent concentration by a forward search of the library. The following compounds will not be identified by a library search routine:

a. Internal standard compounds
b. Surrogate compounds
c. Volatile target compounds

Peaks that are suspected to be aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-7-pentanone and 4-methyl-3-pentene-2-one) are searched and reported as part of the 30 tentatively identified compounds.

14.3.3 Guidelines for Making Tentative Identifications

Major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ±20 percent.
Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

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 compounds.

If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made the compound will be reported as unknown. Further identification may be possible, such as molecular weights or classifications (i.e., unknown hydrocarbon, unknown acid, etc.)

Pesticide target compounds may be tentatively identified by a library search.

14.4 Technical Acceptance Criteria For Sample Analysis

The samples must be analyzed on a GC/MS system meeting the DFTPP initial calibration, continuing calibration, and blank technical acceptance criteria. The sample must undergo cleanup procedures, when required, on a GPC meeting the acceptance criteria for GPC calibration.

The sample must be extracted and analyzed within the holding times.

The sample must have an associated method blank meeting the blank acceptance criteria.
All Matrix Spike Blank recoveries must fall within the laboratory derived limits. Recoveries above the upper control limit are acceptable as long as the analyte was not detected in the associated samples above the quantitation limit.

All surrogates should fall within the laboratory derived limits (Up to one BN and/or one AP surrogate may fall outside the control limit as long as the recovery is greater than 10%).

The relative retention time of each surrogate must be within ±0.06 RRT units of its relative

retention time in the continuing calibration standard.

The instrumental response (EICP area) for each of the internal standards must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the most recent continuing calibration analysis.

The retention time shift for each of the internal standards must be within ±0.50 minutes (30 seconds) between the sample and the most recent continuing calibration standard analysis.

Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the 12-hour standard calibration range unless a more dilute aliquot of the sample extract is also analyzed.

14.5 Corrective Actions for Sample Analysis

The technical acceptance criteria must be met before data are reported. Contamination from laboratory sources requires re-extraction and reanalysis.

14.5.1 Surrogate Compounds

If the technical acceptance criteria for surrogate compound recoveries is not met, the following corrective actions are taken in the given order:

a. Calculations, injection volumes, preparation volumes are checked to insure that an error was not made; if all calculations, volumes, etc., were correct the analyst will proceed to the next step in the corrective action process.

b. The sample is re-injected to insure that an error during injection was not made. If after re-injection, surrogate recoveries are outside of the acceptance criteria, the analysis will proceed to the next step in the corrective action process.

c. The sample is re-extracted. Exceptions: (1) in the case where the recoveries in a sample, MS/MSD agree (i.e., all samples exhibited recoveries outside of criteria limits) it will be noted in the Case narrative. (2) Insufficient sample remains for re-extraction. In this instance, the client will be contacted in order to determine the next procedure to follow. If this situation should arise, it will be documented in the Case narrative. (see form B: Re-extraction request form).

d. After re-extraction, the sample is re-injected. If after re-analysis surrogate recoveries are within criteria limits, this extract is considered the first because the original problem may have been due to a laboratory error. If, after re-analysis surrogate recoveries are not within criteria limits, a matrix effect may be assumed.

If this should occur, both analyses may be reported. The instance will be documented in the Case Narrative.

14.5.2 Internal Standard Compounds

If the technical acceptance criteria for internal standard recoveries is not met, the following corrective actions are taken in the given order:

a. Calculations, internal standard solution volumes and injected volumes are checked to insure that an error was not made. If all calculations and volumes were correct the analyst will proceed to the next step in the corrective action process.

b. The sample is re-injected to insure that the instrument was working properly. If after re-analysis, the internal standard recoveries are with criteria limits, the second analysis will be reported only. If after re-analysis the internal standard recoveries are outside of criteria limits, both analyses will be reported and it may be assumed that a matrix effect was involved. If this instance should arise, it will be documented in the Case Narrative.

Exception: If internal standard recoveries of a sample, MS/MSD agree (i.e., recoveries are outside of criteria limits for all three samples, it may be assumed that a matrix effect is involved and no corrective action is necessary. The instance will be documented in the Case Narrative.

14.5.3 Relative Retention Times

If the technical acceptance criteria for the relative retention times of the internal standard compounds or surrogate compounds are not met, the following corrective actions are taken in the given order:

a. Carrier gas, zone temperatures and instrument temperature programs are checked to insure that an error was not made or that the gas tank was not dry or clogged. If no errors are found the analyst will proceed to the next step in the corrective action process.

b. The sample is re-analyzed to insure that an error was not made during the first injection. If, after reanalysis, the relative retention times are not within the technical acceptance criteria, it may be assumed that a matrix effect was involved. Both analyses will be reported and the instance will be documented in the Case Narrative. If, after re-analysis, the relative retention times are within the technical acceptance criteria, the second analysis will be reported only.

Exception: If the relative retention times of a sample, MS/MSD agree (i.e., relative

retention times are outside of criteria limits for the sample, MS and MSD, it may be assumed that a matrix effect was involved and further corrective action is not necessary.

14.5.4 Matrix Spike Blanks.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analysis must be included with the report.

14.6 Injection Logs: Injection Logs must contain the following information:

a. Date, time, and analyst initials
b. File number (FRN), sample ID, vial #, and job #
c. Injection volume, final volume, initial volume and dilution factor
d. Indicate if tailing of degradation was present in the tune
e. References for the standard, tune mix, IS mix
f. Daily maintenance performed
g. Any non-conformances with the samples

15.0 CALCULATIONS:

15.1. Target Compounds

Target compounds identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (see Table 4). The EICP area of primary characteristic ions of analytes listed in Tables 3 and 6 are used for quantitation.

In instances where manual quantitation is necessary due to co-elution baseline noise or matrix interferences, all instances will be initialed and dated by the analyst. The quantitation report is documented as such by an "m" next to the compound that has been edited. In all instances of manual integration, a hardcopy of the EICP for that compound will be supplied with the raw data, this applies to all target compounds, internal standards and surrogate compounds.

The average response factor (RRF) from the initial calibration analysis (linear model) is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, the reason is then documented in the case Narrative. The area of a secondary ion cannot be used for the area of a primary ion unless a relative factor is calculated using the secondary ion.

15.2 Water Samples

The following Equation (Eq. 8) is used to determine the concentration of target compounds identified in water samples:

Equation 8

$$Concentration\ \mu g/L = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRF)(V_o)(V_i)}$$

Where,

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the internal standard

* I_s = Amount of internal standard injected in nanograms (ng)

V_o = Volume of water extracted in milliliters (mL)

V_i = Volume of extract injected in microliters (µL)

V_t = Volume of the concentrated extract in microliters (µL) (V_t = 1,000 µL if sample was not subjected to GPC; V_t = 500 µL if sample was subjected to GPC)

RRF= Relative response factor determined from the 12-hour calibration standard

GPC= GPC factor.

GPC= 1.0 if water sample was not subjected to GPC;

Df = Dilution factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

$$\frac{\text{µL most conc. extract used to make dilution + µL clean solvent}}{\text{µL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0

15.3 Soil/Sediment Samples

The following Equation (Eq. 9) is used to determine the concentration of target compounds in soil/sediment samples:

Equation 9

$$Concentration\ \mu g/Kg\ (Dry\ weight\ basis) = \frac{(A_x)(I_3)(V_t)(Df)(GPC)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

Where,

A_x, I_s, A_{is} are as given for water, above.

V_t = Volume of the concentrated extract in microliters (µL) (V_t = 500 µL)

V_i = Volume of the extract injected in microliters (µL)

$D = \frac{100 - \%\ moisture}{100}$

W_s = Weight of sample extracted in grams (g)

GPC= GPC factor (GPC = 2.0 to account for GCP cleanup)

RRF= Relative response factor determined from the 12-hour calibration standard.
Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for semivolatile by this method is defined as follows:

$$\frac{\mu L \text{ most conc. extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator is used to account for the amount of extract not recovered from the use of GPC cleanup. Concentrating the extract collected after GPC to 0.5mL maintains the sensitivity of the soil/sediment method.

15.4 Tentatively Identified Compounds

Non-Target Compounds
An estimated concentration for non-target compounds tentatively identified is quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences is to be used. The equations for calculating concentrations are the same as equations 8 and 9. Total area counts (or peak heights) from the total ion chromatograms are used for both the compounds to be measured and the internal standard. A relative response factor (RRF) of one (1) is assumed. The resulting concentration is to be qualified as "J" (estimated, due to lack of a compound specific response factor), and "N" (Presumptive evidence of presence), indicating the quantitative and qualitative uncertainties is calculated for all tentatively identified compounds as well as those identified as unknowns.

15.5 Rounding

For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop is (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is off, and round down if that digit is even.

15.6 Organic Significant Figures

For volatile and semivolatile results, report analytical results to one significant figure if the value is less than 10, and two significant figures if the value is above 10.

16.0 METHOD PERFORMANCE:

16.1 Method Detection Limits (MDLs)
MDL's are seven blank samples spiked with 20ng/mic of all compounds of interest. These are

extracted and analyzed as both waters and soils along with a blank.

16.2 Initial Demonstration of Capability (IDOC): The initial demonstration with each sample preparation and determinative method combination utilized must be performed by generating data of acceptable accuracy and precision for target analytes in a clean matrix. This is also done for new staff or when significant changes in instrumentation are made as stated in section 8.0 of Method 8000.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:

17.1 When internal standards are out of range a re-injection is required unless the problem can be determined to be a result of excessive matrix interference.

17.2 When surrogates are out of range, a re-extraction is required unless excessive visible chromatographic matrix interference is present. In this case, the Project Manager should be consulted to decide how to proceed.

17.3 When a positive hit for an analyte is above the calibration range a dilution must be performed to bring the value within calibration range .

17.4 When there are low spike recoveries in the matrix spike blank the entire extraction batch needs to be re-extracted. If there are high spike recoveries the associated sample data needs to be examined to assess if it may be biased.

18.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA:

18.1 Corrective actions for out of control data require Project Manager, Laboratory Director and/or QA Officer notification. This can be accomplished either verbally, written using a Job Exception Report or both.

19.0 CONTINGIENCIES FOR HANDLING OUT –OF- CONTROL OR UNACCEPTABLE DATA:

19.1 Contingency measures for handling out of control or unacceptable data requires The Project Manager to notify the client for input.

20.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

20.1 There are two types of aqueous waste generated in the lab:

1. A-Waste: All non-nitric acid and alkaline aqueous waste.
2. AN-Waste: All aqueous waste containing nitric acid.

These types of waste are to be disposed of into appropriately market plastic containers.

The following are the other types of lab waste and where to dispose of:

1. C-Waste: all solvent waste gets dumped into appropriately marked metal cans. These cans need to be grounded whenever they are emptied to reduce explosion hazards. Discarded standards will also be dumped into C-waste cans.

2. Solid Waste: all contaminated paper, solid sample waste, sodium sulfate and all other non-glass material that has been contaminated is to be wrapped in foil and gathered to be dumped into 55 gallon drums.

3. Glass: contaminated glass needs to be rinsed off with methylene chloride and disposed of with all other glass in glass specific containers with special extra thick polypropylene liners. These containers are for glass only.

4. Extract Vials: extract vials are to be archived after they have been shot. After archival period, vials are to be crushed into a 55 gallon drum.

21.0 REFERENCE

21.1 USEPA Methods for Evaluating Solid Waste; SW-846, Third Edition, Update III, Method 8270C, 12/96.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA:

22.1 Attachment A: SOP Procedure Summary
22.2 Attachment B: AFCEE Summary
22.3 Attachment C: USACE Summary
22.4 Table 1: Target Compound List and EQLs
22.5 Table 2: Ion Abundance Criteria
22.6 Table 3: Characteristic Ions for Internal Standards
22.7 Table 4: Internal Standards and Corresponding Target Compounds Assigned for Quantitation
22.8 Table 5: Relative Response Factor Criteria for ICV and CCV
22.9 Table 6: Characteristic Ions for Target Compounds and Surrogates
22.10 Attachment D: Job Summary Checklist

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 10.0: Provided calibration concentrations in units of measure comparable to final sample concentrations.
23.2 Added Job Summary Checklist as Attachment D.
23.3 Added corporate safety and waste management sections.

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TITLE: **ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C**

SUPERCEDES: Revision 5

23.4 Updated appropriate sections to reflect the change over to new instrumentation.
23.5 Where necessary, changed the work 'should' to 'shall'.

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TITLE: **ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C**

SUPERCEDES: Revision 5

ATTACHMENT A - SOP PROCEDURE SUMMARY

I. Preparing the instrument;
1. Cut column, change liner and septa, inject conditioning solution
2. Ramp GC oven temp. to 325°C and ramp GC inj. Port pressure to 80 psi to see if pressure holds.

II. Shoot DFTPP tune mix
1. Shoot 1 ul of the dftpp tune mix
2. Evaluate the DFTPP peak using the 3rd Edition or criteria
3. Evaluate the tailing factors of pentachlorophenol and benzidine.
4. Evaluate the degradation of 4,4'-DDT to 4,4'-DDD and 4,4'-DDE.

III. Shoot single or 5pt. calibration;
1. Shoot 1 ul of the 50ng continuing standard (CCC)
2. Evaluate the continuing; 4pts may be out but none over 40%d.
3. If CCC does not pass criteria, then a 5pt. curve (ICC) must be shot.

IV. Load Samples;
1. Load blanks and MSBs in the beginning and dark samples toward the end.
2. Very thick samples may be diluted.
3. All samples must be shot within 12 hours of the tune injection.

V. Analyze data;
1. Quantitate all samples; need raw and enhanced spectra for positive and negative hits and 20 TICs .
2. Shoot dilutions on any samples with positive hits over 160ng.
3. Shoot reinjections (RI's) on any sample that has internal standards out, unless there is severe matrix interference that accounts for the low recovery.
4. Samples with more than one BN or AP surrogate out needs to be re-extracted (RE).

VI. AIMS Entry;
1. Enter tunes, ICC's and CCC's.
2. Enter all samples to be included with the job.
3. Identify and enter all TIC's
4. Calculate, close and run data validator.

VII. Review Data;
1. Correct or explain any errors on the data validator.
2. Make copies of logbooks, tunes, curves and standards and include them with the report.
3. Check that all calculations have been made correctly.
4. Turn in job for validation.

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AttachmentB

Semivolatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using method SW8270C. This technique determines quantitatively the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column GC/mass spectrometer. The RLs are listed in Table 7.2.10-1.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 51 30 percent to 60 percent of mass 198
- mass 68 less than 2 percent of mass 69
- mass 70 less than 2 percent of mass 69
- mass 127 40 percent to 60 percent of mass 198
- mass 197 less than 1 percent of mass 198
- mass 198 base peak, 100 percent relative abundance
- mass 199 5 percent to 9 percent of mass 198
- mass 275 10 percent to 30 percent of mass 198
- mass 365 greater than 1 percent of mass 198
- mass 441 present, but less than mass 443
- mass 442 greater than 40 percent of mass 198
- mass 443 17 percent to 23 percent of mass 442

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample.

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C	1,2,4-Trichlorobenzene	10.0	µg/L	0.7	mg/kg
	1,2-DCB	10.0	µg/L	0.7	mg/kg
	1,3-DCB	10.0	µg/L	0.7	mg/kg
	1,4-DCB	10.0	µg/L	0.7	mg/kg
	2,4-DNT	10.0	µg/L	0.7	mg/kg
	2,6-DNT	10.0	µg/L	0.7	mg/kg
	2-Chloronaphthalene	10.0	µg/L	0.7	mg/kg
	2-Methylnaphthalene	10.0	µg/L	0.7	mg/kg
	2-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3,3'-Dichlorobenzidine	20.0	µg/L	1.3	mg/kg
	4-Bromophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Chloroaniline	20.0	µg/L	1.3	mg/kg
	4-Chlorophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Nitroaniline	50.0	µg/L	3.3	mg/kg
	Acenaphthylene	10.0	µg/L	0.7	mg/kg
	Acenaphthene	10.0	µg/L	0.7	mg/kg
	Anthracene	10.0	µg/L	0.7	mg/kg
	Benz (a) anthracene	10.0	µg/L	0.7	mg/kg
	Benzo (a) pyrene	10.0	µg/L	0.7	mg/kg
	Benzo (b) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (b) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (g,h,i) perylene	10.0	µg/L	0.7	mg/kg
	Benzyl alcohol	20.0	µg/L	1.3	mg/kg
	Bis (2-chloroethoxy) methane	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroethyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroisopropyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-ethylhexyl) phthalate	10.0	µg/L	0.7	mg/kg
	Butyl benzylphthalate	10.0	µg/L	0.7	mg/kg
	Chrysene	10.0	µg/L	0.7	mg/kg
	Di-n-butylphthalate	10.0	µg/L	0.7	mg/kg
	Di-n-octylphthalate	10.0	µg/L	0.7	mg/kg
	Dibenz (a,h) anthracene	10.0	µg/L	0.7	mg/kg
	Dibenzofuran	10.0	µg/L	0.7	mg/kg
	Diethyl phthalate	10.0	µg/L	0.7	mg/kg
	Dimethyl phthalate	10.0	µg/L	0.7	mg/kg
	Fluoranthene	10.0	µg/L	0.7	mg/kg
	Fluorene	10.0	µg/L	0.7	mg/kg
	Hexachlorobenzene	10.0	µg/L	0.7	mg/kg
	Hexachlorobutadiene	10.0	µg/L	0.7	mg/kg
	Hexachloroethane	10.0	µg/L	0.7	mg/kg
	Indeno (1,2,3-cd) pyrene	10.0	µg/L	0.7	mg/kg
	Isophorone	10.0	µg/L	0.7	mg/kg

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C (concluded)	n-Nitrosodiphenylamine	10.0	µg/L	0.7	mg/kg
	n-Nitrosodi-n-propylamine	10.0	µg/L	0.7	mg/kg
	Naphthalene	10.0	µg/L	0.7	mg/kg
	Nitrobenzene	10.0	µg/L	0.7	mg/kg
	Phenanthrene	10.0	µg/L	0.7	mg/kg
	Pyrene	10.0	µg/L	0.7	mg/kg
Semivolatile organics Acid Extractables SW8270C	2,4,5-Trichlorophenol	50.0	µg/L	3.3	mg/kg
	2,4,6-Trichlorophenol	10.0	µg/L	0.3	mg/kg
	2,4-Dichlorophenol	10.0	µg/L	0.3	mg/kg
	2,4-Dimethylphenol	10.0	µg/L	0.3	mg/kg
	2,4-Dinitrophenol	50.0	µg/L	3.3	mg/kg
	2-Chlorophenol	10.0	µg/L	0.3	mg/kg
	2-Methylphenol	10.0	µg/L	0.3	mg/kg
	2-Nitrophenol	10.0	µg/L	0.3	mg/kg
	4,6-Dinitro-2-methylphenol	50.0	µg/L	3.3	mg/kg
	4-Chloro-3-methylphenol	20.0	µg/L	1.3	mg/kg
	4-Methylphenol	50.0	µg/L	2.0	mg/kg
	4-Nitrophenol	50.0	µg/L	1.6	mg/kg
	Benzoic acid	100	µg/L	5.0	mg/kg
	Pentachlorophenol	50.0	µg/L	3.3	mg/kg
	Phenol	10.0	µg/L	0.3	mg/kg

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Table 7.2.10-2. QC Acceptance Criteria for Method SW8270C

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc. Sur.
SW8270C	1,2,4-Trichlorobenzene	37–120	≤20	44–125	≤30	2	4
	1,2-DCB	33–120	≤20	45–125	≤30	1	3
	1,3-DCB	32–120	≤20	39–125	≤30	1	3
	1,4-DCB	32–120	≤20	36–125	≤30	1	3
	2,4-DNT	51–120	≤20	48–125	≤30	3	4
	2,6-DNT	49–120	≤20	48–125	≤30	3	4
	2-Chloronaphthalene	49–120	≤20	45–125	≤30	3	4
	2-Methylnaphthalene	46–120	≤20	47–125	≤30	2	5
	2-Nitroaniline	48–120	≤20	44–125	≤30	3	2
	3,3'-Dichlorobenzidine	20–120	≤20	25–128	≤30	5	6
	3-Nitroaniline	26–126	≤20	27–125	≤30	3	2
	4-Bromophenyl phenyl ether	53–120	≤20	46–125	≤30	4	1
	4-Chloroaniline	20–120	≤20	25–125	≤30	2	5
	4-Chlorophenyl phenyl ether	50–120	≤20	47–125	≤30	3	4
	4-Nitroaniline	36–120	≤20	34–125	≤30	3	2
	Acenaphthylene	50–120	≤20	44–125	≤30	3	4
	Acenaphthene	47–120	≤20	46–125	≤30	3	4
	Anthracene	54–120	≤20	53–125	≤30	4	1
	Benz (a) anthracene	56–100	≤20	52–125	≤30	5	6
	Benzo (a) pyrene	53–120	≤20	50–125	≤30	6	6
	Benzo (b) fluoranthene	45–124	≤20	45–125	≤30	6	6
	Benzo (g,h,i) perylene	38–123	≤20	38–126	≤30	6	6
	Benzo (k) fluoranthene	45–124	≤20	45–125	≤30	6	6
	Benzyl alcohol	30–120	≤20	25–125	≤30	1	3
	Bis (2-chloroethoxy) methane	46–120	≤20	43–125	≤30	2	5
	Bis (2-chloroethyl) ether	37–120	≤20	38–125	≤30	1	3
	Bis (2-chloroisopropyl) ether	26–131	≤29	25–125	≤30	1	3
	Bis (2-ethylhexyl) phthalate	42–126	≤20	47–127	≤30	5	6
	Butyl benzyl phthalate	46–120	≤20	49–125	≤30	5	6
	Chrysene	55–120	≤20	53–125	≤30	5	6
	Di-n-butyl phthalate	54–120	≤20	56–125	≤30	4	1
	Di-n-octyl phthalate	37–137	≤20	41–132	≤30	5	6
	Dibenz (a,h) anthracene	42–127	≤20	41–125	≤30	6	6
	Dibenzofuran	54–120	≤20	51–125	≤30	3	4
	Diethyl phthalate	41–120	≤20	50–125	≤30	3	4
	Dimethyl phthalate	25–127	≤20	49–125	≤30	3	4
	Fluoranthene	54–120	≤20	54–125	≤30	4	1
	Fluorene	50–120	≤20	49–125	≤30	3	2
SW8270C (Continued)	Hexachlorobenzene	52–120	≤20	47–125	≤30	4	1
	Hexachlorobutadiene	27–120	≤20	40–125	≤30	2	5
	Hexachloroethane	28–120	≤20	34–125	≤30	1	3
	Indeno (1,2,3-c,d) pyrene	43–125	≤20	38–125	≤30	5	6
	Isophorone	50–128	≤20	43–125	≤30	2	5
	n-Nitroso-di-n-propylamine	34–128	≤20	40–125	≤30	1	3
	n-Nitrosodiphenylamine	48–120	≤20	49–125	≤30	4	1
	Naphthalene	39–120	≤20	40–125	≤30	2	5
	Nitrobenzene	44–120	≤20	41–125	≤30	2	4
	Phenanthrene	51–120	≤20	50–125	≤30	4	1
	Pyrene	49–128	≤20	46–125	≤30	5	6
	2,4,5-Trichlorophenol	49–120	≤20	49–125	≤30	3	1
	2,4,6-Trichlorophenol	49–126	≤20	48–125	≤30	3	1
	2,4-Dichlorophenol	48–120	≤20	45–125	≤30	2	5
	2,4-Dimethylphenol	28–120	≤20	32–125	≤30	2	5
	2,4-Dinitrophenol	25–130	≤20	25–132	≤30	3	4
	2-Chlorophenol	37–120	≤20	44–125	≤30	1	3
	2-Methylphenol	38–120	≤20	40–125	≤30	1	3
	2-Nitrophenol	39–123	≤20	42–125	≤30	2	4
	4,6-Dinitro-2-Methyl Phenol	40–130	≤20	29–137	≤30	4	1
	4-Chloro-3-Methyl Phenol	47–120	≤20	46–125	≤30	2	5
	4-Methylphenol	32–120	≤20	41–125	≤30	1	3
	4-Nitrophenol	20–120	≤20	25–138	≤30	3	2
	Benzoic Acid	20–120	≤20	25–125	≤30	2	5
	Pentachlorophenol	38–120	≤20	25–125	≤30	4	1
	Phenol	20–120	≤20	39–125	≤30	1	5

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Number
SW8270C (Continued)	*Surrogates:*					
	2,4,6-Tribromophenol	42–124		36–126		1
	2-Fluorobiphenyl	48–120		43–125		2
	2-Fluorophenol	20–120		37–125		3
	Nitrobenzene-D5	41–120		37–125		4
	Phenol-D5	20–120		40–125		5
	Terphenyl-D14	51–135		33–125		6
	Internal Standards:					
	1,4-Dichlorobenzene-D4					1
	Naphthalene-D8					2
	Acenaphthene-D10					3
	Phenanthrene-D10					4
	Chrysene-D12					5
	Perylene-D12					6

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Summary of Calibration and QC Procedures for Method SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression r ≥ 0.995 for each analyte		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification

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Summary of Calibration and QC Procedures for Method SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression r ≥ 0.995 for each analyte		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification

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Summary of Calibration and QC Procedures for Method SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression r ≥ 0.995 for each analyte		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification

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Attachment C

Summary of Measurement quality objectives for Method 8270 Semivolatiles

QC Element	Target Analyte/Surrogate	Poor Performers/Sporadic Marginal Failures[2]
Initial Calibration (I.9.2.2.7)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %RSD ≤ 30%	No allowance No allowance
	Primary Evaluation (all target analytes): r ≥ 0.995, %RSD ≤ 15%, r² ≥ 0.990	
	Alternative Evaluation: Mean %RSD for all target analytes ≤ 15%, with maximum allowable restriction noted at right for individual analytes.	Alternative Evaluation: Maximum allowable %RSD for each individual target analyte ≤ 40%
ICV (I.9.3)	%Rec = 70% - 130%	No allowance
CCV (I.9.5 / I.9.5.2 / I.9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements	No allowance
	Primary Evaluation (CCCs): %Drift ≤ 20%, %D ≤ 20%	No allowance
MB (I.10.2.1 / I.11.4.1)	Target Analytes: Analytes < one-half MRL	Common Lab Contaminants: Analytes < MRL
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 60% - 120% (~15 analytes) = 45% - 135% (~30 analytes) = 20% - 150% (~15 analytes) Solids: %Rec = 60% - 120% (~20 analytes) = 45% - 135% (~25 analytes) = 30% - 150% (~15 analytes)	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 25% - 150%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	Water: %Rec = 45% - 135% Solids: %Rec = 45% - 135%	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 20% - 150%
MSD/MD (I.10.2.4 / I.11.4.4)	Water: RPD ≤ 50% Solids: RPD ≤ 60%	Sporadic Marginal Failures[1]: Water: RPD ≤ 60% Solids: RPD ≤ 60%
Surrogates (I.10.2.5 / I.11.4.5)	%Interference-Free Matrix[2]: Water: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Solids: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Project Sample Matrix[2] Water: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds Solids: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 20% - 150%

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if the full list of target compounds as presented in Table 13 are reported, then 5 SMFs are allowed to the expanded criteria presented for the LCS. If the MS includes only a subset of compounds and for Surrogates, allow up to 1 SMF.
[2] B = base, N = neutral, and A = acid compounds (cmpds).

Number of Allowable QC Failures

N	X
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

Note: N = number of reported method target analytes
X = sporadic marginal failures allowed

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TABLE 1

Semivolatiles Target Compound List and Contract Estimated Quantitation Limits

	Semivolatiles	CAS Number	Estimated Quantitation Limits Water µg/L	Low Soil µg/Kg
34.	Phenol	108-95-2	10	330
35.	bis-(2-Chloroethyl)ether	111-44-4	10	330
36.	2-Chlorophenol	95-57-8	10	330
37.	1,3-Dichlorobenzene	541-73-1	10	330
38.	1,4-Dichlorobenzene	106-46-7	10	330
39.	1,2-Dichlorobenzene	95-50-1	10	330
40.	2-Methylphenol	95-48-7	10	330
41.	Bis(2-chloroisopropl)ether	108-60-1	10	330
42.	4-Methylphenol	106-44-5	10	330
43.	N-Nitroso-di-n-propylamine	621-64-7	10	330
44.	Hexachloroethane	67-72-1	10	330
45.	Nitrobenzene	98-95-3	10	330
46.	Isophorone	78-59-1	10	330
47.	2-Nitrophenol	88-75-5	10	330
48.	2,4-Dimethylphenol	105-67-9	10	330
49.	bis(2-Chloroethoxy) methane	111-91-1	10	330
50.	2,4-Dichlorophenol	120-83-2	10	330
51.	1,2,4-Trichlorobenzene	120-82-1	10	330
52.	Naphthalene	91-20-3	10	330
53.	4-Chloroaniline	106-47-8	20	1300
54.	Hexachlorobutadiene	87-68-3	10	330
55.	4-Chloro-3-methylphenol	59-50-7	20	1300
56.	2-Methylnaphthalene	91-57-6	10	330
57.	Hexachlorocyclopenta-diene	77-47-4	10	330
58.	2,4,6-Trichlorophenol	88-06-2	10	330
59.	2,4,5-Trichlorophenol	95-95-4	10	330
60.	2-Chloronaphthalene	91-58-7	10	330
61.	2-Nitroaniline	88-74-4	50	3300
62.	dimethylphthalate	131-11-3	10	330
63.	Acenaphthylene	208-96-8	10	330
64.	2,6-Dinitrotoluene	606-20-2	10	330
65.	3-Nitroanline	99-09-2	50	3300
66.	Acenaphthene	83-32-9	10	330
67.	2,4-Dinitrophenol	51-28-5	50	330
68.	4-Nitrophenol	100-02-7	50	3300

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			Estimated Quantitation Limits	
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
69.	Dibenzofuran	132-64-9	10	330
70.	2,4-Dinitrotoluene	121-14-2	10	330
71.	Diethlphthalate	84-66-22	10	330
72.	4-Chlorophenyl-phenyl ether	7005-72-3	10	330
73.	Fluorene	86-73-7	10	330
74.	4-Nitroaniline	100-01-6	50	3300
75.	4,6-Dinitro-2-methylphenol	534-52-1	50	3300
76.	N-Nitroso-diphenylamine	86-30-6	10	330
77.	4-Bromophenyl-phenylether	101-55-3	10	330
78.	Hexachlorobenzene	118-74-1	10	330
79.	Pentachlorophenol	87-86-5	50	3300
80.	Phenanthrene	85-01-8	10	330
81.	Anthracene	120-12-7	10	330
82.	Benzyl Alcohol	100-51-6	20	1300
83.	Di-n-butylphthalate	84-74-2	10	330
84.	Fluoranthene	206-44-0	10	330
85.	Pyrene	129-00-0	10	330
86.	Butylbenzylphthalate	85-68-7	10	330
87.	3,3-Dichlorobenzidine	91-94-1	20	1300
88.	Benzo(a)anthracene	56-55-3	10	330
89.	Chrysene	218-01-9	10	330
90.	bis(2-Ethylhexyl)phthalate	117-81-7	10	330
91.	Di-n-octylphthalate	117-84-0	10	330
92.	Benzo(b)fluoranthene	205-99-2	10	330
93.	Benzo(k)fluoranthene	207-08-9	10	330
94.	Benzo(a)pyrene	50-32-8	10	330
95.	Indeno(1,2,3-cd)-pyrene	193-39-5	10	330
96.	Dibenzo(a,h)-anthracene	53-70-3	10	330
97.	Benzo(g,h,i)perylene	191-24-2	10	330
98.	Benzoic Acid	65-85-0	50	3300

TABLE 2

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	Less than 2.0 percent of mass 69
70	Less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see Note)
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	Greater than 1.0% of than mass 198
441	Present but less than mass 443
442	40.0 – 110.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

Note: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be qreater to 110 percent that of m/z 198.

TABLE 3

Characteristic Ions for Internal Standards
for Semivolatile Compounds

Internal Standards	Primary Quantitation Ion	Secondary Ions
1,4-Dichlorobenzene-d4	152	115, 150
Naphthalene-d8	136	68
Acenaphthene-d10	164	162,160
Phenanthrene-d10	188	94, 80
Chrysene-d12	240	120, 236
Perylene-d12	264	260, 265

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TABLE 4
Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}	Phenanthrene-d	Chrysene-d_{12}	Perylene-d_{12}
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Benzo(b)fluoranthene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-nitroso-di-phenylamine	Butylbenzylphthalate	Benzo(k)fluoranthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenylphenolether	3,3'-Dichlorobenzidine	Benzo(a)phyrne
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloroaphthalene	Hexachlorobenzene	Benzo(a)-anthracene	Indeno(1,2,3-cd)-pyrene
1,4-Dichlorobenzene	bis(2-Chloroethoxy) methane	2-Nitroaniline	Pentachlorophenol	bis(2-ethyl-hexyl)phthalate	Benzo(g,h,i)-perylene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethylphthalate	Carbzole	Chrysene	Dibenzo(a,h)-anthracene
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene	Phenanthrene	Terphenyl-d_{14} (surr)	
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline	Anthracene	Di-n-octyl-phthalata	
4-Methylphenol	4-Chloroanaline	Acenaphthene	Di-n-butylphthalate		
N-Nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitorphenol	Fluoranthene		
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol			
2-Fluorophenol(surr)	2-Methylnaphthalene	Dibenzofuran			
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,4-Dinitrotoluene			
4-methylphenol	Benzoic acid	2,6-Dinitrotoluene			
Aniline	4-chloroaniline	Diethylphthalate			
Benzyl Alcohol	N-Nitrosobutyl-amine	4-Chlorophenyl-phenylether			
		Fluorene			
		4-Nitroaniline			

		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			

TABLE 5

Relative Response Factor Criteria for Initial and Continuing
Calibration of Semivolatile Target Compounds and Surrogates

Semivolatile Compounds	Minimum RRF	Maximum % RSD	Maximum % Diff
Acenaphthene (CCC)	none	30	±20
1,4-Dichlorobenzene (CCC)	none	30	±20
Hexachlorobutadiene (CCC)	none	30	±20
N-Nitrosodiphenylamine (CCC)	none	30	±20
Di-n-octylphthalate (CCC)	none	30	±20
Flouranthene (CCC)	none	30	±20
Benzo(a)pyrene (CCC)	none	30	±20
4-Chloro-3-methylphenol (CCC)	none	30	*20
2,4-Dichlorophenol (CCC)	none	30	±20
2-Nitrophenol (CCC)	none	30	±20
Phenol (CCC)	none	30	±20
Pentachlorophenol(CCC)	none	30	±20
2,4,6-Trichlorophenol (CCC)	none	30	±20
N-Nitroso-di-n-propylamine (SPCC)	0.050	None	none
Hexachlorocyclopentadiene (SPCC)	0.050	None	none
2,4-Dinitrophenol (SPCC)	0.050	None	none
4-Nitrophenol (SPCC)	0.050	None	none

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TABLE 6

Characteristic Ions for Semivolatile
Target Compounds and Surrogates

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
Bis(2-chloroisopropyl)ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272

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Parameters	Primary Quantitation Ion	Secondary Ion(s)
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Benzyl Alcohol	108	79, 77
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100

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Parameters	Primary Quantitation Ion	Secondary Ion(s)
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octylphthalate	149	—
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138,277
Benzoic Acid	122	105, 77
SURROGATES		
Phenol-d5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribormophenol	330	332, 141
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d14	244	122, 212

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Attachment D: Job Summary Checklist

Job Number: _____ Instrument # _____
Method: _____

Yes/No	Primary Review
	Tunes passed?
	ICC's passed?
	CCV's passed?
	Quantitations have been performed correctly?
	Qualitative identifications are accurate?
	Client specific requirements have been followed?
	Method and process SOP's have been followed?
	Method and/or QUAPP specific QC criteria have been met?
	QC samples are within established limits?
	Dilution factors are correctly recorded and applied?
	Non-conformances and/or anomalous data have been properly documented and communicated?
	Job folder complete?

Check	Secondary Review
	Qualitative identification.
	Quantitative accuracy.
	Calibration.
	QC samples.
	Method and/or QUAPP specific QC criteria.
	Adherence to method and process SOP's.

Comments: _____
_____

Analyst: _____ Date: _____
AIMS Entry: _____ Date: _____
Review: _____ Date: _____

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES
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REVIEWED & APPROVED BY:	Signature	Date
Verl Preston, Quality Manager	Verl D. Preston	5/31/06
Christopher Spencer, Laboratory Director		5/31/06
James Lis, Supervisor		5/31/06

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1.0 IDENTIFICATION OF TEST METHODS

1.1. Methods 8260B -5 mL aqueous purge, 8260B - 25mL aqueous purge, 8260B – 5gr soil and 8260B – medium level soil.

2.0 APPLICABLE MATRIX

2.1. Applicable matrices include all aqueous samples, sediment, and soil.

3.0 REPORTING LIMIT

3.1. The standard reporting limit (RL) is established at or above the lo-level standard in the calibration curve. For a 5-ml purge volume, thee RL for the majority of compounds is 1 ug/l.

4.0 SCOPE AND APPLICATION

4.1. The analytical method is utilized for the analysis of water, sediment and soil from hazardous waste sites for the organic compounds listed in table 1.

4.2. The method includes sample preparation and analyses by purge and trap gas chromatograph/mass

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spectrometer (GC/MS). Method can be used for 5mL purge or 25mL purge (concentrations adjusted accordingly).

5.0 SUMMARY OF TEST METHOD

5.1. Volatile compounds are extracted from sample matrix by the purge and trap method. Analytes are desorbed onto a capillary column. An appropriate ramping temperature program is applied to maximize separation and achieve the correct resolution between the analytes. A mass spectrometer detector (MSD) interfaced to the gas chromatograph (GC) is utilized to detect analytes of interest.

5.2. Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

6.0 DEFINITIONS

6.1. VBLK - Volatile blank: VBLK's are made from laboratory produced volatile free water. They are analyzed before samples to ensure a clean laboratory environment and analytical system.

6.2. IBLK – Instrument Blank: IBLK's are made from laboratory produced volatile free water. They are analyzed after high level samples to verify that the system is clean and demonstrate the absence of carryover.

7.0 INTERFERENCES

7.1. Airborne contamination may result from solvent vapors. VBLKs and IBLKs will be utilized to demonstrate a clean system and laboratory environment.

7.2. Some volatile compounds can permeate through a sample septum seal during storage or shipment. A weekly volatile holding blank is stored will all samples in the sample incubator to monitor contamination.

7.3. Contamination by carryover can occur whenever a sample with high concentrations of target compounds precedes a sample with low levels. The purging device, syringe and lines are flushed between every analysis to reduce carry over contamination. The trap is baked at 260^0 C between each analysis.

8.0 SAFETY

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8.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.2. *SPECIFIC SAFETY CONCERNS OR REQUIREMENTS*

8.2.1. The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

8.2.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

8.2.3. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

8.3. PRIMARY MATERIALS USED

8.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1. Glassware

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9.1.1. Syringes - Hamilton Syringes size, 10ul, 25ul, 50ul, 100ul, 500ul, 1ml, 5ml, 10ml, 25ml
9.1.2. Pasteur Pipets – disposable
9.1.3. Vials and caps - 2ml disposable
9.1.4. Vials and caps - 40ml disposable
9.1.5. Volumetric flasks - pyrex 2ml, pyrex 10ml, pyrex 50ml, pyrex 100ml

9.2. pH paper - wide range -.EM Science

9.3. Analytical Balance Mettler - Toledo Inc. Mettler AE160

9.4. Purge and trap devices

9.4.1. PTA-30 W/S by Dynatech Autosampler
9.4.2. Tekmar 2000 Concentrator
9.4.3. Varian Archon Autosampler
9.4.4. Tekmar 3000 Concentrator
9.4.5. Encon Concentrator

9.5. Trap Packing - Supelco Vocarb 3000

9.5.1. Packing Material:
- 9.5.1.1. 10cm Carbpack B
- 9.5.1.2. 6cm Carboxen 1000
- 9.5.1.3. 1cm Carboxen 1001

9.6. Gas Chromatograph/Mass Spectrometer (GC/MS) - GC: HP5890, MS: Finnigan INCOS 50 and Finnigan INCOS 50XL

9.7. Gas chromatograph Column - J&W Scientific DB-624 or Phenomenex ZB-624

9.7.1. Internal diameter: 0.53mm
9.7.2. Length: 75m
9.7.3. Coating: Cyanopropylphenyl Methyl Silicone
9.7.4. Film thickness: 3.0µm

9.8. Data System -

9.8.1. Finnigan INCOS software/Data General DG-10 computer
9.8.2. Dell computer with Teknivent Vector/Warp enviroquant software
9.8.3. Gas Chromatograph/Mass Spectrometer (GC/MS) - GC: HP6890, MS: Hewlett-Packard/Agilent 5973N

9.9. Gas chromatograph Column - J&W Scientific DB-624 or Phenomenex ZB-624

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9.9.1. Internal diameter: 0.25mm or .18mm
9.9.2. Length: 60m or20m
9.9.3. Coating: Cyanopropylphenyl Methyl Silicone
9.9.4. Film thickness: 1.4µm or 1µm

9.10. Data System - Hewlett-Packard Kayak XM600 computer with Chemstation software.

10.0 REAGENTS AND STANDARDS

10.1. Reagent Water - For volailte analysis, the reagent water is volatile free and is prepared by passing water through a carbon trap.

10.2. Methanol - Burdick & Jackson, purge and trap grade

10.3. Stock Standards- Are purchased as certified standard mixtures. Traceability is documented following the procedures in the "Standards Traceability and Preparation Logbooks" SOP# AGP-STD-14. Individual compounds are prepared using reagent grade chemicals following the "Primary Standards Preparation" SOP# AMV-STD-25.

10.3.1. Stock Target Compound Mix – Is composed of three different mixtures.
- 10.3.1.1. The Gas Mix (See Table 6 for component list) is purchased from Supelco (or equivalent vendor) at a concentration of 2000ug/ml.
- 10.3.1.2. The 54 Component Mix (See Table 7 for component list) is purchased from Supelco (or equivalent vendor) at a concentration of 2000ug/ml.
- 10.3.1.3. The 8260+ Mix (See Table 8 for component list) is purchased from Restek (or equivalent vendor) and is composed of four separate mixtures.
  - 10.3.1.3.1. 8260+ Mix #1 is purchased at a concentration of 1000ug/ml.
  - 10.3.1.3.2. 8260+ Mix #2 is purchased at a concentration of 5000ug/ml.
  - 10.3.1.3.3. 8260+ Mix #3 is purchased at a concentration of 20000ug/ml.
  - 10.3.1.3.4. 8260+ Mix #4 is purchased at a concentration of 5000ug/ml.

10.3.2. Stock Calibration Verification Mix – Is composed of two different mixtures.
- 10.3.2.1. The Second Source Mix (See Table 9 for component list) is purchased from Ultra (or equivalent vendor) at a concentration of 2000ug/ml.
- 10.3.2.2. The 8260+ Second Source Mix (See Table 10 for component list) is purchased from Supelco (or equivalent vendor) and is composed of two separate mixtures.
  - 10.3.2.2.1. 8260+ Second Source Mix #1 is purchased at a concentration of 1000ug/ml.
  - 10.3.2.2.2. 8260+ Second Source Mix #2 is purchased at a concentration of 5000ug/ml.

10.3.3. Stock Internal Standard Solution – A mixture of 1,4-Dichlorobenzene-d4, Chlorobenzene-d5 and 1,4-Difluorobenzene in Methanol is purchased from Restek (or

equivalent vendor) at a concentration of 2500ug/ml.

10.3.4. Stock System Monitoring Solution – A mixture of Dibromofluoromethane, Toluene-D8, 4-Bromofluorobenzene and 1,2-Dichloroethane-d4 in Methanol is purchased from Ultra (or equivalent vendor) at a concentration of 2500ug/ml.

10.3.5. Stock Matrix Spike Solution – A 5 component mixture of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene in Methanol is purchased from Restek (or equivalent vendor) at a concentration of 2500ug/ml.

10.3.6. Stock BFB Solution – A solution of 4-Bromofluorobenzene in Methanol is purchased from Supelco (or equivalent vendor) at a concentration of 25000ug/ml.

10.4. Secondary IS and System Monitoring Calibration Dilution Standards – these solutions are used for the manual injections required to prepare the initial calibration.

10.4.1. Internal Standard Solution: 80ul of stock standard IS solution (2500ug/ml) is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.4.2. System Monitoring Compound Solution: 80ul of stock standard Surrogate solution (2500ug/ml) is added to approximately 1 ml of purge and trap methanol in a 2 ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol for a final concentration of 100ng/ml.

10.4.3. To calculate appropriate expiration dates, refer to "Standards Traceability and Preparation Logbooks", SOP No. AGP-STD-14.

10.5. Working Standards

10.5.1. Intermediate Calibration Solution: (Three individual mixtures)

10.5.1.1. 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.5.1.2. 500ul of stock standard 54 Component Mix solution (2000ug/ml) is added to approximately 9ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.5.1.3. 1000ul of each of the four stock standard 8260+ Mixes are added to approximately 9ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade

methanol.

10.5.2. Matrix Spike Solution: 100ul of stock standard 5 component solution (2500ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5 ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

10.5.3. A Full List Matrix Spike standard is made from stock Calibration Verification Standards and is composed of two mixes.

10.5.3.1. 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.5.3.2. 200ul of each of the two stock standard 8260+ Second Source Mixes are added to approximately 1ml of purge and trap methanol in a 2ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol.

10.5.4. Working Internal Standard and System Monitoring Compound Solutions – for autoinjection by instrument.

10.5.4.1. Working Internal Standard Solution: An Internal Standard Mixture is made from IS stock standard (2500ug/ml) at 140ng/ul to 175ng/ul, depending on sample loop size, for the Dynatek autosampler and is made at 220ng/ul to 280ng/ul, depending on sample loop size, for the Varian Archon autosampler.

10.5.4.2. Working System Monitoring Calibration Solution: A System Monitoring Compounds Mixture is made from Surrogate stock standard (2500ug/ml) at 140 ng/ul to 175 ng/ul depending on sample loop size, for the Dynatek autosampler and is made at 220ng/ul to 280ng/ul, , depending on sample loop size, for the Varian Archon autosampler.

10.5.5. Tuning Mixture: 4ul of stock solution 4-Bomofluorobenzene (BFB) tuning mixture is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

10.5.6. Working Initial Calibration Standards

10.5.6.1. Water: 25 ml

10.5.6.1.1. 20ul, 10ul and 5ul each of Intermediate Calibration Solution (10.5.1) is added to reagent water in each of three 50ml volumetric flasks. 20ul, 10ul and 5ul each of the Secondary IS Calibration Dilution Solution and also the Secondary System Monitoring Calibration Dilution Solution are added to the respective 50 ml volumetric flasks. The flasks are brought to volume with

reagent water to prepare the 40, 20 and 10 ug/L standards respectively.

10.5.6.1.2. 4ul and 1ul each of Intermediate Calibration Solution ((10.5.1) plus 4ul and 1ul each of the Secondary IS Calibration Dilution Solution and also the Secondary System Monitoring Calibration Dilution Solution are added to reagent water in 100 ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 4f and 1 ug/L standards respectively.

10.5.6.1.3. Each standard is then transferred into a 40ml vial and loaded onto the Dynatech or Archon autosampler.

10.5.6.2. Water: 5 ml

10.5.6.2.1. 100ul, 50ul, 25ul, 10ul and 2.5ul each of Intermediate Calibration Solution (10.5.1) plus the same amounts of Secondary IS Calibration Soluti9on and Secondary System Monitoring Calibration Solution are (100ng/ml) are added to five individual 50ml volumetric flasks continuing reagent water to prepare 200, 100, 50, 20 and 5 ug/L standards respectively.

10.5.6.2.2. Each standard is then transferred into a 40ml vial and loaded onto the Dynatech or Archon autosampler.

10.5.6.3. Soil:

10.5.6.3.1. 100ul, 50ul, 25ul, 10ul and 2.5ul of Intgermediate Calibration Solution (10.5.1) plus the same amounts of Secondary IS Calibrraiton Solution and Secondary System Monitoring Calibration Solution (at 100ng/ml) are added to five individual 50ml volumetrics containing reagent water. The final concentration of each standard is 200, 100, 50, 20 and 5 ug/kg, respectively.

10.5.6.3.2. 5 ml of each standard is then transferred into five individual 40ml vials and loaded onto the Archon autosampler.

10.5.7. Continuing Calibration Standard

10.5.7.1. Water: 25 ml

10.5.7.1.1. 5ul of stock target compound mix is added to 50ml of DI water to make a final concentration of 10ppb. Pour standard into 40ml vial; working standard internal standard and system monitoring compounds are added by Dynatech or Archon autosampler.

10.5.7.2. Water: 5 ml

10.5.7.2.1. 25ul of stock target compound mix is added to 50ml of DI water to make a final concentration of 50ppb. Pour standard into 40ml vial; working standard internal standard and system monitoring compounds are added by Dynatech or Archon autosampler.

10.5.7.3. Soil:

10.5.7.3.1. 25ul of stock target compound is added to 50ml of DI water to make a final concentration of 50ppb.. Take 5ml and transfer it into a 40ml; working

standard internal standard and system monitoring compounds are added by Archon autosampler.

10.6. Storage of standards

10.6.1. Stock and secondary dilution standards are stored in teflon-sealed crimp cap vials at -10^0 C to -20^0 C.

10.6.2. Aqueous standards are stored in teflon-sealed crimp cap bottles at 4^0 C plus or minus 2^o C.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1. Samples are collected in 40 mL vials with caps and septa, preserved to a pH < 2 with Hydrochloric Acid and stored at 4±2 degrees C until time of analysis.

11.2. Holding time for unpreserved samples is 7 days from sample date. For preserved samples the holding time is 14 days from sample date.

11.3. For some clients, regulatory agencies or QAPPS, the specified holding times may be different than those described in 11.2. In those cases, consult the specific Protocol/Method/QAPP or Project Manager for holding time details.

11.4. Sample Storage

11.4.1. Volatile samples are stored at 4±2°C from the time of collection until analysis.

11.4.2. Volatile samples are stored together in refrigerators specifically designated for volatiles only.

11.4.3. Storage blanks are stored with samples until analysis.

11.4.4. Samples and extracts are stored separately.

11.4.5. Volatile samples and standards are stored separately.

11.5. Preparation Of MS/MSD Samples

11.5.1. Water Samples: 40ml vial is spiked with 8ul of 50ng/ul matrix spike standard for 25ml purge and 40ul for the 5ml purge. This corresponds to a final concentration in the samples of 10 ug/L and 50 ug/L respectively. Analysis proceeds according to procedures described for water analysis.

11.5.2. Low Level Soil/Sediment Samples: 5ul of matrix spiking solution is added to a 5g aliquot of sample. This corresponds to a final concentration in the samples of 50 ug/kg. Analysis proceeds according to procedures described for low-level soil/sediment samples.

11.5.3. Medium Level Soil/Sediment Samples: 1ml of methanol containing the soil extraction is combined with 50 mls of water and 50 ul of spiking solution is added to the water methanol extraction solution. Sample analysis proceeds according to procedures described for medium level soil/sediment samples.

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12.0 QUALITY CONTROL

12.1. Blank Analysis:

12.1.1. Method Blank: A method blank consisting of a clean reference matrix (reagent water or purified quartz sand) must be analyzed prior to the analysis of samples but following any standard analysis.

12.1.1.1. Target compounds detected in a method blank must fall below the reporting limit, unless specified in client QAPP.

12.1.1.2. If internal standard or systems monitoring compound recoveries are not met, the method blank must be reanalyzed before the analysis of samples.

12.1.2. Storage (Holding) Blank: A weekly holding blank is analyzed to determine if cross contamination occurs within the volatile holding area. The results are reviewed by the quality assurance department and deemed acceptable or not acceptable. Corrective action, if necessary, will be taken.

12.1.3. Instrument Blank: An instrument blank consisting of a clean reference matrix analyzed after the analysis of samples containing target compounds which exceed the calibration range. Multiple instrument blanks are shot until the instrument blank meets the criteria for method blanks.

12.2. Matrix Spike Blank (MSB/LCS): An aliquot of clean reference material spiked with the matrix spiking solution is analyzed with each analytical batch. The standard from which the MSB/LCS is prepared is purchased from an alternate vender from the continuing (CCV) standard. The solution is spiked at a concentration of 10ug/L for 25ml analysis and 50ug/L for 5ml analysis. A matrix spike blank duplicate is performed when insufficient volume is available for sample specific MS/MSD quality control.

12.2.1. The MSB/LCS must fall within internally derived statistrical control limits or where applicable the limits specified by a project QAPP.

12.2.2. Routine compounds included in the MSB/LCS are:

1,1-Dichloroethene
Chlorobenzene
Toluene
Benzene

12.2.3. When required, the MSB/LCS a 'full-compound' spike will be prepared and the MSB/LCS will be spiked with all compounds of interest. Due to the potentially large number of target compounds for method 8260B, it is possible that a couple of spiking compound could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the

case narrative and the data will be found to be acceptable.

12.2.4. If the results of sample matrix spikes fall outside of the quality control range due to matrix, the MSB is used to verify that the laboratory can perform a spike on a clean matrix.

12.3. Matrix Spike And Matrix Spike Duplicate Analysis: A matrix spike and matrix spike duplicate consisting of an actual field sample which has been spiked with the matrix spiking solution.

12.3.1. Matrix spike and matrix spike duplicate analysis will not be performed on rinsates or field/trip blanks.

12.3.2. If a sample has not been designated for MS/MSD analysis by the client, a sample will be selected at the analyst's discretion. MS/MSD analysis will be performed at a minimum of every 20 samples.

12.3.3. If insufficient sample was received for a designated MS/MSD the client will be contacted with the laboratory's in-house designated sample for MS/MSD analysis. If no MS/MSD is required, the instance will be documented in the SDG narrative.

12.3.4. If medium level analysis is required on the client designated sample, the laboratory analyst will choose a low level sample on which to perform the quality control analysis. Medium level QC will also be performed.

13.0 CALIBRATION AND STANDARDIZATION

13.1. Instrument Tuning and Performance Check:

13.1.1. The GC/MS system is calibrated using Perflurotributylamine (PFTBA) according to the recommended tuning conditions suggested by the vendor.

13.1.2. An instrument performance check of Bromofluorobenzene (BFB) is analyzed at the beginning of each 12-hour analysis period.

13.1.2.1. The analysis of the instrument performance check is performed using the following procedure:

13.1.2.1.1. 1ul of a 50ng/ul solution is directly injected, resulting in a 50ng injection of BFB into the GC/MS.

13.1.2.1.2. A blank containing 50 ng BFB is purged.

13.1.2.2. The mass spectrum of BFB is acquired using the following procedure:

13.1.2.2.1. A single scan on the peak.

13.1.2.2.2. An average of the peak.

13.1.2.2.3. The apex scan, one scan immediately preceding the apex and one scan immediately following the apex are averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB.

13.1.2.3. The mass spectrum of BFB must pass the technical acceptance criteria given in

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Table 2.

13.2. Initial Calibration (ICAL):

13.2.1. The instrument performance check must meet the technical acceptance criteria prior to the analysis of an initial curve or samples. The GC/MS system is calibrated using five levels of concentrations. All compounds of interest are included. (See section 17.0 for initial calibration acceptance criteria.)

13.2.2. Solutions containing target compounds and system monitoring compounds are analyzed at concentrations of 5, 20, 50, 100 and 200 ug/L. (1, 4, 10, 20 and 40 ug/L for 25 mL)

5 ml (aqueous) or 5 gram (soil) Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (ml)	Final Conc. Water (ug/L)	Final Conc. Soil (ug/kg)
VSTD005	MeOH	100ng/ul	5	100	5	5
VSTD020	MeOH	100ng/ul	10	50	20	20
VSTD050	MeOH	100ng/ul	25	50	50	50
VSTD100	MeOH	100ng/ul	50	50	100	100
VSTD200	MeOH	100ng/ul	100	50	200	200

25 ml Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (ml)	Final Conc Water (ug/L)
VSTD001	MeOH	100ng/ul	1	100	1
VSTD004	MeOH	100ng/ul	4	100	4
VSTD010	MeOH	100ng/ul	5	50	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD040	MeOH	100ng/ul	20	50	40

13.3. Continuing Calibration Verification (CCV):

13.3.1. Every 12 hours of sample analysis the laboratory must demonstrate that the instrument has drifted or changed minimally by performing an instrument performance check and continuing calibration verification. (See section 17.0 for continuing calibration acceptance criteria.)

14.0 PROCEDURE

14.1. Once initial calibration criteria has been met, and prior to analyzing samples and required blanks, Each GC/MS system must be routinely checked by analyzing a Continuing Calibration

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Verification (CCV) standard containing all compounds (including internal standards and system monitoring compounds) at a concentration of 50ug/L for 5ml or 10ug/L for 25ml analysis.

14.1.1. If time remains after initial calibration criteria have been met, it may not be necessary to perform a CCV. The 50 ug/L (10ug/L for 25ml) standard may be evaluated against the new initial curve and used as the CCV.

14.1.2. If there is no time remaining in the 12-hour period, the instrument performance check (BFB) must be analyzed along with a new CCV.

14.1.3. Procedure for Continuing Calibration: 25uls of internal standards, system monitoring compounds, and target compound mixture is added to a 50ml volumetric flask. A 5ml aliquot is analyzed. 25ml purge analysis requires 5ul into a 50 ml volumetric flask filled with volatile free water.

14.2. Sample Analysis

14.2.1. BFB tuning criteria and GC/MS calibration verification must be met before sample analysis begins.

14.2.2. The acquisition time of the BFB tune establishes a 12hr. batch. The CCV, MSB, and VBLK must be analyzed within 12hrs, unless specified by the client request. The remaining time in the 12hr batch is utilized to run samples of similar matrix. All aqueous samples are considered a water matrix. All solid samples, with the exception of sludges, are considered soil matrix. Sludges are run medium level.

14.2.3. Samples and standard solutions are brought to ambient temperature before analysis.

14.2.4. Prior to the analysis of samples, a method blank must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for method blanks must be met prior to sample analysis.

14.3. Water Samples (See also SOP No. AMV-5030-42)

14.3.1. A 5ml sample aliquot is spiked with internal and system monitoring compounds to a final concentration of 50 ug/L each. 25ml analysis requires a final concentration of 10ug/L. The spike may be performed manually with a Hamilton gas tight syringe or the Dynatech/Archon auto sampler may be used. The sample is then loaded onto the auto sampler where it is in turn transferred to the purge chamber.

14.3.2. The sample is purged for 11.0 ± 1 minute at ambient temperature.

14.3.3. At the end of the purge time, the sample is desorbed onto the gas chromatograph column

by rapidly heating the trap to 250^{0}C while the trap is back flushed with Helium between 20 - 60 ml/minute for two minutes. The sample is desorbed onto the column and the gas chromatograph temperature ramping program is commenced.

14.3.4. While the trap is in the bake mode, the purge chamber is flushed with two 5ml aliquots of reagent water in order to avoid possible contamination from carryover of target compounds.

14.3.5. After the sample has desorbed, the trap is conditioned at 260^{0}C for 8 minutes. After baking, the trap is ready for the next sample.

14.3.6. Dilutions may be necessary if the concentration of any target compound exceeds the working range of the calibration.

14.3.7. In the event that a dilution is required, a measured volume of sample is added to a volumetric flask then to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and a 5ml volume is taken for analysis. Analysis may then proceed as described.

14.4. Low Level Soil/Sediment Samples (See also SOP No. AMV-5035-43)

14.4.1. The low level soil method is based on a heated purge of a 5g sample mixed with reagent water containing a final concentration of 50 ug/L of internal and system monitoring compounds.

14.4.2. If a dilution of the soil/sediment is required, a smaller portion of soil may be used. The smallest amount of soil that may be used is 1g. If a higher dilution is required, the sample must be analyzed as a medium level soil/sediment.

14.4.3. Initial and continuing calibrations that are used for the quantitation of low soils/sediments are analyzed using the same purge and trap conditions as samples.

14.4.4. The sample consists of the entire contents of the sample container. The contents are mixed thoroughly with a narrow metal spatula. A 5g portion is taken for analysis. The weight is recorded to the nearest 0.01g.

14.4.5. A 5ml aliquot of reagent water containing internal standards and system monitoring compounds is added to the sample immediately prior to heating and purging.

14.4.6. After reagent water is added, the soil/sediment sample is heated to 40^{0}C $\pm$ 1^{0}C then purged for 11 $\pm$ 1 minutes.

14.4.7. After purging, the sample is subjected to desorbing as described for water analysis.

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14.5. Medium Level Soil/Sediment Samples

14.5.1. The medium level soil/sediment methods is based on an extraction of a portion of the sample with methanol. A portion of the extract is then added to a 5 ml aliquot of reagent water containing internal and system monitoring compounds at a final concentration of 50ug/L.

14.5.2. The sample consists of the entire contents of the sample container. The contents are thoroughly mixed with a thin metal spatula. A 4 g portion of the sample is weighed into a 20ml vial. The weight of the sample is recorded to the nearest 0.01g.

14.5.3. A 10ml aliquot of methanol is quickly added to the sample. The vial is capped and the sample is shaken for 2 minutes.

14.5.4. A determined amount of methanol extract is added to a 5ml aliquot of reagent water containing internal and system monitoring compounds at a final concentration of 50ug/L. Analysis may proceed according to procedures described for water samples.

14.5.5. Table 3 may be used to determine the volume of methanol extract required for a given dilution factor.

14.6. pH Determinations For Water Samples

14.6.1. After the sample aliquots are taken from the VOA vials, the pH of the sample is determined by placing several drops of sample, using a disposable pipet, onto pH paper. A checkmark will be entered in the injection logbook if the sample pH is <2, however if the sample demonstrates a pH>2, the actual pH will be noted in the injection logbook..

14.7. Percent Moisture Determinations

14.7.1. Immediately after weighing the sample for analysis, a 5-10g portion is weighed into a tared aluminum weigh pan. The sample is then dried overnight at 105^{0}C. The sample is allowed to cool. The final weight is recorded. Using equation 4, the percentage moisture, which is used for reporting concentrations relative to the dry weight of the soil/sediment samples, may be determined. The following calculation is used to determine percent moisture:

$$\%\text{moisture} = \frac{\text{Wt. of sample (g)} - \text{wt. of dried sample (g)}}{\text{wt. of sample (g)}} \times 100$$

15.0 CALCULATIONS

15.1. Calculations For MS/MSD Samples

15.1.1. The calculations to determine concentrations are the same equations described for

sample analysis of a given matrix.

15.1.2. The percent recovery of the matrix spiking compounds is determined using equation:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,
SSR = Spiked sample result
SR = Sample results
SA = Spike added

15.1.3. The relative percent difference (RPD) of the recoveries of each compound between the matrix spike and matrix spike duplicate is determined using equation:

$$RPD = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)} \times 100$$

Where,
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

15.2. Calculations For Initial Calibration

15.2.1. The relative response factor (RRF) for each target compound and each system monitoring compound is calculated using equation.

$$RRF = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

Where,
Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 6)
Ais = Area of the characteristic ion (EICP for the specific internal standard (see Tables 5 and 6A)
Cis = Concentration of the internal standard
Cx = Concentration of the compound to be measured

15.2.2. The relative response factor of the Xylenes requires the use of the area response and the concentration of the peak that represents the single isomer.

15.2.3. The relative response factor of 1,2-dichloroethene is calculated using the sum of the

areas of both isomers and the sum of the concentrations.

15.2.4. The average response factor (RRF) is calculated for all compounds of interest.

15.2.5. The relative standard deviation (% RSD) is calculated over the working range of the curve for all compounds using equation:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^{n}(xi - \bar{x})2}{n-1}}$$

Where,

Xi = each individual value used to calculate the mean

X = the mean of n values

n = the total number of values

15.3. Calculations For Continuing Calibration

15.3.1. The relative response factor (RRF) for all target compounds and system monitoring compounds is calculated using equation 1.

15.3.2. The percent difference between the initial calibration and the continuing calibration is determined for all target compounds and system monitoring compound using equation:

$$\%Difference = \frac{RRFc - \overline{RRFi}}{\overline{RRFi}} \times 100$$

Where,

RRFc = Relative response factor from continuing calibration standard

$\overline{RRFi}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

15.4. Percent Moisture Determinations

15.4.1. Immediately after weighing the sample for analysis, a 5-10g portion is weighed into a tared aluminum weigh pan. The sample is then dried overnight at 105^{0}C. The sample is allowed to cool. The final weight is recorded. Using the equation for % moisture,

concentrations relative to the dry weight of the soil/sediment samples, may be determined.

$$\%\text{moisture} = \frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

15.5. Quantitation of volatile target compounds is done using the internal standard method. The internal standards used for each compound are assigned those indicated in table 5. The Internal Standard RRF of the continuing calibration is used in the quantitation calculation.

15.5.1. Water Samples: The following equation is used to calculate water samples:

$$\text{Concentration ug/L} = \frac{(Ax)(Is)(DF)}{(Ais)(RRF)(Vo)}$$

Where,

Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 2)

Ais = Area of the characteristic ion (EICP) for the specific internal standard (see Tables 5 and 6A)

Is = Amount of internal standard added in nanograms (ng)

RRF= Relative response factor from the ambient temperature purge of the calibration standard.

Vo = Volume of water purged in milliliters (mL)

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., Vo above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5 mL with reagent water and purged, Df = 5 mL/2.0 mL = 2.5. If no dilution is performed, Df = 1.

15.5.1 Low Level Soil/Sediment Samples

15.5.1.1The following equation is used for low level soil/sediment samples:

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(Ax)(Is)}{(Ais)(RRF)(Ws)(D)}$$

Where,

Ax, Is, Ais are as given for water.

RRF = Relative response factor form the heated purge of the calibration standard.

$$D = \frac{100 - \%\text{ moisture}}{100}$$

Ws = Weight of sample added to the purge tube, in grams (g).

15.5.2 Medium Level Soil/Sediment Samples

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15.5.2.1The following equation is used for quantitation of medium level soil/sediment samples:

$$\text{Concentration ug/Kg (Dry weight basis)} = \frac{(Ax)\,(Is)\,(Vt)\,(1000)\,(Df)}{(Ais)\,(RRF)\,(Va)\,(Ws)\,(D)}$$

Where,
Ax, Is, Ais are as given for water.
RRF = Relative response factor from the ambient temperature purge of the calibration standard.
Vt = Total volume of the methanol extract in milliliters (mL).
NOTE: This volume is typically 10 mL, even though only 1 mL is transferred to the vial.
Va = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 uL) in microliters (uL) added to reagent water for purging.
Ws = Weight of soil/sediment extracted, in grams (g).
$D = \frac{100 - \%\ \text{moisture}}{100}$
Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

(The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the sample methanol extract (Vt). The factor of 1,000 in the numerator converts the value of Vt from mL to uL.)

15.6 When quantitating the sample concentration of Xylenes (total), the areas of both the M&P Xylene peak and the O-Xylene peak are summed and the RRF determined using equation 1 are used. The concentration of each peak may be determined separately and then summed to determine the concentration of Xylene (total).

15.7 When quantitating the concentration of 1,2-Dichloroethene (total), the concentrations of the two isomers (cis and trans) are summed.

15.8 Secondary ion quantitation may be used if interferences (such as matrix effects) may cause a bias in quantitation. If ions other then those listed in table 6 are used, the analyst will document the reason, and it will be noted in the job narrative.

15.9 If manual integration of any compound (including internal standards, system monitoring compounds, target or tentatively identified compounds) is required, the EICP of that compound will be provided. All manual integrations will be identified with an "m" and

initialed and dated by the GC/MS analyst.

15.10 Tentatively Identified Compounds

15.10.1 An estimated concentration for tentatively identified compounds will be determined using the equations described above for a given matrix using the total area counts of both the tentatively identified compound and the nearest internal standard which is free of interferences.

15.10.2 The RRF used to determine all concentrations of tentatively identified compounds will be an assumed RRF of one (1).

15.10.3 All tentatively identified compounds will be qualified as "J" (estimated) and "N" (presumptive evidence).

15.11 System Monitoring Compounds

15.11.1 The recovery of all system monitoring compounds in samples, blanks matrix spikes and matrix spike duplicates, is calculated using equation:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

15.11.2 The recovery limits for each system monitoring compound are laboratory established on an annual basis. The recoveries must be within the criteria limits. If they fall outside criteria limits, the results must be evaluated and the sample reanalyzed, if necessary.

15.11.3 The relative retention time (RRT) of each system monitoring compound must be within the acceptance windows of ±0.06 RRT.

15.12 Internal Standards

15.12.1 The internal standards of all samples, blanks, matrix spikes and matrix spike duplicates must be monitored. The EICP area of each internal standard must be within the range of -50.0 percent to 200.0 percent of those in the continuing calibration.

15.12.2 The relative retention time (RRT) of each internal standard must be within 0.5 minutes (30 seconds) of those in the continuing calibration.

15.13 Verification of Calculated Result

15.13.1 The laboratory analyst/data entry analyst will print out and review sample worksheets and hand calculate the result for positive hits, internal standards and surrogates for comparison to the AIMS calculated result. Corrective action will result, if needed.

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16.0 METHOD PERFORMANCE

16.1. Each analyst prior to sample analysis will perform 4 replicate second source QC check standards, at 20ug/L, as an Initial Demonstration of Capability. The average recovery and standard deviation are keep in AIMS and kept with each analyst's training file.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1. Technical Acceptance Criteria For Initial Calibration

17.1.1. SPCCs - System performance check compounds are compounds used to check compound instability degradation. The following avaerage minimum average response factors must be met before the cuarbve can be used.:

17.1.1.1.	Chloromethane	0.10
17.1.1.2.	1,1-Dichloroethane	0.10
17.1.1.3.	Bromoform	0.10
17.1.1.4.	Chlorobenzene	0.30
17.1.1.5.	1,1,2,2-Tetrachloroethane	0.30

17.1.2. CCCs - Calibration Check Compounds - Evaluates the calibration based on the integrity of the system. The % RSD for the CCCs MUST be equal or less than 30%. The CCCs are:

17.1.2.1. Vinyl chloride
17.1.2.2. 1,1-Dichloroethene
17.1.2.3. Chloroform
17.1.2.4. 1,2-Dichloropropane
17.1.2.5. Toluene
17.1.2.6. Ethylbenzene

17.1.3. If the % RSD of any of the target analytes is 15% or less, the average response factor is assumed constant and the average response factor may be used for quantitation.

OR

17.1.4. If the mean of the RSD values for all analytes is less than or equal to 15%, then the average response factor may be used for quantitation.

OR

17.1.5. If the % RSD of a target analyte is greater than 15%, linear regression may be used providing the coefficient of determination is greater than or equal to 0.99.

17.2. Technical Acceptance Criteria For Continuing Calibration

17.2.1. SPCCs - A system performance check is made daily or during every 12 hour analytical shift. Each compound must meet its minimum response factor (see Initial Calibration Criteria).

17.2.2. CCCs - Used to check the validity of the initial calibration. The % Difference for each CCC shall be less than or equal to 20% from the initial calibration for the continuing calibration to be valid. All non-CCC target compounds must be less than 100% difference.

17.2.3. Internal Standard Retention Time – The retention times for all internal standards must be evaluated to make sure that they are no more than 30 seconds from that of the midpoint of the initial calibration. If the retention time shift is greater than 30 seconds, the system must be inspected for malfunctions and maintenance must be performed, as required.

17.2.4. Internal Standard Response – The EICP area for all internal standards must be evaluated to make sure that they have not change by a factor greater than two (-50% to +100%) from that of the midpoint of the initial calibration. If the response exceeds these limits, the system must be inspected for malfunctions and maintenance must be performed, as required.

17.3. Technical Acceptance Criteria of Quality Control Samples

17.3.1. Samples, blanks, matrix spikes, and matrix spike duplicates must meet internal standard and system monitoring compound recovery limits. Where the Internal Standard recovery limit equals sample internal standard characteristic ion area (EICP) divided by the CCV internal standard characteristic ion area (EICP) , multiplied by 100.

18.0 18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA.

18.1. Corrective Actions For MS/MSD

18.1.1 If the recoveries of the internal standards and system monitoring compounds do not agree with the unspiked sample (i.e. the sample recoveries were within control limits and MS/MSD recoveries were outside of control limits) the MS/MSD must be reanalyzed.

18.1.2 If the recoveries of the internal standards and system monitoring compounds agree with the unspiked sample (i.e. both the sample and MS/MSD recoveries were outside of control limits) re-analysis is not required. The instance will be documented in the SDG narrative.

18.1.3 Limits for the matrix spiking compounds are established by the laboratory on an annual basis. If the concentrations determined in the MS/MSD do not meet the control limits, no corrective action is necessary as long as the MSB/LCS was within control limits. The instance will be documented in the job narrative.

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18.2 Corrective Actions For Initial Calibration

18.2.1 If technical acceptance criteria cannot be met, it may be necessary to re-analyze the initial calibration. If after re-analysis, the criteria has not been met, it may be necessary to inspect the GC/MS system for possible problems.

18.2.2 Corrective actions may require one or several of the following procedures:

18.2.2.1Open new/remake standard mixes
18.2.2.2The ion source may be cleaned
18.2.2.3The column may be cut at the injection port end
18.2.2.4Change the purge trap on the purge and trap unit
18.2.2.5Correct purge gas flow to optimize response
18.2.2.6The column may be baked out
18.2.2.7The purge trap may be baked out
18.2.2.8The column may be replaced

18.3 Corrective Actions for Failure to Meet the Continuing Calibration Acceptance Criteria

18.3.1 If the technical acceptance criteria given above are not met, it may be necessary to re-analyze the continuing calibration check. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.

18.3.2 Other Corrective actions may be taken. The following details possible corrective actions:

18.3.2.1Open new/remake standard mixes
18.3.2.2The ion source may be cleaned
18.3.2.3The column may be cut at the injection port end
18.3.2.4The trap on the purge and trap unit may be replaced
18.3.2.5The purge gas flow may be adjusted
18.3.2.6The column may be baked out
18.3.2.7The purge trap may be baked out
18.3.2.8The column may be replaced

18.4 Corrective Actions For Samples

18.4.1 If the internal standard or system monitoring criteria are not met, the sample must be re-analyzed to insure that it was not an internal problem that affected recoveries. If, after re-analysis, recoveries are outside of control limits, a matrix effect can be assumed.

18.4.2 When dilutions are performed, target compound concentration must fall within the upper range of the initial calibration. If any target compound exceeds the calibration range, the

sample would require dilution. The sample immediately following a sample with target compounds above the calibration range must be monitored to insure that there is no carryover present. If there is a possibility of carryover, that sample must be re-analyzed.

18.4.3 If matrix effects exist, and both analyses exhibit recoveries outside of control limits, both analyses will be reported and documented in the job narrative.

18.4.4 If, after re-analysis, recovery criteria are met, only the second analyses will be reported. If the second analyses occurs outside of the contract required holding time, both analyses will be reported in that instance.

18.4.5 In the case of a matrix spike or matrix spike duplicate, these samples should only be reanalyzed if the recoveries do not agree with the unspiked sample. If recoveries agree, the unspiked sample will not require re-analysis. The instance will be documented in the SDG narrative.

18.5 Corrective Actions for Failure to Meet the Matrix Spike Blank Acceptance Criteria

18.5.1 Limits for the matrix spiking compounds are established by the laboratory on an annual basis. The MSB/LCS must fall within these control limits. When required, the MSB/LCS will be spiked with all compounds of interest, otherwise spiked to include a minimum of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Due to the potentially large number of target compounds for method 8260B, it is possible that a couple of spiking compounds could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable

18.5.2 If the technical acceptance criteria are not met, it may be necessary to re-analyze the matrix spike blank. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.

18.5.3 Other Corrective actions may be taken. The following details possible corrective actions:

18.5.3.1Open new/remake standard mixes
18.5.3.2The ion source may be cleaned
18.5.3.3The column may be cut at the injection port end
18.5.3.4The trap on the purge and trap unit may be replaced
18.5.3.5The purge gas flow may be adjusted
18.5.3.6The column may be baked out
18.5.3.7The purge trap may be baked out
18.5.3.8The column may be replaced

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19.0 CONTINGENCIES FOR HANDELING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1. Inform project manager for client input and fill out job exception report.

19.2. Rerun samples to confirm results.

19.3. Resample if client or project manager requests.

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for " Waste Management and Pollution Prevention."

20.2 Waste Streams Produced by the Method

20.2.1 The following waste streams are produced when this method is carried out.

20.2.2 Spill Response: Any spills must be cleaned up immediately and handled correctly. Any wastes that have a pH $<$ 7 must be disposed of in an "A" waste container. Any wastes having a pH $>$ 7 must be disposed of in a "D" waste container.

20.2.3 Aqueous waste generated from analysis: Any wastes that have a pH $<$ 7 must be disposed of in an "A" waste container. Any wastes having a pH $>$ 7 must be disposed of in a "D" waste container.

20.2.4 Solvent waste generated from analysis: Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste. Waste receptacles are then taken to sample control where they are then properly disposed of.

20.2.5 Solid waste generated from analysis: Solid volatile analysis waste consists of soils and glass. The soil is wrapped in tin foil and placed in the solid waste receptacle. Soils used for dry weight measurements are also disposed of in this manner. Glass waste such as pipets and vials are rinsed and disposed of in approved glass receptacles

20.2.6 Expired Standards. Expired and used standards are stored in a laboratory approved metal waste receptacle labeled "BV". Waste receptacles are then taken to sample control where they are then properly disposed of.

21.0 REFERENCE

21.1. Method 8260B, "Test Methods for Evaluating Solid Waste"; SW846, Third Edition, December

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22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Compounds Determined by Method 8260B
22.2. BFB Key Ions and Ion Abundance Criteria
22.3. Volume of Medium Level Extracts for Dilution
22.4. Characteristic Masses (m/z) for Purgeable Organic Compounds
22.5. GCMS Volatile Job Summary and Data Review Checklist
22.6. Tables 6-12: Composition of Stock Standards

23.0 CHANGES FROM THE PREVIOUS REVISION

23.1. Laboratory Director change, signature added
23.2. Department Supervisor change, signature added.
23.3. Section 3.0: Changed section to Reporting Limit instead of Method Detection Limit and added appropriate information.
23.4. Section 14.7.1: Incorporated Interim Change IC-A
23.5. Updated Tables 6 through 10
23.6. Added Tables 11 & 12
23.7. Minor grammatical changes
23.8. Changed 'should' to 'shall' or 'must' as needed.

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TABLE 1: COMPOUNDS DETERMINED BY METHOD 8260B

		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-03-3	pp	nd	nd	nd	nd	c

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		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	l	nd	nd	nd	nd	pc
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (I.S.)	540-36-3	nd	nd	nd	nd	c	c

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		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	l	nd	nd	nd	nd	c
Ethanol	64-17-5	l	c	c	nd	nd	c
Ethyl acetate	141-78-6	l	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	l	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	l	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	l	nd	nd	nd	c
Methanol	67-56-1	l	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	l	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

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		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	l	nd	nd	nd	c
B-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	c
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	l	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-33	c	nd	c	c	c	c
Toluene-d8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluene	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethane	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
Xylene (Total)	1330-20-7	c	nd	c	c	c	c

c= Adequate response by this technique
b= Chemical Abstract Services Registry Number
pp= Poor purging efficiency resulting in high EQLs
l= Inappropriate technique for this analyte
pc= Poor chromatographic behavior
nd= Not determined
surr= Surrogate
IS= Internal Standard
ht= Method analyte only when purged at 80 C

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The following compounds are also amenable to analysis by Method 8260:

Bromombenzene
n-Butylbenzene
sec-Butlybenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
Cis-1,2-Dichloroethene
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

TABLE 2
BFB Key Ions and Ion Abundance Criteria

mz	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to9% of m/z 176

*Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 3

Volume of Medium Level Extracts for Dilution

Dilution Factor	Volume of Extract
1	100ul
2	50ul
5	20ul
10	10ul
20	5ul
25	4ul
40	2.5ul
50	2ul
100	1ul
200	50ul of a 1/10 Dilution

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TABLE 4

Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40,39
Acrolein	56	55,58
Acrylonitrile	53	52,51
Allyl alcohol	57	58,39
Allyl chloride	76	41,39,78
Benzene	78	-
Benzyl chloride	91	126,65,128
Bromoacetone	136	43,138,93,95
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44,84,86,111
Chloroacetonitrile	48	75
Chlorobenzene	112	77,114
1-Chlorobutane	56	49

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Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Chlorodibromomethane	129	208,206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44,43,51,80
bis-(2-Chloroethyl) sulfide	109	111,158,160
2-Chloroethyl vinyl ether	63	65,106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88,90,51
3-Chloropropionitrile	54	49,89,91
3-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,2-Dichlorobenzene-d_4	152	115,150
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
cis-1,4-Dichloro-2-butene	75	53,77,124,89
trans-1,4-Dichloro-2-butene	53	88,75
Dichlorodifluoromethane	85	87
1,1-Dichlorothane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichlorothene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78

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Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43,81,49
1,1-Dichloropropene	75	110,77
cis-1,3-Dichloropropene	75	77,39
trans-1,3-Dichloropropene	75	77,39
1,2,3,4-Diepoxybutane	55	57,56
Diethyl ether	74	45,59
1,4-Dioxane	88	58,43,57
Epichlorohydrin	57	49,62,51
Ehtanol	31	45,27,46
Ethyl acetate	88	43,45,61
Ethylbenzene	91	106
Ethylene oxide	44	43,42
Ehtyl methacrylate	69	41,99,86,114
Hexachlorobutadiene	225	223,227
Hexachloroethane	201	166,199,203
2-Hexanone	43	58,57,100
2-Hydroxypropionitrile	44	43,42,53
Iodomethane	142	127,141
Isobutyl alcohol	43	41,42,74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134,91
Malonitrile	66	39,65,38
Methacrylonitrile	41	67,39,52,66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86,49
Methyl ethyl ketone	72	43
Methyl iodide	142	127,141

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Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41,100,39
4-Methyl-2-pentanone	100	43,58,85
Naphthalene	128	-
Nitrobenzene	123	51,77
2-Nitropropane	46	-
2-Picoline	93	66,92,78
Pentachloroethane	167	130,132,165,169
Propargyl alcohol	55	39,38,53
B-Propiolactone	42	43,44
Propionitrile (ethyl cyanide)	54	52,55,40
n-Propylamine	59	41,39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182,145
1,2,4-Trichlorobenzene	180	182,145
1,1,1,2-Tetrachloroethane	131	133,119
1,1,2,2-Tetrachloroethane	83	131,85
Tetrachloroethene	164	129,131,166
Toluene	92	91
1,1,1-Trichloroethane	97	99,61
1,1,2-Trichloroethane	83	97,85
Trichloroethene	95	97,130,132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64

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Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
INTERNAL STANDARDS/SURROGATES		
Benzene-d6	84	83
Bromobenzene-d5	82	162
Bromochloromethane-d2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115,150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174,176
Chloroform-d1	84	
Dibromofluoromethane	113	

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TABLE 5

Job Summary Check List (Page 1 & 2)

GCMS VOLATILE JOB SUMMARY

JOB No._____ METHOD_____
SOIL LEVEL_____
LAB DUE DATE_____ INSTRUMENT_____
PURGE VOLUME_____

TUNE

	FILE#_____	FILE#_____	FILE#_____
	PASSED? Y N	PASSED? Y N	PASSED? Y N

CONTINUING CALIBRATION VALUES CALCULATED FROM CCV____ INIT____

	FILE#_____	FILE#_____	FILE#_____
	PTS OUT?_____	PTS OUT_____	PTS OUT_____
	ACCEPTABLE Y N	ACCEPTABLE Y N	ACCEPTABLE Y N

INITIAL CALIBRATION

	REFERENCED CURVE ID_____	REFERENCED CURVE ID_____	
	PASSED? Y N	PASSED? Y N	
ADD STD		REFERENCED CURVE ID_____	
	FILE#_____	FILE#_____	FILE#_____

MSB	FILE#_____	FILE#_____	FILE#_____
	PASSED? Y N	PASSED? Y N	PASSED? Y N
	CMPDS OUT_____	CMPDS OUT_____	CMPDS OUT_____
MSBD	FILE#_____	FILE#_____	FILE#_____
	PASSED? Y N	PASSED? Y N	PASSED? Y N
	CMPDS OUT_____	CMPDS OUT_____	CMPDS OUT_____
VBLK	FILE#_____	FILE#_____	FILE#_____
	VBLK____	VBLK____	VBLK____
	ACCEPTABLE? Y N	ACCEPTABLE Y N	ACCEPTABLE Y N

COMMENTS AND CORRECTIVE MEASURES (see reverse side for comment # explanations)

#_____ Sample(s)_____
#_____ Sample(s)_____
#_____ Sample(s)_____
#_____ Sample(s)_____
#_____ Sample(s)_____

Other Comments_____
_____
_____
_____

ANALYST_____ DATE_____
2ND REVIEW_____ DATE_____
AIMS ENTRY_____ DATE_____
VALIDATOR_____ DATE_____

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Comment #	Comment
1	NA
2	NA
3	Sample(s) was diluted for excessive foaming.
4	Sample(s) was diluted for non-target compounds (TICS) exceeding 5X the total response of one of the Internal Standards.
5	Sample(s) was diluted for sample matrix which resulted in method non-compliance for an Internal standard.
6	Sample(s) was diluted for sample matrix which resulted in method non-compliance for a surrogate
7	Sample(s) was diluted for TCLP matrix
8	Sample(s) was diluted for high levels of target compound(s).
9	NA
10	NA
11	Sample(s) was diluted due to insufficient volume for a lower dilution.
12	Sample(s) was diluted for viscosity.
13	Sample(s) was diluted for other reason.
14	As a result of low volume, the sample was analyzed from a vial with headspace.
15	Sample(s) was re-analyzed for surrogate recoveries outside of limits.
16	Sample(s) was re-analyzed for Internal Standard recoveries outside of limits
17	Matrix effect on Surrogate was confirmed by the analysis of ms & sd
18	Sample contains compounds which saturated the detector.This will result in non-linear results between the sample and the "DL"
19	Samples were analyzed by method 8260B.
20	Sample pH was greater than 2.
21	There was insufficient volume for re-anaylsis of the sample(s).
22	There was insufficient volume for dilution of the sample(s).
23	The VBLK was contaminated with compounds below the reporting limit.
24	The VBLK was contaminated with compounds above the reporting limit.
25	The MSB had a compound(s) outside of the method limits.
26	Sample was re-run and confimed results not consistent with historical.
27	See accompaning Job Exception Report
28	
29	
30	

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Table 6

Certificate of Composition

DESCRIPTION: Volatile Organic Compounds Mix 6

CATALOG NO.: 48799-U MFG DATE: Nov-2005

LOT NO.: LB34727 EXPIRATION DATE: Feb-2007

SOLVENT: METHANOL

MVSC 72 8-20
MVSC 73 1-7

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
BROMOMETHANE	74-83-9	99.9 (a)	2000	LB22203
CHLOROETHANE	75-00-3	98.7 (a)	2000	LB29285
CHLOROMETHANE	74-87-3	99.9 (a)	2000	LA66620
DICHLORODIFLUOROMETHANE	75-71-8	99.9 (a)	2000	LB24923
TRICHLOROFLUOROMETHANE	75-69-4	99.9 (a)	2000	LA79530
VINYL CHLORIDE	75-01-4	99.9	2000	LB18727

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
a) GC; detector HALL
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

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TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

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Table 7
54 Component

Certificate of Analysis

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DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111 MFG DATE: Nov-2003

LOT NO.: LB16275 EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
BENZENE	71-43-2	99.9	2000	2000	+/-	15.1	LB03979
BROMOBENZENE	108-86-1	99.9	2000	2009	+/-	17.4	LA97903
BROMOCHLOROMETHANE	74-97-5	99.7	2000	1967	+/-	33.3	LA67395
BROMODICHLOROMETHANE	75-27-4	99.9	2000	2103	+/-	0.1	LB15447
BROMOFORM	75-25-2	99.9	2000	1974	+/-	38.7	LB15898
CARBON TETRACHLORIDE	56-23-5	99.9	2000	1960	+/-	32.4	LA55581
CHLOROBENZENE	108-90-7	99.9	2001	2029	+/-	14.3	LB09884
CHLOROFORM	67-66-3	99.9	2000	2000	+/-	18.8	LA55585
CIS 1,3-DICHLOROPROPENE (Z)	10061-01-5	96.1	2000	2036	+/-	12.1	LA60646
CIS-1,2-DICHLOROETHYLENE	156-59-2	97.6	2000	1947	+/-	26.7	LA97197
DIBROMOCHLOROMETHANE	124-48-1	99.9	2001	2022	+/-	11.2	LA87237
DIBROMOMETHANE	74-95-3	99.8	2000	2000	+/-	33.6	LA39031
ETHYLBENZENE	100-41-4	99.5	2000	2040	+/-	8.0	LA40866
HEXACHLOROBUTADIENE	87-68-3	98.2	2001	1946	+/-	45.0	LA95300
ISOPROPYLBENZENE (CUMENE)	98-82-8	99.0	2000	2012	+/-	17.3	LB01119
M-XYLENE (5)	108-38-3	99.8	2001	*****			LB15074
METHYLENE CHLORIDE	75-09-2	99.9	2000	1957	+/-	28.9	LA88418
N-BUTYLBENZENE	104-51-8	98.7	2000	1996	+/-	25.3	LB09309
N-PROPYLBENZENE	103-65-1	99.9	2001	2028	+/-	15.6	LA92696
NAPHTHALENE	91-20-3	99.9	2000	1950	+/-	39.5	LA97766
O-XYLENE	95-47-6	99.5	2000	2022	+/-	9.8	LB08117
P-ISOPROPYLTOLUENE	99-87-6	99.9	2000	1986	+/-	20.7	LA41611
P-XYLENE (5)	106-42-3	99.9	2000	*****			LB04801
SEC-BUTYLBENZENE	135-98-8	99.4	2000	1993	+/-	31.6	LA51283
STYRENE	100-42-5	99.9	2001	2012	+/-	11.8	LB09037
TERT-BUTYLBENZENE	98-06-6	99.9	2000	1981	+/-	21.8	LB09550
TETRACHLOROETHENE	127-18-4	99.9	2001	2029	+/-	29.4	LB05248

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

Elwood Doughty
Quality Control Supervisor

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Certificate of Analysis

DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111 MFG DATE: Nov-2003

LOT NO.: LB16275 EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
TOLUENE	108-88-3	99.7	2001	2020	+/-	15.8	LA90411
TRANS 1,3-DICHLOROPROPENE (E)	10061-02-6	98.5	2000	2052	+/-	12.9	LB06449
TRANS-1,2-DICHLOROETHYLENE	156-60-5	99.9	2000	1910	+/-	36.2	LB02428
TRICHLOROETHYLENE	79-01-6	98.5	2001	1980	+/-	20.2	LB04303
1,1-DICHLOROETHANE	75-34-3	97.0	2000	1968	+/-	32.1	LA54711
1,1-DICHLOROETHYLENE	75-35-4	99.9	2000	1980	+/-	46.1	LB04593
1,1-DICHLOROPROPENE	563-58-6	98.0	2000	1958	+/-	20.8	LB12558
1,1,1-TRICHLOROETHANE	71-55-6	99.9	2000	1973	+/-	26.8	LB14220
1,1,1,2-TETRACHLOROETHANE	630-20-6	99.1	2001	2000	+/-	16.1	LB01555
1,1,2-TRICHLOROETHANE	79-00-5	99.3	2000	2038	+/-	12.6	LB03464
1,1,2,2-TETRACHLOROETHANE	79-34-5	97.5	2000	1974	+/-	31.7	LA86969
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	97.9	2000	1978	+/-	43.5	LB06608
1,2-DIBROMOETHANE	106-93-4	99.6	2001	2029	+/-	0.1	LA87068
1,2-DICHLOROBENZENE	95-50-1	99.9	2000	2008	+/-	29.2	LA96474
1,2-DICHLOROETHANE	107-06-2	99.9	2000	1974	+/-	25.7	LA88777
1,2-DICHLOROPROPANE	78-87-5	99.9	2000	2019	+/-	9.6	LB08115
1,2,3-TRICHLOROBENZENE	87-61-6	99.75	2000	1962	+/-	18.9	LA50762
1,2,3-TRICHLOROPROPANE	96-18-4	99.1	2000	2006	+/-	17.8	LA39379
1,2,4-TRICHLOROBENZENE	120-82-1	98.6	2000	1957	+/-	52.1	LB12944
1,2,4-TRIMETHYLBENZENE	95-63-6	98.2	2000	2000	+/-	22.0	LA39081
1,3-DICHLOROBENZENE,	541-73-1	99.9	2001	2013	+/-	16.7	LA72024
1,3-DICHLOROPROPANE	142-28-9	99.9	2000	2024	+/-	11.8	LB00875
1,3,5-TRIMETHYLBENZENE	108-67-8	99.0	2000	2011	+/-	13.6	LA94493
1,4-DICHLOROBENZENE	106-46-7	99.9	2000	1992	+/-	16.2	LA50188
2-CHLOROTOLUENE	95-49-8	99.9	2000	2005	+/-	23.6	LA95842
2,2-DICHLOROPROPANE	594-20-7	98.3	2000	1968	+/-	19.4	LB01750
4-CHLOROTOLUENE	106-43-4	99.9	2001	1990	+/-	15.0	LB05252

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

Elwood Doughty
Quality Control Supervisor

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Table 8
8260 + Mix

8260X #1A MSCS 1/-720

Chemical Standard Batch Sheet
Lot #: A042263

Catalog #: 552504A	Target: 1000 - 40000 ug/ml	
Description: Custom Volatiles Standard Mix A		
Solvent: P&T Methanol	Solvent Lot: 44337	Final Volume: 100 ml

Made by: Joe Tallon	Date: 1/4/2006 8:09:50A	
Tested by:	Date:	
	By:	Date:
Packaged by: Jackie Glasgow / Staci Bodle	Date: 1/4/2006 10:49:12/	No. Units: 12
Balance Used: AT261	Serial #: 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Carbon disulfide	75-15-0	FA1A5D	J11J02	0.99	1,000.00	100.00	100.00	1,000.00
Methyl-tert-butyl ether (	1634-04-4	FA1B6C	10660BD	0.97	1,000.00	100.00	100.00	1,000.00
Iodomethane (methyl	74-88-4	FA1C2A	13906AB	0.99	1,000.00	100.00	100.00	1,000.00
Ethyl methacrylate	97-63-2	FA1C1D	09316HC	0.99	1,000.00	100.00	100.00	1,000.00
Tetrahydrofuran	109-99-9	FA1B8B	01057MC	0.99	5,000.00	500.00	500.00	5,000.00
trans-1,4-dichloro-2-butene	110-57-6	FA1C1C	160-22DD	0.99	5,000.00	500.00	500.00	5,000.00
Acetonitrile	75-05-8	FA1B13A	12067KC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	1,000.00	100.00	100.00	1,000.00
Methyl acetate	79-20-9	FA1C11C	47640/1	0.99	1,000.00	100.00	100.00	1,000.00
Methylcyclohexane	108-87-2	FA1E4A	02759BC	0.99	1,000.00	100.00	100.00	1,000.00
Cyclohexane	110-82-7	FA1C7A	03145KB	0.99	1,000.00	100.00	100.00	1,000.00

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8260+ #18 MVSC5 1-5-20

Chemical Standard Batch Sheet
Lot #: A042264

Catalog #: 552504B	Target: 5000 ug/ml	
Description: Custom Volatiles Standard Mix B		
Solvent: P&T Methanol	Solvent Lot: A041266	Final Volume: 50 ml

Made by: Joe Tallon	Date: 1/4/2006 8:30:59A	
Tested by:	Date:	
	By:	Date:
Packaged by: Jackie Glasgow / Staci Bodle	Date: 1/4/2006 10:54:16/	No. Units: 12
Balance Used: AT261	Serial #: 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
2-Chloroethyl vinyl ether	110-75-8	FA1A11D	03206CI	0.99	5,000.00	250.00	250.00	5,000.00

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Certificate of Composition 8260 + #3

MVSC 42 4 → 13

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20460869 LOT NO.: LB25705 MFG DATE: Dec-2004

SOLVENT: DEIONIZED WATER

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)		SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20008	+/- 100.0	LB21530
ACRYLONITRILE	107-13-1	99.9	20000	+/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

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8260+#4 mvsc 5 1→10

Chemical Standard Batch Sheet
Lot #: A042268

Catalog #: 556843	Target: 5000 ug/ml	
Description: Custom Vinyl Acetate Standard		
Solvent: P&T Methanol	Solvent Lot: A038421	Final Volume: 25 ml

Made by: Joe Tallon	Date: 1/4/2006 9:40:21A	
Tested by:	Date:	
	By:	Date:
Packaged by: Jackie Glasgow / Staci Bodle	Date: 1/4/2006 10:58:29A	No. Units: 12
Balance Used: AT261	Serial #: 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Vinyl acetate	108-05-4	FA1A9A	08831CW	0.99	5,000.00	125.00	125.00	5,000.00

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24 1→75



Gravimetric Certificate

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Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No.: 552501 Lot No.: A044128

Description: Custom Ketones Standard

Expiration Date[1]: March 2008 Storage: Freezer

Component #	Compound	CAS#	Percent Purity[2]	Concentration (weight/volume)[3]	Percent Uncertainty[4]
1	2-Butanone (MEK)	78-93-3	99%	5,000.00 ug/ml	+/-0.08 %
2	2-Hexanone	591-78-6	99%	5,000.00 ug/ml	+/-0.08 %
3	4-Methyl-2-pentanone (MIBK)	108-10-1	99%	5,000.00 ug/ml	+/-0.08 %
4	Acetone	67-64-1	99%	5,000.00 ug/ml	+/-0.08 %

Solvent: P/T Methanol/Water (90:10)

F. Joseph Ipiton - Mix Technician Balance: 1119141429

[1] ...ration date of the unopened ampul stored at recommended temperature.
[2] ... was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
[3] Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (seven mass levels).
[4] Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured under Restek's ISO 9001 Registered Quality System Certificate #FM60387

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table 9
Second Source

ULTRA SCIENTIFIC
Analytical Solutions

60 Comp S. Source

Certificate of Analysis MUSC 71

VOC Mixture

Product DWM-588 **Expiration Date:** Dec-2008
Lot Number: CB-2659 **Page:** 1 of 3

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
bromochloromethane	000074-97-5	JS-16015HS	2006 ± 10 µg/mL
bromodichloromethane	000075-27-4	DU-14522LS	2006 ± 10 µg/mL
bromoform	000075-25-2	DU-06126KS	2006 ± 10 µg/mL
carbon tetrachloride	000056-23-5	01704MF	2006 ± 10 µg/mL
chloroform	000067-66-3	BS-03041BS	2006 ± 10 µg/mL
dibromochloromethane	000124-48-1	DO-12622CI	2006 ± 10 µg/mL
dibromomethane	000074-95-3	EM-01514TJ	2006 ± 10 µg/mL
methylene chloride	000075-09-2	44267	2006 ± 10 µg/mL
trichlorofluoromethane	000075-69-4	DR-16417BR	2006 ± 10 µg/mL
1,2-dibromoethane	000106-93-4	TB-101777	2006 ± 10 µg/mL
1,1-dichloroethane	000075-34-3	64552/1	2006 ± 10 µg/mL
1,2-dichloroethane	000107-06-2	KN-09446KN	2006 ± 10 µg/mL
1,1-dichloroethene	000075-35-4	01218EC	2007 ± 10 µg/mL
cis-1,2-dichloroethene	000156-59-2	13707BO	2006 ± 10 µg/mL
trans-1,2-dichloroethene	000156-60-5	DO-07817JR	2006 ± 10 µg/mL
1,1,1,2-tetrachloroethane	000630-20-6	CO-12312LI	2006 ± 10 µg/mL
1,1,2,2-tetrachloroethane	000079-34-5	10917TB	2006 ± 10 µg/mL
tetrachloroethene	000127-18-4	PS-00344BR	2006 ± 10 µg/mL
1,1,1-trichloroethane	000071-55-6	LU-13149TR	2006 ± 10 µg/mL
1,1,2-trichloroethane	000079-00-5	JB-0701HH	2006 ± 10 µg/mL
trichloroethene	000079-01-6	KN-08846KN	2006 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED ISO 17025 Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

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SUPERCEDES: Revision 7

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product DWM-588
Lot Number: CB-2659

Expiration Date: Dec-2008
Page: 2 of 3

Analyte	CAS#	Analyte Lot	True Value
1,2-dibromo-3-chloropropane	000096-12-8	OGF-01	2005 ± 10 µg/mL
1,2-dichloropropane	000078-87-5	DC-120777	2005 ± 10 µg/mL
1,3-dichloropropane	000142-28-9	PR-17916MR	2006 ± 10 µg/mL
2,2-dichloropropane	000594-20-7	CI-05304BI	2005 ± 10 µg/mL
1,1-dichloropropene	000563-58-6	34768-21	2006 ± 10 µg/mL
cis-1,3-dichloropropene	010061-01-5	35072-03	2006 ± 10 µg/mL
trans-1,3-dichloropropene	010061-02-6	34251-41	2005 ± 10 µg/mL
hexachlorobutadiene	000087-68-3	339923/1	2005 ± 10 µg/mL
1,2,3-trichloropropane	000096-18-4	12020TF	2006 ± 10 µg/mL
naphthalene	000091-20-3	14205KB	2005 ± 10 µg/mL
benzene	000071-43-2	31072	2006 ± 10 µg/mL
n-butylbenzene	000104-51-8	AA-28519CO	2005 ± 10 µg/mL
sec-butylbenzene	000135-98-8	MR-11305DN	2006 ± 10 µg/mL
tert-butylbenzene	000098-06-6	MQ-04010MQ	2006 ± 10 µg/mL
ethylbenzene	000100-41-4	033067	2005 ± 10 µg/mL
isopropylbenzene	000098-82-8	EN-00621TG	2006 ± 10 µg/mL
4-isopropyltoluene	000099-87-6	PP-05104CP	2006 ± 10 µg/mL
n-propylbenzene	000103-65-1	LO-14503MR	2006 ± 10 µg/mL
styrene	000100-42-5	MQ-11229MQ	2005 ± 10 µg/mL
toluene	000108-88-3	43045	2006 ± 10 µg/mL
1,2,4-trimethylbenzene	000095-63-6	BO-13528BI	2006 ± 10 µg/mL
1,3,5-trimethylbenzene	000108-67-8	KM-02011HM	2007 ± 10 µg/mL
o-xylene	000095-47-6	DO-06834CO	2006 ± 10 µg/mL
m-xylene	000108-38-3	DI-00459CJ	2006 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0951-01

250 Smith Street, North Kingstown, RI 02852 USA
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Dr. Edward Fitzgerald,
Senior Scientist

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ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product DWM-588 **Expiration Date:** Dec-2008
Lot Number: CB-2659 **Page:** 3 of 3

Analyte	CAS#	Analyte Lot	True Value
p-xylene	000106-42-3	03747LN	2005 ± 10 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	2005 ± 10 µg/mL
bromobenzene	000108-86-1	CG-02513MF	2006 ± 10 µg/mL
chlorobenzene	000108-90-7	63148HZ	2006 ± 10 µg/mL
2-chlorotoluene	000095-49-8	KS-06506BN	2005 ± 10 µg/mL
4-chlorotoluene	000106-43-4	CR-14512LQ	2005 ± 10 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	2005 ± 10 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	2006 ± 10 µg/mL
1,2,3-trichlorobenzene	000087-61-6	LI-12912PF	2006 ± 10 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	2006 ± 10 µg/mL
bromomethane	000074-83 9	06623AQ	2008 ± 10 µg/mL
chloroethane	000075-00-3	00223KG	2009 ± 10 µg/mL
chloromethane	000074-87-3	07-44048	2009 ± 10 µg/mL
dichlorodifluoromethane	000075-71-8	N960053	2008 ± 10 µg/mL
vinyl chloride	000075-01-4	UN-1086	2009 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851-01

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Table10
8260+ Second Source

8260+#1 Sec Source MUSC61→10

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687608 LOT NO.: LB35787 EXPIRATION DATE: Jan-2007

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)		WEIGHT CONCENTRATION (3)		SUPELCO LOT NO
ACETONITRILE	75-05-8	99.9		40001	+/- 200.0	LB34175
CARBON DISULFIDE	75-15-0	99.9	(a)	999	+/- 5.0	LB09107
CYCLOHEXANE	110-82-7	99.9		1000	+/- 5.0	LB18076
ETHYL METHACRYLATE	97-63-2	99.3		1002	+/- 5.0	LA29651
FREON 113	76-13-1	99.9	(b)	1001	+/- 5.0	LA33286
METHYL ACETATE	79-20-9	98.1		1001	+/- 5.0	LB32233
METHYL CYCLOHEXANE	108-87-2	99.8		1001	+/- 5.0	LB06982
METHYL TERT-BUTYL ETHER	1634-04-4	99.9		1002	+/- 5.0	LB34302
TETRAHYDROFURAN	109-99-9	97.4		4999	+/- 25.0	LA58136
TRANS-1,4-DICHLORO-2-BUTENE	110-57-6	98.2		5002	+/- 25.0	LB10202
1-CHLOROHEXANE	544-10-5	99.9		1000	+/- 5.0	LB18907

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
a) GC; detector FPD
b) GC; detector HALL
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

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8260 HHL See Source MVSC6 11→20

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687609 LOT NO.: LB35788 EXPIRATION DATE: Jan-2007

SOLVENT: DEIONIZED WATER 50 %
METHANOL 50 %

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACETONE	67-64-1	99.9	5004 +/- 25.0	LB31953
IODOMETHANE	74-88-4	99.9	1004 +/- 5.0	LA73149
VINYL ACETATE	108-05-4	99.9	5002 +/- 25.0	LB31606
2-BUTANONE	78-93-3	99.9	5004 +/- 25.0	LB19842
2-HEXANONE	591-78-6	99.9	5004 +/- 25.0	LB08447
4-METHYL-2-PENTANONE	108-10-1	99.9	5004 +/- 25.0	LA99226

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

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Certificate of Analysis

MUSC 66 3-7

DESCRIPTION: 2-Chloroethyl vinyl ether

CATALOG NO.: 40017 MFG DATE: Feb-2005

LOT NO.: LB27794 EXPIRATION DATE: Feb-2008

SOLVENT: METHANOL

ANALYTE	CAS NUMBER	PERCENT PURITY(1)	WEIGHT(2) CONCENTRATION	ANALYTICAL(3) CONCENTRATION	STD DEV	SUPELCO LOT NO
2-CHLOROETHYL VINYL ETHER	110-75-8	99.9	5000	5000	+/- 55.9	LB01239

(1) Determined by capillary GC-FID, unless otherwise noted.
(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

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SUPERCEDES: Revision 7

Certificate of Composition

8260 #3 Sec. Source
MVSC 7
1-7/10

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687606 LOT NO.: LB35789 EXPIRATION DATE: Jul-2006

SOLVENT: DEIONIZED WATER

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20012 +/- 100.1	LB21530
ACRYLONITRILE	107-13-1	99.9	20008 +/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

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SUPERCEDES: Revision 7

Table 11
8260 Add

MVSC 71-18-20
72-01-07

Chemical Standard Batch Sheet
Lot #: A042005

Catalog #: 552546	Target: 2000-80000 ug/ml	
Description: Custom Volatiles Standard		
Solvent: P&T Methanol	Solvent Lot: 44337	Final Volume: 100 ml

Made by: Ryan Miller	Date: 12/19/2005 10:12:4
Tested by:	Date:
	By: Date:
Packaged by: / SLB / JG	Date: 12-20-05 No. Units: 12
Balance Used: AT400	Serial #: 1113372841

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Allyl chloride (	107-05-1	FA1B13D	00305HO	0.99	2,000.00	200.00	200.00	2,000.00
Chloroprene	126-99-8	FA1D8B	051215JLM	0.99	2,000.00	200.00		0.00
Pentachloroethane	76-01-7	FA1C3B	OGL01	0.98	2,000.00	200.00	200.00	2,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	2,000.00	200.00	200.00	2,000.00
Dichlorodifluoromethane	75-71-8	HOOD	A042007	0.99	2,000.00		4.20 (ml)	1,978.41
Dichlorofluoromethane	75-43-4	HOOD	A042008	0.99	2,000.00		3.10 (ml)	1,974.39
Chlorodifluoromethane	75-45-6	VOA Lab	A042009	0.99	2,000.00		2.40 (ml)	2,016.62
Ethyl acetate	141-78-6	FA1C5B	11073ED	0.99	2,000.00	200.00	200.00	2,000.00
Diisopropyl ether (DIPE)	108-20-3	FA1C2B	13450CB	0.99	2,000.00	200.00	200.00	2,000.00
Hexachloroethane	67-72-1	RA1B6D	12719A0	0.99	2,000.00	200.00	200.00	2,000.00
Methyl methacrylate	80-62-6	FA1C2D	09505TO	0.99	2,000.00	200.00	200.00	2,000.00
Methacrylonitrile	126-98-7	FA1C2C	04406MI	0.99	2,000.00	200.00	200.00	2,000.00
Diethyl ether (ethyl ether)	60-29-7	FA1C1A	17676TQ	0.99	2,000.00	200.00	200.00	2,000.00
2-Nitropropane	79-46-9	RA1C11C	04609PN	0.98	10,000.00	1,000.00	1,000.00	10,000.00
Pr itrile	107-12-0	FA1C3D	10101EB	0.98	20,000.00	2,000.00	2,000.00	20,000.00
Cyclohexanone	108-94-1	RA1D2B	10513PA	0.99	20,000.00	2,000.00	2,000.00	20,000.00
ert-Butanol (TBA)	75-65-0	RA1H2D	06648PC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
l-Butanol	71-36-3	FA1G1B	8238	0.99	80,000.00	8,000.00	8,000.00	80,000.00
sobutanol	78-83-1	FA1C3A	00439HD	0.99	80,000.00	8,000.00	8,000.00	80,000.00
1,4-Dioxane	123-91-1	RA1H3B	03053BD	0.99	80,000.00	8,000.00	8,000.00	80,000.00

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

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Add + MVSC 74 18→20
L 75 1→7

RESTEK

CERTIFICATE OF COMPOSITION

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 558661 Lot No.: A042271
Description: Custom Volatiles Standard
Expiration Date[1]: July 2007 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	2-Propanol (isopropanol)	67-63-0	99%	20000 ug/mL	+/- 0.1
2	1-Propanol	71-23-8	99%	20000 ug/mL	+/- 0.1
3	n-Hexane (C6)	110-54-3	99%	1000 ug/mL	+/- 0.1
4	Acetaldehyde dimethyl acetal	534-15-6	99%	5000 ug/mL	+/- 0.1
5	Ethyl-tert-butyl ether (ETBE)	637-92-3	99%	1000 ug/mL	+/- 0.1
6	tert-Amyl methyl ether (TAME)	994-05-8	99%	1000 ug/mL	+/- 0.1
7	n-Heptane (C7)	142-82-5	99%	1000 ug/mL	+/- 0.1
8	2-Chlorobenzotrifluoride	88-16-4	99%	1000 ug/mL	+/- 0.1
9	3-Chlorobenzotrifluoride	98-15-7	99%	1000 ug/mL	+/- 0.1
10	4-Chlorobenzotrifluoride	98-56-6	98%	1000 ug/mL	+/- 0.1
11	3-Chlorotoluene	108-41-8	99%	1000 ug/mL	+/- 0.1
12	1,2,3-Trimethylbenzene	526-73-8	99%	1000 ug/mL	+/- 0.1
13	Dicyclopentadiene	77-73-6	98%	1000 ug/mL	+/- 0.1
14	1,3,5-Trichlorobenzene	108-70-3	99%	1000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .32mm x 1.8um
Rtx-502.2 (cat #10921)

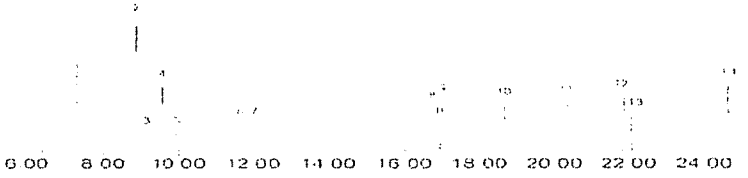
Carrier Gas:
helium @ 2.2 ml/min

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD



Manufactured By FJT
John Fidgett
John Fidgett QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (7 mass levels)
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM90397

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Table 12
BFB
IS & SS

MVSL 3 1-710

RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30067 Lot No.: A038850
Description: 4-Bromofluorobenzene Standard
Expiration Date[1]: January 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1-Bromo-4-fluorobenzene (BFB)	460-00-4	99%	2500 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat #10910)

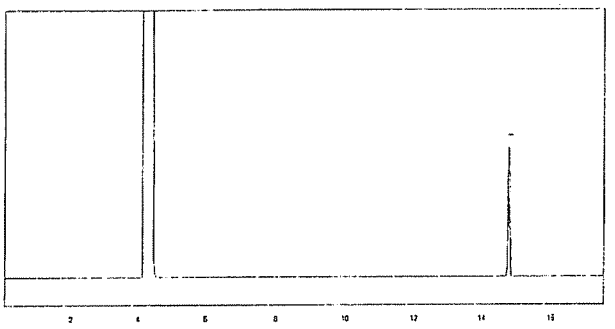
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
50°C to 240°C @ 10°C/min

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: MEW
John Liggett
John Liggett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

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SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	59 of 60

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

MWSC08 (11-20)

RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30091 Lot No.: A036981

Description: L/C VOA Internal Standard Mix

Expiration Date[1]: April 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Difluorobenzene	540-36-3	99%	2500 ug/mL	+/- 0.1
2	Chlorobenzene-d5	3114-55-4	99%	2500 ug/mL	+/- 0.1
3	1,4-Dichlorobenzene-d4	3855-82-1	99%	2500 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x 53mm x 3.0um
Rtx-502.2 (cat #10910)

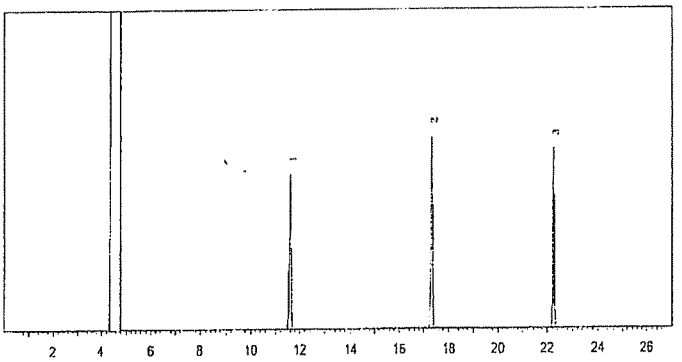
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
40°C (hold 2 min) to 240°C
@ 8°C/min

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: n/a

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels)
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

MVSC { 73 18-20
74 1-17

Method 8260 Surrogate Standard Mixture

Product STM-530 (4) Comp 5.0r **Expiration Date:** Sep-2008
Lot Number: CB-1899 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
4-bromofluorobenzene	000460-00-4	12515BO	2512 ± 13 µg/mL
dibromofluoromethane	001868-53-7	90004843	2512 ± 13 µg/mL
1,2-dichloroethane-d4	017060-07-0	PSO 5A-048	2512 ± 13 µg/mL
toluene-d8	002037-26-5	PSO AG-433	2510 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED ISO 17025 Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald
Dr. Edward Fitzgerald,
Senior Scientist

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

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SOP No.	Revision No.	Effective Date	Page
AGE-8082-63	4	September 29, 2003	1 of 14

TITLE: ANALYSIS OF PCBs – METHOD – 8082

SUPERCEDES: Revision 3

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager	Verl D. Preston	9/29/03
Chris Oprandi, Laboratory Director	for C.O.	9/29/03
Gary Rudz, Supervisor	Gary Rudz	9/29/03

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1.0 IDENTIFICATION OF TEST METHOD

1.1 EPA Method 8082A

2.0 APPLICABLE MATRIX

2.1 Applicable matrix is soil, water, wipes and waste (oil) samples.

3.0 REPORTING LIMIT

3.1 The routine reporting limits are:

0.5ug/L for water samples
16.67ug/Kg for soil samples (100% Dry)
1mg/Kg for oil/waste samples
1 ug/wipe for wipe sample

4.0 SCOPE AND APPLICATION

4.1 This method is used to quantify polychlorinated biphenyls (PCBs) in extracts from aqueous, soil, sludge or oil matrices by direct injection techniques into a capillary column equipped gas chromatograph. An electron capture detector (ECD) is employed for identification and quantification. This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and the integration of gas chromatograms.

TITLE: ANALYSIS OF PCBs – METHOD – 8082

SUPERCEDES: Revision 3

Compound	CAS No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

5.0 SUMMARY OF THE TEST METHOD

5.1 Wastewater samples, approximately 1 liter of sample is extracted with Methylene chloride using a separatory funnel (SOP ASP-3510B-80) or a continuous liquid liquid extractor (SOP No. ASP-3520B-85). Soil samples are extracted using approximately 30g of soil/solid sample using sonication (SOP No. ASP-3550B-90) The extract is then exchanged to hexane and concentrated to 10 ml or less. The final extract is then separated by gas chromatography and detected by an electron capture detector.

Florisil column cleanup procedures and sulfur removal procedures may be utilized to mitigate any interferences that may be encountered during analysis. Although these procedures may eliminate several interferences, contamination of the sample may come from a variety of sources, including solvents, reagents, glassware and any of the hardware used in sample processing. For this reason, reagent and solvent blanks should be analyzed to insure their purity.

6.0 DEFINITIONS

6.1 Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual.

7.0 INTERFERENCES

7.1 Method interferences can be minimized by proper glassware cleaning methods, instrument maintenance, and the use of high purity reagents and solvents.

7.2 Sulfuric acid (ASP-3665A-98) and copper cleanup Method 3660 are part of the extraction procedure for all PCB samples.

7.3 Gel Permeation Cleanup (ASP-3640A-96) and Florisil Cartridge Cleanup (ASP-3620A-95) may be also used on samples when specified by project or historical results warrant further cleanup.

TITLE: ANALYSIS OF PCBs – METHOD – 8082

SUPERCEDES: Revision 3

8.0 SAFETY

8.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document. Safety glasses with side shields are to be worn in all laboratory areas and appropriate Personal Protective Equipment should be worn when working with chemicals and/or samples. Avoid direct contact with chemicals and/or samples since many chemicals can be absorbed through the skin and the samples may contain hazardous constituents. Wash your hands thoroughly with soap and water after contact with any chemical or sample. Always proceed as if a chemical, sample, and/or procedure is hazardous unless you have specific information indicating that it is not.

Sample handling procedures should be performed in an operational fume hood.

The analyst should be wearing; (at a minimum)
- Proper laboratory dress
- Safety Glasses
- Gloves
- Lab Coat

8.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

8.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

TITLE: ANALYSIS OF PCBs – METHOD – 8082

SUPERCEDES: Revision 3

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

8.4 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory

8.4.1 Aroclors have been classified as a potential carcinogen. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure.

8.5 All ^{63}Ni (ECDs) shall be leak tested every six months.

9.0 EQUIPMENT AND SUPPLIES

9.1 Gas chromatograph suitable for on-column injection and all required materials, i.e., syringes, columns, gases, detector and a data processing system capable of measuring peak areas and heights.

9.1.1 Hewlett Packard 5890 gas chromatograph
9.1.2 Hewlett Packard 7673 Auto Sampler
9.1.3 Hewlett Packard 3392 and 3396A Integrators

SUPERCEDES: Revision 3

9.1.4 Capillary columns as described in sections 5.3
9.1.5 Electron Capture Detector
9.1.6 PE Nelson Turbochrome data system
9.1.7 Carrier Gas Hydrogen
9.1.8 Make Up Gas - Argon/Methane
9.1.9 Syringes - various

10.0 REAGENTS AND STANDARDS

10.1 Reagents or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to 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 determination.

10.2 Standards are stored in the GC Standard Incubator at 4±2°C in teflon-sealed amber containers in the dark.

10.3 All stock standard solutions are replaced before the expiration date. All other standard dilutions or working standards are discarded after six month (or at the stock standard expiration date, whichever comes first) or sooner if routine QC indicates a problem

10.3.1 Certified PCB Mixes (Aroclors 1016/1260, 1221, 1232, 1242, 1248, 1254)
10.3.2 Second Source PCB Mixes for all Aroclors (Different Manu. or Lot #) to verify consistant response of newly prepared calibration curve.
10.3.3 Acetone (pesticide grade)
10.3.4 Hexane (pesticide grade)

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Aqueous samples are to be collected in a 1-liter amber glass jar and stored at 4±2°C. Organic preparation is to be performed within 7 days of collection.

11.2 Soil (solid) samples are to be collected in a 4 oz jar and stored at 4±2°C. Organic preparation is to be performed within 14 days of collection.

11.3 Analysis of the extracts for both waters and soils is to be performed within 40 days of preparation.

11.4 Holding times specified in project specific quality assurance plans may supersede the above listed method criteria.

11.5 Extracts are stored in Incubator #5 under refrigeration at 4±2°C.

SUPERCEDES: Revision 3

11.5.1 For CLP, AFCEE, and USACE samples, the extracts are maintained in secure storage incubator SC# 4

12.0 QUALITY CONTROL

12.1 A method blank must be prepared and analyzed with each batch (maximum 20 samples). The acceptance criterion is that the method blank must contain a concentration less than the reporting limit for all target analytes. If the blank fails this criterion, the entire prep batch must be re-extracted and analyzed.

12.1.1 For USACE, the method blank must contain concentrations < ½ the reporting limit.

12.2 Analysis of at least one matrix spike and one matrix spike duplicate per batch. Sample spike and duplicate recoveries should fall within the laboratory Quality Control limits that are updated annually based upon historical data. If the recoveries are not achieved, the data is still valid as long as the matrix spike blank is acceptable. The routine matrix spiking solution is an Aroclor 1016/1260 mixture prepared at 5.0ng/ul. During preparation, 1000ul of this solution is added to all quality control samples (MSB/MS/MSD). The resulting expected concentration for both aqueous and soil sample is 0.50ng/ul in the 10ml extract. This translates to final sample concentrations of: 5.0 ug/L for a 1-Liter Aqueous samples, and 166.7ug/Kg for 30.0grams of Soil sample at 100% dry.

12.2.1 Limited sample volume can allow for the analysis of a matrix spike blank duplicate instead of a MS and MSD pair.

12.3 A matrix spike blank must be prepared and analyzed with each batch. Spike recoveries should fall within the laboratory Quality Control limits that are updated annually based upon historical data.

13.0 CALIBRATION AND STANDARDIZATION

13.1 An Initial Calibration Curve (ICC) must be run for the Aroclor mix 1016/1260 and any Aroclor that is specified by a project.

13.1.1 The curve will consist of a minimum of five concentration points ranging from 0.05ng/uL–2.5ng/uL. The concentration points will be prepared by diluting a certified Aroclor standard. Lower Levels may be prepared at the time of calibration by diluting existing higher levels. Preparation is performed by diluting a level by dilution factor of 10 to get a new level. The following table summarizes the concentration levels used and the associated reporting levels for water and soil samples.

Calibration Standard Conc.	Report Level Water (1L)	Report Level Soil (30g/100% dry)
0.05 ng/uL	0.50 ug/L	16.67 ug/kg
0.10 ng/uL	1.0 ug/L	33.34 ug/kg
0.25 ng/uL	2.5 ug/L	83.35 ug/kg
0.50 ng/uL	5.0 ug/L	166.7 ug/kg
1.0 ng/uL	10.0 ug/L	333.4 ug/kg
2.5 ng/uL	25.0 ug/L	833.5 ug/kg

13.1.2 For each Aroclor, select 3–5 unique chromatographic peaks that represent the major biphenyls present in the particular aroclor.

13.1.3 Aroclor 1016/1260 will be defined by a retention time window empirically determined for each analytical column as follows, for each Component:

13.1.3.1 Calculate 3X Standard deviation (in minutes) of 3 injections of Midpoint Standard (0.5ng) over a 72-hour period.

13.1.3.2 Any component with a 3X Standard deviation of <0.05ng minutes defaults to 0.05 minutes or 3.0 seconds at minimum. It should be noted that the primary means of identification are based upon pattern recognition. Matrix effects may shift actual retention times, but the primary pattern may remain intact.

13.1.3.3 Turbochrom calibration files retention time windows should be updated with these values for each component for each Aroclor component chosen.

13.1.4 The Percent Relative Standard Deviation for each individual aroclor must be ≤20%, or have a Correlation Coefficient "R" ≥0.995 (R squared ≥0.990) for the ICC to be acceptable.

13.1.4.1 The curve may be determined using a linear regression fit if a minimum of 5 points are used.

13.1.4.2 If a quadratic regression fit is required to obtain acceptable data, 6 or more calibration points must be employed.

13.1.4.3 If RSD ≤20% for each peak, then linearity of the detector can be assumed for all other Aroclors over the same analytical range.

13.2 A single point calibration must be run for all other Aroclors.

13.2.1 The Aroclor concentration should be near the midpoint concentration of the 1016/1260 curve (~0.5ng/ul on column).

13.2.2 For each Aroclor, five major chromatographic peaks are chosen that represent the key to the patterns present in each particular aroclor. This is important due

to the assessment of degraded patterns when determining identification. For Aroclor 1221, three peaks are used due to the lower number and response of the biphenyls present.

13.3 An Initial Calibration Curve must be run for the surrogate compounds Decachlorobiphenyl and Tetrachloro-meta-xylene, which is to be contained in the Ar1016/1260 calibration standards.

13.3.1 The ICC for the surrogates will require a minimum of five (5) concentration points. The concentrations will be made by serial dilutions of a certified standard. Levels are: 0.005ng, 0.010ng, 0.020ng, 0.030ng, 0.40ng, and 0.5ng.

13.3.2 The Percent Relative Standard Deviation for each surrogate must be ≤20%, or have a Correlation Coefficient "R" ≥0.995 (R squared ≥0.990) for the ICC to be acceptable. If RSD ≤20% for each peak, then linearity of the detector can be assumed. The curve may be determined linear if a minimum of 5 points are used, and quadratic if a minimum of 6 points are used.

14.0 PROCEDURE

14.1 Set up the Hewlett Packard Gas Chromatograph as a split injection dual column instrument.

14.1.1 Split injection instruments shall have different columns as to maximize the ability to confirm Aroclors present in the extracts in the most efficient manner.

Acceptable ICC's and single point calibrations are run for all Aroclors and surrogates. Both sides of a split injection instrument must be calibrated with the identical injections.

14.2 The response factor of the ICV must be ±15% D for each aroclor if quantification of that aroclor is to be made. The ICVs and CCVs may >15% biased high to confirm non-detects for any particular aroclor. ICVs and CCVs that are biased >15% low will require a rerun of a compliant standard, or a new calibration curve run.

14.2.1 An ICV will consist of a concentration point at or near the midrange of the curve, (generally 0.5ng on column). The concentration point will be prepared through serial dilutions of a certified Aroclor standard.

14.3 The retention time window determined as in 13.1.3 for each peak must be adjusted using the ICV on a daily basis to account for any shifts in the instrument's operating conditions.

14.3.1 The 1016/1260 mix contains all of the unique components (or congeners) of each individual Aroclor and if the ICV is acceptable for this, it can be understood that the ICV is acceptable for all Aroclors.

14.4 Continuing Calibration Verifications (CCVs) must bracket every 10 samples. Any other Aroclor (with an ICC) may be used along with the midpoint standard of the surrogate as a CCV pair.

14.4.1 The response factor for each peak in the CCV must be ±15% D. All data is acceptable as long as it is bracketed by an acceptable ICV and CCV, or CCV and CCV.

14.5 Each sample is injected into the gas chromatograph and its acceptable data is evaluated for Aroclor patterns and surrogate recovery. Spike recovery is also evaluated in spiked samples.

15.0 CALCULATIONS

15.1 Analyzing, confirming and calculating Aroclors.

15.1.1 Retention time windows, pattern recognition and the experience of the analyst will be employed in the identification process. If a chromatographic peak falls outside the established retention time window but an Aroclor pattern is discernible, positive identification shall be made. Turbochrom's compare function can be used to overlay AR 1016/1260 mix against unknown samples. If any Aroclors are present, corresponding congener peaks will be observed. The retention times of the congeners in other Aroclors will match the congener peaks in 1016/1260, but in different response ratios depending on the Aroclor.

15.1.2 The presence of multiple Aroclors will be noted in the summary report.

15.1.3 Single Aroclors shall be confirmed by a clearly recognizable pattern or based on historical data from a specific site.

15.1.4 Calculating the ng amount of an Aroclor in soil and aqueous matrices. This is actually an average of the concentration of the individual peaks.

15.1.4.1 Calculating the ng amount of Aroclor in each peak.

$$\frac{\text{Area of the peak (a)}}{\text{Calibration factor of peak (a)}} = \text{ng amount of peak (a)}$$

or the ng amount from the curve equation

15.1.4.2 Calculating the ng amount of Aroclor in the sample

$$\frac{\text{Peak(n)ng + peak(n+1)ng + peak(n+2)ng +}}{\text{Total Peaks}} = \text{ng amount of sample}$$

15.1.4.3 Converting ng amount to ug/Kg, ug/L and ug/wipe

$$\text{ug/Kg} = \frac{\text{(ng) x (final volume in ml) x (dilution factor)}}{\text{(injection vol. in ul) x (sample wt.) x (\% Dry)}} \quad \text{X 1000}$$

For Oils % dry = 100

$$\text{ug/L} = \frac{\text{(ng) x (final volume in ml) x (dilution factor)}}{\text{(injection volume in ul) x (sample volume in L)}}$$

$$\text{ug/wipe} = \frac{\text{(ng) x (final volume in ml) x (dilution factor)}}{\text{(injection vol. in ul) x (sample wt.) x (\% Dry)}}$$

For wipes, sample weight = 1, % Dry = 100

16.0 METHOD PERFORMANCE

16.1 An initial demonstration of capability is performed for either aqueous and soil matrices per analyst and compared to the method criteria.

16.2 MDL studies are performed annually on a matrix and instrument-specific basis.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 Calib. Check: A calibration check standard must be analyzed after every 10 sample measurements (including quality control). The percent difference between the check standard CF and the calibration standard average CF must be less than 15%. If the acceptance criteria are met, continue. If the acceptance criteria are not met, reanalyze. If reanalysis meets the acceptance criteria, continue. If reanalysis does not meet the acceptance criteria the system must be corrected before samples may be analyzed.

17.2 MBLK: A laboratory method blank must be analyzed with every set of 20 samples at a minimum of 1 per batch. Acceptance criteria are less than the report limit. If the acceptance criteria are met, the QC sample indicates no contamination due to the preparation procedure and is considered acceptable. If analyte is measured above the reporting limit, reanalyze. If reanalysis is acceptable, continue. If reanalysis again indicates contamination the sample results are not useable for drinking water samples. Results for other sample matrices may be used if they are greater than 10 times the blank contamination.

17.2.1 For USACE, the method blank must not contain compounds at levels > ½ the report level.

17.3 MSB: A matrix spike blank (lab fortified blank) must be analyzed with every batch of 20 samples or a minimum of 1 per day. The routine matrix spiking solution is an Aroclor 1016/1260 mixture prepared at 5.0ng/ul. During preparation, 1000ul of this solution is added to all quality control samples (MSB/MS/MSD). The resulting expected concentration for both aqueous and soil sample is 0.50ng/ul in the 10ml extract. This translates to final sample concentrations of: 5.0 ug/L for a 1-Liter Aqueous samples, and 166.7ug/Kg for 30.0grams of Soil sample at 100% dry. Statistical in-house acceptance limits are updated annually and are maintained in the laboratory LIMS system. If the required recovery limits are met, the QC sample indicates control of the preparation procedure and is considered acceptable. If the recovery limits are not met, reanalyze. If reanalysis yields acceptable recovery, continue. If the recovery limits are again not met the batch results are not useable unless the control sample recovery is high and the sample concentrations are below the reportable limit.

17.4 MS: A matrix spike sample must be set for one in every batch of 20 samples. Statistical in-house acceptance limits are updated annually and are maintained in the laboratory LIMS system. If the acceptance criteria are met, no adverse matrix effects are indicated. If acceptance criteria are not met, reanalyze. If reanalysis yields acceptable recovery, continue. If the recovery limits are again not met dilute the MS sample and reanalyze. To minimize bias, samples for matrix spike analysis shall be chosen at random. All analytes in the spike solution shall be measured unless they are not of interest in the spiked sample.

17.5 MSD: Along with every matrix spike sample, a duplicate MS must also be set. This sample is the matrix spike duplicate (MSD). Acceptance criteria are <30% RPD. If the acceptance criteria are met, continue. If the acceptance criteria are not met, reanalyze. If reanalysis yields acceptable recovery, continue. If the recovery limits are again not met report the data yielding the best recovery noting the duplicate failure.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 Blank contamination and recoveries outside this range may lead to: Re-extraction if within holding time and volume available, noting recoveries in case narrative, or flagging values as estimated. The spike results, sample matrix, and reported positives in the prep batch are also to be considered. The Project Manager will be notified with a job exception, and acceptability may be determined by citing historical, sample, and method results on a case by case basis

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

SUPERCEDES: Revision 3

19.1 When QC results, unknown positives, or sample matrix present the analyst with questionable data, the spike results, sample matrix, and reported positives in the prep batch are all to be considered. Acceptability may be determined by citing historical, sample, and method results on a case by case basis. The project manager shall be notified of any method anomalies, and can then contact the client as to specific instructions on the usability of the data and any further actions

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

20.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic waste generated in the lab.
- Solvent waste generated by the extraction
- Expired primary and working PCB standards.
- Vials containing sample extracts.

20.3 All solvent waste generated by the extraction is to be disposed of in a labeled "C" waste container.

20.4 All acidified aqueous waste is to be disposed of into a labeled "A" waste container

21.0 REFERENCES

21.1 Method 8082 U.S. Environmental Protection Agency, Office of Solid Waste and Energy Response, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," 3rd edition, SW-486, update III, Dec. 1996.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Job Summary Sheet and Data Review Checklist

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Laboratory Director change – signature updated.

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23.2 Section 8.0: Added specific safety information developed by STL Corporate EH&S
23.3 Section 11.2: Included soil preservation and holding time information.
23.4 Section 11.5.1: Included special storage requirements for CLP, AFCEE and USACE
23.5 Section 12.1.1: Added USACE method blank acceptance criteria
23.6 Section 12.2: Included spike solution preparation and correlated spike concentrations to final concentrations in samples
23.7 Section 13.1.1: Developed table to correlate calibration concentrations to final concentrations in samples
23.8 Section 13.1.4: Expanded information regarding regression fit used for calibration.
23.9 Section 14.3: Made reference to section 13.1.3 for development of the retention time window.
23.10 Section 17.2.1: Added USACE method blank acceptance criteria
23.11 Section 17.4: Removed reference to default MS acceptance limits (70-130%) and replaced with requirement for development of statistical in-house acceptance limits.
23.12 Section 17.5: Removed reference to default MSB acceptance limits (70-130%) and replaced with requirement for development of statistical in-house acceptance limits.
23.13 Section 20.0: Added specific waste management information developed by STL Corporate EH&S.
23.14 Section 22.1: Added Job Summary Sheet and Stat Review Checklist as attachment

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22.1 Job Summary Sheet and Data Review Checklist

Method: _____ Client: _____ Job # A03 -

Level: 1 2 3 4 No. Samples _____ Tat DUE /

Inst.: Ch. A / B *Matrix: W / S / Oil / Wipe* RUSH ☐

CCV Criteria - Date: Comments:

Ch: A / B / /2003

Ch: A / B / /2003

Ch: A / B / /2003

CU TREATED:	Y N	DL CODE
ACID TREAT	Y N	002 / 008
DILUTIONS:	Y N	Matr. / Pos.

BATCH REVIEW:

Blanks Clean? Y / N Surr. In Limits Y / N

MSB Rec. In Limits? Y / N

MSBD Rec. In Limits? Y / N RPD Y / N

SURROGATE Review List Samples: =>

TCX Out / DCB In _____ DCB Out / TCX In _____ BOTH Out _____ Diluted Out _____

MS Rec. In Limits? Y / N / DL NA

MSD Rec. In Limits? Y / N / DL RPD Y / N / DL

Comments: _____

☐ No Positives Above RL

☐ Heavy Matrix Effects

PROCESSING	DATE	ANALYST(S)
COMPARED		/
COMMENTS ENTERED		
REPORTING CHANNEL	A / B	
TX0'D & MOVED		
AIMS ENTERED:		
WORKSHEETS PRINTED:		
REVIEWED & CLOSED:		
RUNLOG COPIED (L4)		
CLIENT FORMS PRINTED (L4)		
STANDARD SUMMARY (L4)		

Manual Integrations Needed for Samples? (Y / N)

BA	Baseline Adjustment
NP	Negative Peaks
CE	Co-Elution
PS	Peak Splitting
ME	Matrix Effects
OT	Other

Review of Manual Integrations Y / NA

Initial & Date _____ ___/___

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LABORATORY STANDARD OPERATING PROCEDURES

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TITLE: ICP-MS Analysis Method 200.8/6020/ CLP(ILMO5.2)/AFCEE

SUPERCEDES: Revision 4

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl Preston, Quality Manager	Verl D. Preston	9/26/06
Christopher Spencer, Laboratory Director	Christopher Spencer	9/26/06
Jennifer Pierce, Supervisor	Jennifer Pierce	9/26/06

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1.0 IDENTIFICATION OF TEST METHOD

1.1 This SOP is used for the determination of Dissolved/Soluble, Total, and Total Recoverable elements using a Perkin Elmer Elan 9000 ICP-MS. This SOP is based on 40 CFR136 Method 200.8, SW846 Method 6020 and CLP (ILMO5.2).

2.0 APPLICABLE MATRIX

2.1 Digestates of ground waters, surface waters, drinking waters and soils.

3.0 REPORTING LIMIT

3.1 Table 22.1 lists the PQLs for each analyte. For routine analyses, the PQLs listed are the lowest concentrations that can be reported. The PE Elan 9000 may also be used to analyze other elements that are not listed.

4.0 SCOPE AND APPLICATION

4.1 Reference SOP No. AME-3020-70, *Sample Preparation for ICP-MS* for the sample preparation procedures for aqueous samples. See SOP No. AME-3050B-65 for soil preparations.

4.2 Table 22.2 lists the analytical isotopes and additional monitored masses.

4.3 Table 22.3 lists achievable Instrument Detection Limits (IDLs) and Linear Dynamic Ranges (LDRs).

4.4 Table 22.4 lists achievable Method Detection Limits (MDLs). IDLs/LDRs and MDLs are

determined quarterly and annually, respectively. Current laboratory IDLs, LDRs, and MDLs are maintained in the LIMS system.

4.5 The prepared samples are categorized as Dissolved Aqueous Samples, Total Aqueous samples, Total Recoverable Aqueous Samples, and Solid Samples. All samples are digested. Samples shipped from the sample preparation lab are ready for ICP-MS analysis.

4.6 All samples, standards, and blanks are matrix matched.

4.6.1 In aqueous solution containing 2% (v/v) nitric acid. Rinsing blank contains 2% HNO_3.

4.6.2 When solid samples are analyzed: the samples, standards, blanks and the rinsing blank contain 1% Nitric acid and .5% Hydrochloric acid. Samples are diluted 1:10 to match standard matrix. The CLPSL (ERA purchased standard) is diluted 1:100.

5.0 SUMMARY OF THE TEST METHOD

5.1 Sample solution is introduced by pneumatic nebulization into radio frequency (RF) plasma where 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. The ions are detected by an electron multiplier and the ion information is processed by a data handling system.

5.2 This SOP contains the procedures for the daily operation of the PE Elan 9000. The sample preparation procedure is described in SOP *Sample Preparation for ICP-MS*. This SOP contains procedures for calibrating, standard preparation, maintenance, data handling, and quality control.

5.3 Analysis procedures are created for Method 200.8, Method 6020, and CLP (ILMO5.2). Other procedures can be developed according to the clients' requirement. When customizing an analytical procedure, the "Protocol", "Elemental Menu", "Method", "Interference" and "Procedure" files must be modified accordingly, and the supervisor should be consulted.

5.4 The normal steps on a given day in operating the Elan 9000 include:

5.4.1 Perform any routine maintenance, if required.

5.4.2 Start-up and warm-up instrument: set the instrument from *pumping* mode to *Operate* mode.

5.4.3 Tune the instrument (monthly).

5.4.4 Run Daily solution to check instrument performance

5.4.5 Record the instrument operating parameters into the Operators logbook.

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5.4.6 Prepare standards (at least weekly).

5.4.6 Put together a sequence of samples with all required quality control that is analyzed as a single unit.

5.4.7 Set-up the autosampler.

5.4.8 Prepare all the samples for analysis including the required spikes, and quality control samples.

5.4.9 Analyze the samples.

5.4.10 Print out hard copy of the data and transfer data to the network.

5.4.11 Print data set log.

5.4.12 Check the data for compliance with the method.

5.4.12 Log in compliant data.

5.4.13 Set the instrument to *pumping* mode unless another run will be analyzed.

5.4.14 Dispose of samples and standards appropriately.

5.4.15 Clean-up area.

5.4.16 If the instrument is not operating properly or requires any maintenance, report to the Supervisor for help.

6.0 DEFINITIONS

6.1 PE Elan 9000 - Abbreviation for the Perkin Elmer Elan 9000 ICP-MS instrument.

6.2 Mass Calibration – During Mass Calibration, the software matches a pre-defined, software generated mass spectrum to the output of the quadrupole. This ensures that the mass measured by the instrument, for the selected standard, are accurate.

6.3 ELGA Water - This is blank reagent water that is deionized, filtered, and has a resistivity of 18 $M\Omega cm^{-1}$.

6.4 Calibration Blank – Reagent water acidified with the same acid matrix as in the calibration standards. Also referred to as ICB and CCB.

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6.5 Calibration Standard (CAL) - A series of solutions with known concentrations used to calibrate the instrument response with respect to analyte concentration.

6.6 Dissolved Metals (Soluble Metals) - Samples that have gone through a digestion procedure. They are filtered and preserved in 2% HNO_3.

6.7 Instrument Detection Limit (IDL) - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of seven to ten replicate measurements of the calibration blank signal at the selected analytic mass (es).

6.8 Internal Standard – Analytes added to samples and standard solutions in known concentrations to monitor for instrument drift and physical interferences.

6.9 Matrix Duplicate (MD, also called Laboratory Duplicates) – Another aliquot of the same sample taken in the laboratory and analyzed separately with identical procedures. This is used to evaluate the precision of the laboratory procedure.

6.10 Preparation Blank (PB) (also called Method Blank) - An aliquot of reagent water or other blank matrix that are treated exactly as a sample. It is used to determine whether the method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

6.11 Laboratory Fortified Blank (LFB or LCS) – An aliquot of reagent water to which known amount quantities of the method analytes are added in the laboratory. (CLPSL)- a soil purchased ERA standard which contains known amount quantities of the method analytes. The LFB/CLPSL is analyzed exactly like a sample. It is used to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

6.12 Matrix Spike (MS, also called Laboratory Fortified Sample Matrix, LFM) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample. It is used to determine whether the sample matrix contributes bias to the analytical results.

6.13 Linear Dynamic Range - The concentration range over which the instrument response to an analyte is linear. PE ELAN9000 has dual detection modes: Pulse Counting (PC) mode and Analog mode. PC mode works at lower concentration levels; Analog mode works at higher concentration levels. However, Analog mode is not used to avoid the routine Detector Cross Calibration. Therefore, the Linear Dynamic Range is limited by the highest count rates of PC mode. Table 22.3 lists the Linear Dynamic Range of each element (using PC mode). The Linear Dynamic Range is verified every three months.

6.14 Method Detection Limit (MDL) – the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. Refer to SOP *The Determination of Method Detection Limits* for the determination of MDL. MDL

is determined annually for water samples. The MDLs for solid samples are determined annually.

6.15 Solid Sample – For the purpose of this method, a sample taken from material classified as soil, sediment or sludge.

6.16 Stock Standard Solution – A concentrated solution containing method analytes of known concentrations.

6.17 Total Recoverable Analyte – aqueous samples that have undergone a digestion procedure on an unfiltered acid preserved drinking water sample with a turbidity of < 1 NTU.

6.18 Tune Solution – A solution that is used to determine acceptable instrument performance monthly.

6.19 Water Sample – For the purpose of this method, a sample taken from one of the following sources: drinking, surface, ground, and storm runoff, industrial or domestic wastewater.

6.20 ICV/QCS - Initial Calibration Verification or Quality Control Standard solution. A multi-element standard of known concentrations prepared to verify instrument calibration. An external source different than the Calibration Standards is used to prepare this standard. The concentration of each element is at 20 µg/L for CLP or 25 µg/L for 200.8/6020.

6.21 ICSA and ICSAB- Interference Check Solutions. Solutions containing both interfering and analyte elements of known concentration that is used to verify background and interelement correction factors.

6.22 CCV - Continuing Calibration Verification. A multi-element standard, of known concentrations, prepared to monitor and verify the instrument's daily confirming performance.

6.23 Low Level Verification Standard (CRI) - A purchased (High Purity) multi-element standard at the lab's PQL. See Table 22.10 for concentrations.

7.0 INTERFERENCES

7.1 Isobaric Elemental Interferences are caused by isotopes of different elements that form singly or doubly charged ions of the same nominal mass-to-charge ratio and that cannot be resolved by the mass spectrometer in use. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest.

7.2 Abundance Sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. Abundance sensitivity depends on the resolution of the instrument. The spectrometer resolution is adjusted periodically to eliminate this interference by performing a mass tune monthly.

7.3 Isobaric Polyatomic Ion Interferences are present when more than one atom combine to form an ion that have the same nominal mass-to-charge ratio as the isotope of interest. This interference highly depends on sample matrix. Masses are chosen to minimize these and when not possible they are mathematically corrected for.

7.4 Physical Interferences are associated with physical process, including the transportation, handling and the nebulization of the sample. These interferences may result in differences between instrument response for the sample and the calibration standards. To minimize these interferences, dissolved solids should not exceed 0.2% (v/v). Nitrile gloves are to be used. Powdered latex gloves contain large amounts of zinc, copper and cadmium. In addition, peristaltic pump, mass flow controller, and Internal Standards are used.

7.5 Memory Interferences may occur when the isotopes of elements in a previous sample contribute to the signals measured in a new sample. Flushing the system with rinsing blank for a sufficient time eliminates the memory effects.

8.0 SAFETY

8.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.2. Because this method uses acids and metals with toxic natures, safety glasses, lab coats and gloves must be worn to minimize exposure. Care must be taken in the handling and disposing of all standards and samples.

8.3. The torch produces intense ultraviolet light and must not be viewed without protection.

8.4. The matrix of standards and samples consists of Nitric Acid. Gloves should be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care should be taken when dispensing concentrated acids. Concentrated acids should be dispensed only in the fume hood.

8.5. PRIMARY MATERIALS USED

8.5.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1 The Perkin Elmer Elan 9000 ICP-MS

9.2 Class 100 clean hood. This clean hood is used for sample dilution and standard preparation, etc.

9.3 Glass class A volumetric flasks in various sizes from 50 mL to 2000 mL. These flasks are used for standard preparation. Volumetric flasks are labeled and are used only for preparing corresponding solutions.

9.4 Polyethylene or Teflon bottles are used for storing prepared solutions.

9.5 Adjustable Eppendorfs in various sizes from 0.02 mL to 10 mL. These are used for standard and sample preparation. The Eppendorfs are verified using an analytical balance on a weekly basis. They are calibrated on a quarterly basis. A logbook of the calibration results is kept as a record. At least one Eppendorf in each of the following ranges are used:

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- 20 µL
- 50 µL → 200 µL
- 100 µL → 1000 µL
- 500 µL → 2500 µL
- 2000 µL → 10000 µL

9.6 Disposal polypropylene pipette tips for the adjustable Eppendorfs in various sizes.

9.7 Disposable 17×100 mm-polypropylene culture tubes used in the autosampler to hold the samples.

9.8 Spare parts for the PE Elan 9000

- Sampling cones
- Skimmer cones
- Nebulizers
- Torches
- Spray chambers
- BLK/BLK pump tubing
- Lens
- White/BLK rinse pump tubing
- Autosampler sample probes
- Load coil
- O-Rings
- Injector tips

10.0 REAGENTS AND STANDARDS

10.1 All standards and samples are prepared using 18-MΩ cm^{-1} ELGA water. The metals lab has an ELGA water system attached to a deionized water system. The ELGA water is monitored daily and maintenance is performed as needed.

10.2 All standards are prepared with Class A volumetric flasks, and calibrated adjustable Eppendorfs.

10.3 All standards and samples are prepared with Trace Metals grade HNO_3. Reagent grade HNO_3 is only used for routine glassware cleaning.

10.4 All the standards are matrix matched to the sample digestates.

10.4.1 2% HNO_3 for aqueous samples

10.4.2 1% HNO_3 and 0.5% HCl for soil samples.

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10.5 All stock solutions are certified and the certifications are kept on record. All stock solutions are logged into an incoming Chemical History Logbook that is stored in the lab.

10.5.1 The Calibration Standards and Tuning Standards are prepared from multielement stock solutions purchased from High Purity. Stock Internal Standard and Stock Tuning Standard are prepared from single-element stock standards purchased from Ultra Scientific. The Quality Control Sample (QCS) used for the ICV is prepared from a stock solutions purchased from CPI.

10.5.2 Stock Internal Standard is prepared by adding 12.5 ml of Sc, In, 1 ml Y (10,000µg/ml) and Tb (1000 mg/L) individual standards to a 100 mL volumetric flask half filled with 2% nitric solution. Bring up to final volume with 2% nitric solution (see 10.6). This solution contains 100 mg/L of each element.

10.5.3 Stock Tuning Standard is prepared by adding 1.0ml Rh, 1.0ml In, 1.0mlU, .4ml Be, 4ml Li, .1ml Mg, .1ml Co, .1ml Pb to a 1000 mL volumetric flask of blank solution. Bring up to final volume with blank rinse. This solution contains 1ppm of Rh, In, U, Mg, Co, Pb and 4ppm of Li and Be in 2% HNO_3.

10.5.4 Autolens Stock solution is prepared by adding .04ml of Be, Co,.4ml In,.4ml U to 100 ml volumetric of blank solution.

10.5.5 Dual Detector solution: 600ppb of Be, and 200ppb of Mg, Cu, Rh, Cd, and Pb

10.6 Reagent Blank consists of ELGA water that contains 2% HNO_3 for aqueous samples or 1% HNO_3 and 0.5% HCl for solid samples. The Reagent Blank is used for the following:

- Calibration Blank
- ICB
- CCB
- Prep Blanks
- Sample dilutions

The Instrument Rinse Blank consists of ELGA water that contains 2% HNO_3 for aqueous samples or 1% HNO_3 and 0.5% HCl for solid samples.

10.6.1 The Instrument Rinse and reagent blanks for water samples are prepared in a 25-liter plastic carboy. Use a 500 mL graduated cylinder to add the acids. The rinse blank is prepared by adding 500 mL concentrated HNO_3 to the carboy that is half filled with ELGA water. Fill the carboy to the 25-liter mark with ELGA water.

10.6.2 For soil analysis, prepare the Rinse Blank as above only this time use 250 mL of HNO_3 and 125 mL of HCl to the carboy. Be extremely careful when handling

concentrated acids in these amounts (work in the fume hood wearing lab coat, gloves and safety glasses).

10.7 The following calibration standards are to be prepared weekly or as needed:

- Std1 (10 µg/L)
- Std2 (25 µg/L)
- Std3 (50 µg/L)

These standards and solutions are prepared from High Purity 10 mg/L stock standards: ICPMS-CAL1.

10.7.1 Std1 is prepared by adding 20mL of STD3 to 100mL volumetric flask and brought up to volume with blank solution. See Table 22.6 for concentrations of elements in Std1.

10.7.2 Std2 is prepared by adding 250mL of STD3 to 500mL volumetric flask and brought up to volume with blank solution. See Table 22.6 for concentrations of elements in Std2.

10.7.3 Std3 is prepared in a 500-mL volumetric flask half filled with 2% nitric solution. Add 2.5mL ICPMS-CAL1 stock (High purity, 10mg/L). Bring up to final volume with 2% nitric solution. See Table 22.6 for concentrations of elements in Std3.

10.8 Working Tuning Solution and Working Internal Standard are to be prepared bi-weekly or when needed.

10.8.1 The Working Tuning Solution is prepared by filling a 200 mL volumetric flask half filled with the reagent blank. Add 2 mL of stock tuning solution. Bring up to final volume with reagent blank. This solution contains 10 µg/L of Mg, Co, In, Pb, U, Rh and 40ug/L of Be, Li. When analyzing aqueous samples the reagent blank used is 2% HNO_3 and when analyzing soils the reagent blank used is 1% $HN0_3$ and .5%HCL.

10.8.2 The Working Internal Standard is prepared by filling a 2000 mL volumetric flask half filled with reagent blank. Add .250 ml of the 100 mg/mL Stock Internal Standard. Bring up to final volume with reagent blank. This solution contains 12.5 µg/L of Sc, In, Tb and 250ug/L of Y in 2% HNO_3 for aqueous samples, in solid samples the acid concentration is 1%HNO_3 and .5%HCL. The internal standard is then transferred to a 2000 mL Teflon bottle.

10.9 ICV is prepared from "CPI" stock standard and 1000µg/ml Sn: The ICV is used for the verification of the calibration.

10.9.1 ICV is prepared by filling a 200 mL volumetric flask half full of blank rinse. Add 0.5 ml of ICPMS-QCS1 and 0.5 ml of 1000µg/ml Sn. Bring up to volume with 2% nitric blank.

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This solution contains 25 µg/L of each analyte.

10.9.1.1 For CLP method the ICV will be at 20 µg/L. The procedure is the same as above except the volumes of ICPMS-QCS1 and 1000µg/ml Sn are at 0.4 ml each.

10.10 Interference Check Solutions (ICSA and ICSAB) are prepared from High Purity stock ICSA and ICSAB solutions. Fill a 200 ml volumetric flask half way with blank rinse and add 40 mls of the ICSA to the flask. Fill the flask with blank rinse to the mark. This is a 1:5 dilution of the stock solutions. Repeat this same procedure to prepare the ICSAB solution.

10.11 The low level verification check standard, CRI, is a purchased custom blend stock standard from Ultra Scientific at the CRQL (contract required quantitation limit) stated in the CLP method. For all other methods the low level verification standard is purchased from High Purity at our laboratory PQL and analyzed immediately following the ICB. See table 22.10 for concentrations.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 The maximum holding time for metals is 180 days from sample collection. Aqueous samples are preserved with nitric acid to a pH < 2. Soil samples do not require additional preservation.

11.2 For CLP, AFCEE and USACE samples, all samples are refrigerated at 4 degrees C +/-2 degrees C from the time of collection to digestion. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

11.3 For non-CLP samples, the aqueous samples are maintained at ambient temperature both before and after digestion. Non-Aqueous samples are refrigerated at 4 degrees C +/-2 degrees C prior to digestion and digestates are maintained at ambient temperature.

11.4 The digestion lab prepares the total metals digestates and the soluble metals digestates. The samples are forwarded to the instrumental lab after digestion. When analyses are complete, the digestions are placed in a main sample storage area. The main storage area is located near the digestion lab. The digestions are stored for 6 months before they are finally disposed of.

11.4.1 **Controlled Access Storage**: CLP, AFCEE and USACE samples require controlled access storage with strict Chain-of –Custody procedures. Digestates for these samples are obtained from and returned to the cooler custodian. The custodian maintains both the original samples and the digestates in the locked controlled access storage cooler.

11.4.1.1 The original samples are kept for 60 days following delivery of the final report package.

11.4.1. Digestates are maintained for 365 days for CLP samples; 60 days for AFCEE and USACE samples.

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12.0 QUALITY CONTROL

Section 12.1 to Section 12.11 are required by Method 200.8, Method 6020 and CLP (ILMO5.2)

12.1 Overview: This section provides details of the quality control used to determine if data are useable or not. If data are unusable, the samples must be redigested and/or reanalyzed depending on the situation. For details on how an actual analytical run is laid out see Section 14.0 - Procedures. For details on any of the calculations which are required in this section see Section 15.0 - Calculations.

12.2 For the analysis of samples containing higher level of Ag such that the final analysis solution contains > 100 µg/L silver, sample size needs to be reduced, and a redigestion is required.

12.3 All containers used are made of polypropylene, polyethylene or Teflon. The preparation of standard solutions and the dilution of samples are performed in clean hood to avoid contamination.

12.4 Standards - Working Calibration Standards and Quality Control Sample are prepared weekly or when needed using Class A volumetric flasks. The standards are logged into the standards logbook to insure traceability. Stock Standards are purchased as often as necessary to insure a fresh source.

12.5 Instrument Calibration - The instrument is calibrated for each analytical run using simple linear calibration. All relevant information is printed for reference. A Blank and 3 levels of standards are used to calibrate each element. A linear plot of each element is produced. The correlation for each element must be 0.995 or greater. If the correlation is less than 0.995 for a particular element, then the data for that element may not be used from that particular analytical run. A non-weighted least squares regression calibration model is used to prepare the correlation coefficient.

12.6 ICV - The ICV uses the QCS standard solution. It is analyzed immediately after calibration. The measured values must be within ±10% of the true values. If the ICV is outside the control limits for an element, the instrument must be recalibrated or that element cannot be used from that analytical run. See Table 22.7 for the true values of the ICV.

12.7 CCV - The CCV uses the Calibration Standard 2 solution (25 µg/L). It is analyzed after every ten samples and at the end of the analytical run. The measured values must be within ±10% of the true values. If the CCV is outside the control limits for an element, the instrument must be recalibrated, the previous ten samples must be reanalyzed for that element, or that element cannot be used from that analytical run.

12.8 ICB and CCB's - After analyzing the ICV, analyze an ICB. After analyzing each CCV, analyze a CCB. Concentrations in the ICB and CCB must be less than three times the IDL.

12.9 Preparation Blank (PB, also called Method Blank, MB) should be processed one in every twenty samples and is prepared in the digestion lab. The acceptance criteria, is that the Prep Blank must be less than three times the IDL.

12.9.1 For Method 200.8, if the PB value is ≥10% of the sample concentration or is 2.2 times the

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MDL (whichever is greater), the PB and all associated samples must be redigested.

12.10 Laboratory Fortified Blank (LFB) should be processed one in every twenty samples. The digestion lab prepares an LFB for Total Recoverable Waters, Dissolved Waters and Soil samples. The spiked concentration is 20 ppb for all elements. .

12.11 Matrix Spike (MS) should be processed one in every ten samples. The digestion lab prepares MS for Total Recoverable Water samples and Solid samples. The instrumental lab needs to prepare MS for Soluble Water samples: add 50uL of ICPMS-CAL1 to 10 mL of a duplicate sample. Table 22.8 lists the concentration of each element in the final analysis solution.

Section 12.12 to Section 12.14 are required by Method 6020 and CLP (ILMO5.2).

12.12 Matrix Duplicate (MD) - A matrix duplicate is performed on the sample that has a matrix spike at a frequency of one per ten samples. A control limit of 20% relative percent difference (RPD) should not be exceeded for analyte values greater than 100 times the IDL. If the RPD is outside the control limits for an element, then all the samples for that element must be redigested or/and reanalyzed.

12.13 Post Spike (PS) - A post digestion spike may be performed on the sample that has a matrix spike. The spike recovery must agree within ±25%. If the post spike for an element is outside the control limits, the sample must be diluted and reanalyzed. Results must agree to within 10% of the original sample if the concentration of the analyte in the diluted sample is at least 20 times higher than the MDL. Post Spike is NOT performed routinely.

12.14 Serial Dilution (L) - A five fold dilution is performed on the sample that has a matrix spike at a frequency of at least one per twenty samples. If the concentrations of the analytes in the diluted sample are at least 20 times higher than the MDL, the results must be within ±10% of the original sample. If not, an interference effect is suspected.

Section 12.15 is a requirement for CLP (ILMO5.2); 12.18 is for CLP (ILMO5.2), AFCEE and 6020.

12.15 To verify linearity near the CRQL, a CRI must be analyzed at a frequency of not less than once per 20 samples. The initial CRI is immediately followed by the ICSA and ICSAB. If the percent recovery of the CRI falls outside the control limits of 70-130% (50-150% for Cobalt, Zinc and Manganese). The CRI will be reanalyzed. If the value is out of range again the instrument will be recalibrated.

Section 12.16 is a requirement for AFCEE

12.16 To verify linearity near the CRQL, a CRI must be analyzed at a frequency of not less than once per 20 samples. The initial CRI is immediately followed by the ICSA and ICSAB. If the percent recovery of the CRI falls outside the control limits of 50-150%. The CRI will be reanalyzed. If the value is out of range again the instrument will be recalibrated.

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Section 12.17 is a requirement for USACE

12.17 Low Level Verification Standard – It is verified after the ICB and followed by the ICSA and ICSAB. The criteria is, ±20% of the true value. If the criteria is not met, this standard will be reanalyzed. If the values are out of control again, the instrument will be recalibrated.

12.18 The ICSA and ICSAB interference checks should fall between ±20% of the true values. If not, the analysis should be terminated and recalibration is necessary. The ICSA and ICSAB are verified following the ICB for 6020. They follow the CRI for CLP and AFCEE samples. They follow the Low Level Verification Standard for USACE samples. It is verified once every 12 hours.

13.0 CALIBRATION AND STANDARDIZATION

13.1 This section is included with section 14.0

14.0 PROCEDURES

14.1 The following is a daily checklist for the operation of PE Elan 9000. This is only a summary of the basic steps. Some steps are followed by a reference for further information.

- Check oil in the roughing and backing pump.
- Empty the drain waste, if necessary. Section 14.2
- Fill the autosampler rinse, if necessary. Section 14.3
- Prepare and refill the Working Internal Standard, if necessary. Section 10.8.2
- Change the pump tubings if necessary.
- Change the torch when necessary.
- Clean the spray chamber when necessary.
- Change the sampling cone and skimmer cone when necessary.
- Refill the water tank in the Water Recirculator, if necessary.
- Open the argon valve on the wall next to the door.
- Start up the instrument. Section 14.4.
- Prepare the Working Calibration Standards and QC samples. Section 10.0.
- Open method, Autosampler table and dataset
- Type in an analytical run. Section 14.6.
- Tune the instrument if necessary. Section 14.7.
- Perform Daily Performance Check. Section 14.8
- Place the standards and QC samples in the autosampler.
- Prepare the samples and place them into the autosampler.
- Start the analysis. Section 14.4
- When the analysis is complete, view the printed data.
- Empty the samples and standards into the nitric acid waste.
- Perform a validation of the data. Section 14.10.

14.2 Emptying the waste: the acid digestates must be disposed of properly.

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14.3 Filling the Autosampler Rinse:

- Pull the tubing from the 20-liter rinse container.
- Prepare the rinse blank according to Section 10.6.1
- Place the tubing into the rinse container and tighten lid.

14.4 Start up the instrument

14.5 The instrument should always be left on *pumping* mode. This is required to maintain the vacuum. If the instrument is not being used on a given day, it is only necessary to turn the computer, monitor, and printer off.

14.6 Typing in an Analytical Run - Each analytical run must be recorded in the analytical run log. Each analytical run is entered in the following format:

```
Blank
Std1
Std2
Std3
ICV
ICB
CRI
ICSA
ICSAB
CCV1
CCB1
↑
10 samples
↓
CCV2
CCB2
↑
7 samples
↓
CRI (only in ASP/CLP(ILMO5.2)/AFCEE)
ICSA
ICSAB
CCV3
CCB3
↑
10 samples
↓
CCV4
CCB4
```

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Run a CCV and CCB after every 10 samples and at the end of the analytical run. For CLP (ILMO5.2) and AFCEE a CRI, ICSA, ICSAB are run every 17 samples followed by a CCV/CCB.

14.7 Mass Calibration

Open STL Tuning workspace. Make sure the probe is in the Working Tuning Solution and click on *OK*. The results will then be displayed on the screen. Perkin Elmer specs require the peak width (AMU) to be within .05 of .65 AMU. Adjust the AMU by increasing or decreasing the DAC value and recalibrating each time. Save updated tune, print copy, and file in Tuning notebook. Processing department also receives copy.

14.8 Daily Performance Check

Open Daily performance workspace. Aspirate Perkin Elmer Stock Daily performance solution for 45 seconds. Click on "Run sample". Follow Perkin Elmer minimum requirements for Daily performance criteria. Five replicates are performed.

14.9 If Daily performance is acceptable, the run can be started. Call up correct method and go to sample table. Fill in sample ID column with batch digestion ID's. Enter method in method column, with DL if required and method. Highlight samples to run. Set up report table for data to go to printer and file. Start the autosampler sequence.

14.10 Validation of the Data. When the analytical run is complete, the data must be checked for compliance with the method. Using Section 9 - Quality Control - check all the quality control samples (ICV, ICB, CCVs, CCBs, ICSA, ICSAB, PB, LFB, and MS) for compliance. If a quality control sample falls outside the required limits for an element, that element must be reanalyzed on another analytical run.

14.11 For all sample analysis three replicate integrations are performed, the average of the three replicates is used for data reporting.

15.0 CALCULATIONS

15.1 Refer to Section 12.0 to determine if data are valid for each element. Any sample reading over the linear range must be diluted.

15.1.1 USACE samples over the highest standard concentration must be diluted.

15.2 The lowest concentrations that can be accurately quantified and reported without qualification are listed in Table 22.1 as the lab's PQLs.

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15.3 For data values less than ten, two significant figures are used for reporting concentrations. For data values greater than or equal to ten, three significant figures are used.

15.4 Aqueous samples:

Sample Concentration (mg/L)= CxV1xD/V2
Where C = Concentration in sample
D = Dilution factor
V = Final volume in Liters
V2 = Initial volume of sample in Liters

15.4.1 Wet-weight basis:

Sample Concentration (mg/kg)= $C \times V/W$
Where: C = concentration in extract (mg/L)
V = Volume of the digestate (L, 50 mL = 0.05 L)
W = Weight of sample aliquot (not dried) extracted (g × 0.001 = kg)

15.4.2 To report percent solids in solid samples, calculate as follows:

% solid (S) = $DW/WW \times 100$
Where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

15.4.3 Dry-weight basis:

Sample Concentration (mg/kg)= $(C \times V)/(W \times S)$, *or*
Sample Concentration (mg/kg)= $C \times V/WW$

15.5 The following calculations are illustrated:

15.5.1 Matrix Spike calculation

The formula for calculating the post spike recovery is:

Recovery (%) = $(S_2-S_1)/SA \times 100$
Where,
S_2 = the spiked sample reading
S_1 = the sample reading
SA = the spike added

Sample Calculations:

A base sample gave a reading of 50.0 µg/L. The sample MS was spiked with 50 µg/L and gave a reading of 95 µg/L. The recovery is *(95-50.0)/50×100%=90%*

15.5.2 Matrix Duplicate calculation

The formula for calculating the relative percent difference (RPD) is:

$RPD\ (\%) = 2\times|D_1-D_2|/(D_1+D_2)\times100$
Where,
D_1 = first sample value
D_2 = second sample value

Sample Calculations:

A sample gave a reading of 50.0 µg/L. The MD gave a reading of 40 µg/L. The RPD is *2×|50-40|/(50+40) ×100%=22%*

16.0 METHOD PERFORMANCE

16.1 Extensive quality control is used to insure compliance with Method 200.8, Method 6020, CLP (ILMO4.1 and ILMO5.2), and AFCEE protocols.

16.2 IDLS, LDRS, and MDLS are listed as tables in this SOP. The current LDR values are saved as check values on the instrument.

16.3 All Certificates of Analysis for the stock solutions are kept on file in the laboratory.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 QC Related Activities

Activity	Frequency
Daily Performance check	Each day, onset of analysis
Calibration	Each day, onset of analysis
ICV (LCS)	Immediately after calibration
ICB	After ICV
ICSA	after ICB and one per 12 hours. (1 per 20 samples CLP and AFCEE)
ICSAB	after ICSA and one per 12 hours. (1 per 20 samples CLP and AFCEE)
CRI	after ICB and one per 20 samples for CLP and AFCEE
Low level verification standard	after ICB in place of CRI for USACE

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Laboratory Fortified Blank	one per 20 samples
Preparation Blank	one per 20 samples
Matrix Spike	one per 20 samples
Matrix Spike Duplicate	one per 20 samples
Matrix Duplicate	one per 20 samples, Method 6020 and CLP(client assigned)
Serial Dilution	one per 20 samples
Post Digestion Spike	one per 20 samples, Method 6020, not routinely analyzed
CCV	one per 10 samples
CCB	after CCV

17.2 Acceptance criteria:

17.2.1 Internal standards in samples must be between 60 and 125% of the intensity in the calibration blank for method 200.8. For method 6020 the internal standard in samples must be between 30 and 120% of the intensity of the calibration blank.

17.2.2 ICV limits are 90 to 110 % of the true values. CCVs are 90% to 110% of the true values.

17.2.3 CCB and Preparation Blank should be less than three times the IDL.

17.2.4 Matrix Spike and Post Digestion Spike should recover with 75 to 125 % of the true value for Method 6020 and CLP. Matrix Spike and Post Digestion Spike should recover with 70 to 130% for Method 200.8.

17.2.4.1 Matrix Spike should recover with 80-120% of the true value for AFCEE. Post Digestion Spike should recover with 75-125% of the true value for AFCEE.

17.2.5 Lab Fortified Blank (LFB) must recover within 85 to 115 % of the true value for Method 200.8. Lab Fortified Blank (LFB) must recover within 80 to 120% for Method 6020 and for CLP (ILMO5.2). If the LFB does not meet these criteria, the associated samples must be re-digested and/or reanalyzed.

17.2.6 RPD for Matrix Duplicate should be lower than 20% if the analyte values are greater than 100 times the IDL.

17.2.7 Serial Dilution: If the concentrations of the analytes in the diluted sample are at least 20 times higher than the MDL, the results must be within ±10% of the original sample. If not, an interference effect is suspected.

17.2.8 Low Level Verification Standard must recover within 80% to 120% of the true values for USACE samples.

17.2.9 CRI recoveries for CLP must be 70% to 130% for all analytes except for Cobalt,

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Manganese, and Zinc, which is 50% to 150%. For AFCEE, the analytes must recover within 50% to 150% of the true values.

17.2.10 The ICSA and ICSAB requirements:

17.2.10.1. ICSA: For the analytes included in this solution, the recoveries must be within ±20% of the true values.

17.2.10.2 ICSAB: All analyte recoveries must be within ±20% of the true values.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1. If calibration fails, ICV and /or ICB are out of control limit, the analysis procedure must be terminated, the problems must be solved, and re-calibration must be started over.

18.2. If CCV and/or CCB fail, affected analytes in the 10 samples before and after that CCV/CCB pair must be reanalyzed.

18.3. If LFB or LCS fall outside of quality control acceptance range, the whole digestion batch must be reanalyzed. If the reanalysis still fails, that whole digestion batch must be redigested.

18.4. If Method Blank has detections > 3X IDL, but samples do not contain that analyte higher than the reporting limit or samples contain that analyte higher than 10 times the Method Blank, the data is usable and reportable. Otherwise, the digestion batch must be reprepared for that analyte.

18.4.1. For Method 200.8, if the PB value is $\geq$10% of the sample concentration or is 2.2 times the MDL (whichever is greater), the PB and all associated samples must be redigested.

18.5. If the RPD for the SD or MD is out of control limits, the data should be reviewed to determine cause. If lab error is suspected, reanalysis or redigestion should be performed. However, sample non-homogeneity may result in poor precision.

18.6. If the Post Spike or Serial Dilution fail: the serial dilutions fails to agree within 10% of the original analysis and/or the post spike fails to meet the 75-125% criteria matrix interference should be suspected and will be described in the final report narrative.

18.7. If LCS, Post Spike and Serial Dilution are within QC limits but the MS fails, matrix interference can be assumed and no further corrective action is necessary.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 Contingencies for unacceptable data will have to be evaluated on an individual basis. Corrective

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action will be prescribed accordingly. In the event that acceptable data can not be obtained, a Job Exception Form must be filed with the Project Manager and the client will be notified.

20.0 WASTE MANAGEMENT / POLLUTION PREVENTION

20.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

20.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Acid waste consisting of sample and rinse solution. All waste generated by the PE ELAN9000 and all samples used contain nitric acid (and sometimes hydrochloric acid) and should be disposed of in the "AN" waste containers.

21.0 REFERENCES

21.1 Method 6020 - Revision 0; September 1994

21.2 Method 200.8 – Revision 5.4; May 1994

21.3 Method CLP(ILMO5.2); December 2001

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Elements analyzed by Method 200.8 and Method 6020, and Laboratory Practical Quantitation Limits
22.2. Analytical isotopes and additional masses that must be monitored
22.3. Achievable Instrumental Detection Limits (IDLs)[a] and Linear Dynamic Ranges (LDRs)
22.4. Achievable Method Detection Limits (MDLs)
22.5. Stock Standard Solutions
22.6. Concentrations of each element in Stock Calibration Standard and Working Calibration Standards (in final samples).
22.7. Concentrations of each element in Quality Control Standard and Initial Verification Calibration solution
22.8. Concentrations of each element in Quality Control Standard and Initial Verification Calibration solution Concentration of each element in ICPMS-CAL1, and spiked final concentration of each element in LFB, MS, and PS. (200.8 and 6020)
22.9. Concentrations of elements in stock and working Interference Check Solutions
22.10. Concentrations of Elements in CRI and Low Level Verification Standard

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22.11. Job Summary Sheet and Data Review Checklist

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 10.5.3: Updated concentrations of solutions used
23.2 Section 10.5.4: Updated concentrations of solutions used
23.3 Section 10.5.5: Updated Beryllium concentration to 600ppb
23.4 Section 10.7 : Updated wording of standard preparation to : weekly or as needed
23.5 Section 12.9.1: Added method 200.8 PB acceptance criteria
23.6 Section 18.4.1: Added method 200.8 PB acceptance criteria
23.5 Section 22.1: Updated job summary form

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Table 22.1 Elements analyzed by Method 200.8 and Method 6020, and Laboratory Practical Quantitation Limits

Elements	Symbol	Total Water RL (µg/L)	Solid RL (ug/kg)
Antimony	Sb	1.0	1000
Arsenic	As	1.0	1000
Barium	Ba	0.5	100
Beryllium	Be	0.5	100
Cadmium	Cd	0.2	100
Chromium	Cr	1.5	N/A
Cobalt	Co	0.2	50
Copper	Cu	3.0	500
Lead	Pb	0.5	100
Lithium	Li	0.5	200
Manganese	Mn	1.0	300
Molybdenum*	Mo	1.0	100
Nickel	Ni	1.0	200
Selenium*	Se	1.0	1000
Silver	Ag	0.5	50
Strontium	Sr	.8	400
Thallium	Tl	.2	100
Tin	Sn	2.0	300
Vanadium*	V	1.5	N/A
Zinc	Zn	20.0	2000.0

* These elements are not included in standard Method 6020

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Table 22.2 Analytical isotopes and additional masses that must be monitored

Elements	Isotope	Elements	Isotope
		Ni	**60**, 62
Sb	**121**, 123	Se*	77, **82**
As	**75**	Ag	**107**, 109
Ba	**135**, **137**	Tl	203, **205**
Be	**9**	V*	**51**
Cd	106,108, **111**, 114	Zn	**66**, 68
Cr	**52**, 53	Sn	**118**
Co	**59**	Mn	**55**
Cu	**63**, 65	Mo*	95, 97, **98**
Li	**7**	Sr	**88**
Pb	206, 207, **208**		

* These elements are not included in SW-846 Method 6020, but are included in STL Buffalo 6020 analysis.

Note: Isotopes used for ICP-MS determination are in bold font and are underlined

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Table 22.3 Achievable Instrumental Detection Limits (IDLs)[a] and Linear Dynamic Ranges (LDRs)

Element	Mass	IDL (µg/L)	LDR (µg/L)
Be	9	0.02	400
V	51	0.10	250
Cr	52	0.29	400
Mn	55	0.09	200
Co	59	0.01	300
Ni	60	0.03	400
Cu	63	0.03	350
Zn	66	.61	400
Zn	68	.61	400
As	75	0.09	400
Se	82	0.32	400
Mo	98	0.008	400
Ag	107	0.01	100
Cd	111	0.05	400
Sb	121	0.04	250
Ba	135	0.02	400
Tl	205	0.02	200
Pb	208	.28	300
Li	7	.12	300
Sn	118	.006	175
Sr	88	.012	100

[a] IDLs are determined from seven calibration blanks on a quarterly basis.

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Table 22.4 Achievable Method Detection Limits (MDLs)

Element	Mass	Total water (µg/L)	Solid (ug/kg)
Be	9	0.2	20
V	51	0.8	N/A
Cr	52	0.6	N/A
Mn	55	0.3	100
Co	59	.05	15
Ni	60	0.3	50
Cu	63/65	0.43	0.17
Zn	66/68	4.5	500
As	75	0.3	300
Se	82	0.5	300
Mo	98	0.3	30
Ag	107/109	0.1	15
Cd	111/114	0.1	30
Sb	121/123	0.3	300
Ba	135/137	0.2	20
Tl	203/205	0.1	30
Pb	208	0.1	30
Sr	88	0.3	100
Sn	118	0.1	100
Li	7	0.3	60

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Table 22.5 Stock Standard Solutions

STL, Buffalo ID	Manufacturer or Vendor	Catalogue #	Description	Concentration
ICPMS-CAL1	High Purity	SM-606-047	For calibration	All elements 10 mg/L
ICPMS-QCS1	CPI	P/N 4400-131904	For verification, contains all elements	All elements 10 mg/L
ICSA	High Purity	ICPMS-ICS Solution A	For checking interference correction	Table 17.9
ICSAB	High Purity	ICPMS-ICS Solution AB	For checking interference correction	Table 17.9
Be	Ultra	ICP-104	Tuning	1000 mg/L
Mg	Ultra	ICP-112	Tuning	1000 mg/L
Sc	Ultra	ICP-021	Internal standard	1000 mg/L
Co	Ultra	ICP-127	Tuning	1000 mg/L
In	Ultra	ICP-049	Tuning/ Internal standard	1000 mg/L
Ba	Ultra	ICP-156	Tuning	1000 mg/L
Tb	Ultra	ICP-065	Internal standard	1000 mg/L
Pb	Ultra	ICP-182	Tuning	1000 mg/L
U	Ultra	ICP-092	Tuning	1000 mg/L

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Table 22.6 Concentrations of each element in Stock Calibration Standard and Working Calibration Standards (in final samples).

Element	ICPMS-CAL1 (Stock) (µg/L)	Std1 (Working) (µg/L)	Std2 (Working) (µg/L) (CCV)	Std3 (Working) (µg/L)
Sb	10,000	10	25	50
As	10,000	10	25	50
Ba	10,000	10	25	50
Be	10,000	10	25	50
Cd	10,000	10	25	50
Cr	10,000	10	25	50
Co	10,000	10	25	50
Cu	10,000	10	25	50
Pb	10,000	10	25	50
Mn	10,000	10	25	50
Mo	10,000	10	25	50
Ni	10,000	10	25	50
Se	10,000	10	25	50
Ag	10,000	10	25	50
Tl	10,000	10	25	50
V	10,000	10	25	50
Sn	10,000	10	25	50
Sr	10,000	10	25	50
Li	10,000	10	25	50
Zn	10,000	10	25	50

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Table 22.7 Concentrations of each element in Quality Control Standard and Initial Verification Calibration solution

Element	ICPMS-QCS1 STOCK (µg/L)	ICV Final conc. In samples (µg/L)(6020 + 200.8 / CLP)
Li	10,000	25 / 20
Sb	10,000	25 / 20
As	10,000	25 / 20
Ba	10,000	25 / 20
Be	10,000	25 / 20
Cd	10,000	25 / 20
Cr	10,000	25 / 20
Co	10,000	25 / 20
Cu	10,000	25 / 20
Pb	10,000	25 / 20
Mn	10,000	25 / 20
Mo	10,000	25 / 20
Ni	10,000	25 / 20
Se	10,000	25 / 20
Ag	10,000	25 / 20
Tl	10,000	25 / 20
V	10,000	25 / 20
Zn	10,000	25 / 20
Sr	10,000	25/20
Sn	10,000	25 / 20

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Table 22.8 Concentration of each element in ICPMS-CAL1, and spiked final concentration of each element in LFB, MS, and PS. (200.8 and 6020)

Element	ICPMS-CAL1 (µg/L)	Water: Spiked Final Conc. In samples (µg/L)[a]	Soil: Spike for MS/PS (µg/kg)
Antimony (Sb)	10,000	20	2
Arsenic (As)	10,000	20	2
Barium (Ba)	10,000	20	2
Beryllium (Be)	10,000	20	2
Cadmium (Cd)	10,000	20	2
Chromium (Cr)	10,000	20	2
Cobalt (Co)	10,000	20	2
Copper (Cu)	10,000	20	2
Lead (Pb)	10,000	20	2
Manganese (Mn)	10,000	20	2
Molybdenum (Mo)	10,000	20	2
Nickel (Ni)	10,000	20	2
Selenium (Se)	10,000	20	2
Silver (Ag)	10,000	20	2
Thallium (Tl)	10,000	20	2
Vanadium (V)	10,000	20	2
Zinc (Zn)	10,000	20	2

[a] LFB, MS and PS use the same concentrations for CLP (ILMO5.2)

CLP spiking concentrations (MS/SD).

- Ba = 2000 µg/L
- Co, Mn, Ni, V and Zn = 500 µg/L
- Cu = 250 µg/L

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- Cr = 200 µg/L
- Sb = 100 µg/L
- Be, Cd, Ag, Tl = 50 µg/L
- As = 40 µg/L
- Pb = 20 µg/L
- Se = 10 µg/L

Table 22.9 Concentrations of elements in stock and working Interference Check Solutions

Element	Stock ICSA (mg/L)	Stock ICSAB (mg/L)	Working ICSA (µg/L)	Working ICSAB (µg/L)
Al	500	500	50,000	50,000
Ca	500	500	50,000	50,000
Fe	500	500	50,000	50,000
Mg	500	500	50,000	50,000
Na	500	500	50,000	50,000
P	500	500	50,000	50,000
K	500	500	50,000	50,000
S	500	500	50,000	50,000
C	1000	1000	100,000	100,000
Cl	3600	3600	360,000	360,000
Mo	10	10	1000	1000
Ti	10	10	1000	1000
As	0	0.1	0	20
Cd	0	0.05	0	10
Cr	0	0.1	0	20
Co	0	0.2	0	40
Cu	0	0.1	0	20

Mn	0	0.1	0	20
Ni	0	0.2	0	40
Ag	0	0.1	0	20
V	0	.2	0	40
Se	0	.1	0	20
Zn	0	0.1	0	20

Table 22.10 Estimated concentrations of Elements in CRI and Low Level Verification Standard

Element	CRI (ug/L)	USACE Low Level Standard (ug/L)
Sb	2.0	1.0
As	1.0	1.0
Ba	.1	0.5
Be	.7	0.5
Cd	.5	0.2
Cr	1.5	1.5
Co	.3	0.2
Cu	3.0	3.0
Pb	.6	0.5
Mn	1	1.0
Mo	1	1.0
Ni	1.0	0.5
Se	1	5.0
Ag	.5	0.5
Tl	.2	0.2
V	1.5	1.5
Sn	2	2.0
Zn	20	20.0
Sr	.8	0.8
Li	1	0.5

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TITLE: ICP-MS Analysis Method 200.8/6020/ CLP(ILMO5.2)/AFCEE

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22.11 Job Summary Sheet and Data Review Checklist

Date: 08/31/2006 STL Buffalo Page: 1
Time: 10:07:25 Metals Job Summary Rept: AM1500
Analysis Type: TOTAL METALS - ICP/MS (WATER)

Job No: 306-2471
Project/Task: NY54331GAP 43
Lab Due Date: 09/07/2006
Client Due Date: 09/16/2006

Test No	Description	QAPP	Protocol	Method	Mtx	Y/N	P	Holding Time	Holding Time	Prep Type	Unit Meas	Detect Limit Type	Detect Limit Value	Code	Aliquot	Report Conc	QC Limits	RPD
08AB0971 PROFICIENCY ICP-MS - T METAL - 200.																		
STA009747 AG - 200.8 SILVER - TOTAL - W RL=			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	0.50000	A00067	0.25 ML	10.00 UG/ML	(85-115)	20.0
STA009734 AS - 200.8 ARSENIC - TOTAL- W RL =			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009735 BA - 200.8 BARIUM - TOTAL - W RL=1.			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(85-115)	20.0
STA009736 BE - 200.8 BERYLLIUM -TOTAL -W RL =			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	0.70000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009737 CD - 200.8 CADMIUM - TOTAL W -RL =			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	0.50000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009740 CO - 200.8 COBALT- TOTAL -W - RL= 0			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	0.50000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009739 CR - 200.8 CHROMIUM - TOTAL -W - RL			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.50000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009741 CU - 200.8 COPPER - TOTAL -W-RL= 1.			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	3.00000	A00067	1.00 ML	10.00 UG/ML	(85-115)	20.0
STA009743 MN - 200.8 MANGANESE - TOTAL -W RL=			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009744 MO - 200.8 MOLYBDENUM - TOTAL -W RL			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009745 NI - 200.8 NICKEL- TOTAL - W RL =			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(85-115)	20.0
STA009742 PB - 200.8 LEAD - TOTAL -W-RL= 0.5			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009733 SB - 200.8 ANTIMONY -TOTAL -W RL =			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(85-115)	20.0
STA009746 SE - 200.8 SELENIUM - TOTAL -W- RL=			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.00000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA01402 SR - 200.8 STRONTIUM- TOTAL			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	0.80000	A00067	1.00 ML	10.00 UG/ML	(70-130)	20.0
STA009748 TL - 200.8 THALLIUM - TOTAL - W RL			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	0.20000	A00067	1.00 ML	10.00 UG/ML	(85-115)	20.0
STA009749 V - 200.8 VANADIUM - TOTAL - W RL			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	1.50000	A00067	1.00 ML	10.00 UG/ML	(85-115)	20.0
STA009750 ZN - 200.8 ZINC - TOTAL - W RL = 4			MCAWW	200.8	Water	N	S	0	180	180	D	UG/L	EQL	20.00000	A00067	1.00 ML	10.00 UG/ML	(85-115)	20.0

Comments: _____

( ) ICP digestates were used; spiked at: ____ mg/mL
( ) Samples were diluted to eliminate matrix effects
( ) Samples were diluted to avoid overrange

Criteria:

Initial Calibration/Second Source Criteria Met? Y / N
CCV/CCB Criteria Met? Y / N
Method Blank Criteria Met? Y / N
ICS Criteria Met? Y / N
MS/MD Criteria Met? Y / N

Analyst Approval: _____ Date: _____
Data Processor: _____ Date: _____
Data Reviewer: _____ Date: _____

STL BUFFALO
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TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

REVIEWED AND APPROVED BY:	SIGNATURE	DATE
Verl Preston, Quality Manager	Verl D. Preston	6/13/06
Christopher Spencer, Laboratory Director		6/13/06
Jennifer Pierce, Supervisor	Jennifer Pierce	6/13/06

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1.0 IDENTIFICATION OF TEST METHODS

1.1 This SOP is used for the determination of Dissolved (Soluble) and Total elements by Thermo Jarrell Ash ICAP 61E Trace Analyzer (referred to also as the Trace). This SOP is specific for methods (SW-846) 6010B, 200.7, and CLP.

2.0 APPLICABLE MATRIX

2.1 Soluble water samples and digestates of waters, TCLPs, total recoverables, soils, sludges, sediments, and other wastes.

3.0 REPORTING LIMIT

3.1 Tables 22.2 and 22.3 list achievable Instrumental Detection Limits (IDLs) and Method Detection Limits. The laboratory IDLs are updated quarterly and the MDLs are updated annually. The current IDLs and MDLs are maintained in the laboratory LIMs system.

3.2 The laboratory standard Practical Quantitation Limits (PQLs) are also listed in Tables 22.2 and 22.3. The standard laboratory PQLS remain static and are only changed if there is a major update to the analytical system.

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

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4.0 SCOPE AND APPLICATION

4.1 This SOP is specific for methods (SW-846) 6010B, 200.7, and CLP. This SOP discusses the procedures as they are performed at STL Buffalo. Table 22.15 summarizes the actual method criteria.

4.2 At STL Buffalo, there are two 61E Trace Analyzers. They are designated as Trace #1 and Trace #2.

4.3 Table 22.1 lists the elements that are analyzed on each Trace.

4.4 Tables 22.2 and 22.3 list the approximate instrument detection limits (IDL's) which can be achieved on each Trace. IDL's are recalculated quarterly or when a significant instrumentation change occurs.

4.5 Table 22.4 lists the wavelengths and typical background points used on each Trace.

4.6 The linear range is the concentration range over which the instrument response to an analyte is linear. Table 22.5 lists the approximate linear ranges of each Trace. Linear ranges are recalculated quarterly or when a significant instrumentation change occurs.

4.7 Interelement correction factors (IECs) are used to correct for interferences caused by spectral overlap of elemental lines. At STL, IECs are verified and calculated quarterly or when an instrumentation change occurs.

4.8 All samples, standards, and blanks are matrix matched to achieve an aqueous solution containing 6% HNO_3 and 5% HCl by volume.

5.0 SUMMARY OF TEST METHOD

5.1 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 radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission 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 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 interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

5.2 This SOP contains the procedures for the daily operation of the ICAP 61E Trace Analyzer. This SOP also contains procedures for calibration, standard and sample preparation, maintenance, data handling, and quality control. This SOP is based on methods 6010B (SW-846), 200.7 and CLP.

5.3 On a given day the normal steps in operating the Trace include:

- Perform any routine maintenance, if required.
- Instrument start-up and warm-up
- Preparation of standards. All standards and quality control standards are prepared from stock solutions, as needed (6-month expiration date.) The Calibration Standards are made every 3-7 days.
- Type-up a run to analyze. A run is simply a sequence of samples with all required quality control that is analyzed as a single unit.
- Set-up the autosampler.
- Prepare all the samples for analysis including the required spikes, serial dilutions, and other quality control samples.
- Analyze the samples.
- When the analysis is complete, check the data for compliance with 6010B, 200.7, AFCEE or CLP whichever is applicable.
- Log in compliant data.
- Dispose of samples and standards appropriately. Clean-up area.

5.4 If the instrument is not operating properly or requires any maintenance, refer to Section 14 for help with routine maintenance and troubleshooting.

6.0 DEFINITIONS

6.1 Trace - Abbreviation for Thermo Jarrell Ash ICAP 61E Trace Analyzer. The Trace ICP has a viewing angle along the long axis of the torch.

6.2 IECs - Interelement correction factors. Used to correct for interferences caused by spectral overlap of element lines. See Section 12.17.1 for procedures on determining IECs.

6.3 Linear Range - Also referred to as linear dynamic range. The linear range is the concentration range over which the instrument response to an analyte is linear. Refer to Section 12.17.3 for the determination of linear ranges.

6.4 IDL - Instrument detection limit. The IDL of an element is the lowest calculated concentration that the instrument can measure. See section 12.17.2 for procedures on determining IDLs.

6.5 MDL – Method Detection Limit. The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero.

6.6 PQL – Practical Quantitation Limit. The minimum amount of a substance that can be *quantitatively* measured with a specified degree of confidence and within *accuracy and precision guidelines*.

6.6 Calibration Standards - A series of solutions containing known amounts of each element with a matrix similar to samples. These solutions are used to calibrate the instrument.

6.7 ICL - The highest calibration standard re-run directly after calibrating the instrument.

6.8 ICV - Initial calibration verification, which must be from a source different from that of the calibration standard

6.9 ICB - Initial calibration blank.

6.10 ICSA - Interference check sample containing only high levels of Al, Fe, Ca, and Mg.

6.11 ICSAB - Interference check sample containing high levels of Al, Fe, Ca, and Mg, and low levels of all other elements that are analyzed by the Trace.

6.12 CCV - Continuing calibration verification.

6.13 CCB - Continuing calibration blank.

6.14 LCS - Laboratory control sample. A quality control sample containing known concentration of analytes that is taken through the entire digestion and analysis procedure.

6.15 Method Blank - A blank sample that is taken through each step of the analytical procedure, including the digestion procedure if it is used.

6.16 Method of Standard Addition - Involves the analysis of an unknown sample and the analysis of an unknown sample with a known amount of a standard added. This procedure may be used when matrix interference is suspected.

6.17 Calibration Blank - A blank solution containing 6% HNO_3 and 5% HCl for calibration.

6.18 Total Metals – The concentration determined on an unfiltered sample following vigorous digestion.

6.19 Soluble or Dissolved Metals – The concentration determined on a sample after passing through a 0.45 um membrane. Acidification and digestion are performed after filtration.

6.20 ELGA water – This is blank reagent water that is deionized, filtered, and has a resistivity of 18 $M\Omega cm^{-1}$.

6.21 Calibration Curvefit- This process allows individual element wavelengths to be standardized using an extended calibration with more standards to produce a more linear and precise response over a greater range. The calibration is then fitted to a curve, which can be resloped during the instrument standardization.

7.0 INTERFERENCES

7.1 There are four main types of interferences. They are - spectral, physical, chemical and memory interferences.

7.1.1 Spectral Interferences

These types of interferences are caused primarily from the overlap of elemental lines and background contributions. Interferences from spectral overlap are eliminated by the use of interelement correction factors. Interferences caused by background contributions are eliminated by the use of background correction. Table 22.3 lists the typical background points.

7.1.2 Physical Interferences

These types of interferences are caused by differences between the physical properties of standards and samples. The major source of these interferences is a high dissolved solids concentration in a sample. Physical interferences are minimized by using an internal standard, diluting the samples and/or performing the method of standard addition.

Additionally, high salt concentrations can cause a buildup of salt at the tip of the nebulizer. This effect is minimized on the Trace by use of an Argon Saturator and a Noordermeer V-Groove nebulizer designed for high dissolved solid use.

7.1.3 Chemical Interferences

These are generally caused by molecular compound formation, ionization effects, and solvent evaporization effects. These effects can be minimized by careful selection of the operating conditions, by buffering the sample, or by standard addition procedures. At STL Buffalo, buffer solution of $Li(NO_3)$ is added on-line to minimize the ionization effects of the high level of easily ionized elements such as K and Na.

7.1.4 Memory Interferences

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. To minimize memory effects, a rinse period of at least 60 seconds is used between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

7.2 The following tests may be performed to check for physical and chemical interferences. A serial dilution is performed on a representative sample from each sample batch. A post digestion spike is performed based upon client requirements. The sample batch does not exceed twenty samples.

7.2.1 Serial Dilution

A serial dilution (1:5) is performed on a representative sample of each matrix of each sample group. See Table 22.15 for recovery criteria for each method. If the element concentration is high enough, such that the analyte in the diluted sample is at least a factor of 10 above the IDL, the serial dilution must agree within ±10% of the original sample. If the serial dilution is outside the 10% limit, a chemical or

physical interference effect should be suspected.

7.2.2 Spike Addition

A post-spike is performed when required per method being analyzed. A representative sample within the sample group (client job) is spiked. Generally, the spike is performed on the same sample as the one on which the serial dilution is performed, unless there is limited volume. Spiking a sample consists of adding a specified amount of four separate spike solutions to the unknown sample. Each spike solution contains various elements of interest. See Table 22.15 for recovery criteria for each analyte.

7.2.2.1 The four spike solutions for Non-CLP samples are:

- **Spike 1 (Custom Inorganic Standard) Made by Ultra Scientific**
This ULTRAgrade ™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

ANALYTE	TRUE VALUE
Antimony	40.0 µg/mL
Arsenic	40.0 µg/mL
Beryllium	40.0 µg/mL
Cadmium	40.0 µg/mL
Chromium	40.0 µg/mL
Cobalt	40.0 µg/mL
Copper	40.0 µg/mL
Lead	40.0 µg/mL
Manganese	40.0 µg/mL
Molybdenum	40.0 µg/mL
Nickel	40.0 µg/mL
Selenium	40.0 µg/mL
Thallium	40.0 µg/mL
Vanadium	40.0 µg/mL
Zinc	40.0 µg/mL
Titanium	40.0 µg/mL
Calcium	2000.0 µg/mL
Iron	2000.0 µg/mL
Magnesium	2000.0 µg/mL

Matrix: 5% HNO_3 in water

All weights are traceable to NIST traceable weights CAT#ICUS-1370.

NOTE: These concentrations might be slightly different between different lots. Current concentrations may be found in the binder of the Certificates of Analysis. This NOTE is also applicable to Spike 2, Spike 3 and Spike 4.

- **Spike 2 (Custom Inorganic Standard) Made by Ultra Scientific**

This ULTRAgrade ™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

ANALYTE	TRUE VALUE
Barium	40.0 µg/mL
Boron	40.0 µg/mL
Aluminum	2000.0 µg/mL
Potassium	2000.0 µg/mL
Sodium	2000.0 µg/mL

Matrix: 5% HNO_3 in water

All weights are traceable to NIST traceable weights CAT# ICUS-574

- **Spike 3**

ANALYTE	TRUE VALUE
Silver	10 µg/mL

See 10.5.1 for preparation.

- **Spike 4**

ANALYTE	TRUE VALUE
Tin	40 µg/mL

See 10.5.2 for preparation.

Table 22.5 lists the final concentration of each element spiked.

To prepare a spike, add 0.05 mL of Spike 1, Spike 2, Spike 3 and Spike 4 to 9.80 mL of sample. Mix thoroughly and analyze.

7.2.2.2 The three spike solutions for CLP samples are:

CLP-1 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.1)	TRUE VALUE (5.2)
Aluminum	2000 µg/mL	2000 µg/mL
Barium	2000 µg/mL	2000 µg/mL
Beryllium	50.0 µg/mL	50.0 µg/mL
Chromium	200.0 µg/mL	200.0 µg/mL
Cobalt	500.0 µg/mL	500.0 µg/mL
Copper	250.0 µg/mL	250.0 µg/mL

Iron	1000 µg/mL	1000 µg/mL
Manganese	500.0 µg/mL	500.0 µg/mL
Nickel	500.0 µg/mL	500.0 µg/mL
Silver	50.0 µg/mL	50.0 µg/mL
Vanadium	500.0 µg/mL	500.0 µg/mL
Zinc	500.0 µg/mL	500.0 µg/mL

CLP-2 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.0)	TRUE VALUE (5.0)
Antimony	500.0 µg/mL	100.0 µg/mL

CLP-3 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.0)	TRUE VALUE (5.0)
Arsenic	2000 µg/mL	40 µg/mL
Cadmium	50 µg/mL	50 µg/mL
Thallium	2000 µg/mL	50 µg/mL
Selenium	2000 µg/mL	10 µg/mL
Lead	500 µg/mL	20 µg/mL

Refer to sample preparation SOPs for the preparation of matrix spikes for CLP samples.

The spike recovery criteria may be found in table 22.15.

8.0 SAFETY

8.1 Many of the metallic elements analyzed for in this method are known to be hazardous to health. Care should be taken in the handling and disposing of all standards and samples. See section 20.0 for procedures on the disposal of standard and sample waste.

8.2 The matrix of all ICP standards and samples is 6% HNO_3, 5% HCl by volume. Gloves should be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care should be taken when dispensing concentrated acids. Concentrated acids should be dispensed only in the fume hood.

8.3 The plasma emits strong UV light and is harmful to vision. **AVOID LOOKING DIRECTLY AT THE PLASMA.**

8.4 The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

8.5 PRIMARY MATERIALS USED

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The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1 Thermo Jarrell Ash ICAP 61E Trace Analyzer is equipped with an autosampler, computer, printer, and source of argon. There are two Trace Analyzers at STL Buffalo. They are designated as Trace #1 and Trace #2.

9.2 Volumetric flasks in various sizes from 50 mL to 1000 mL. These are used for standard preparation and sample dilution.

9.3 Eppendorfs in various sizes. These are used for standard and sample preparation. The Eppendorfs are verified using an analytical balance on a weekly basis. They are calibrated on a quarterly basis along with the repipettors. A logbook of the calibration results is kept as a record. At least one

Eppendorf in each of the following ranges are used:

10 µL → 100 µL
50 µL → 200 µL
50 µL → 250 µL
100 µL → 1000 µL
500 µL → 2500 µL
2000 µL → 10000 µL

9.4 Disposable polypropylene pipette tips for the Eppendorfs in various sizes.

9.5 Disposable 17x100 mm polypropylene culture tubes used in the autosampler as the sample containers.

9.6 28 mL Nalgene brand disposable sample vials used to hold standards and quality control samples in the autosampler.

9.7 Spare parts for the Trace:
- nebulizers
- torches
- spray chambers
- platens

9.8 Red/Red/Red pump tubing (TJA #13017701)

9.9 Orange/Green/Orange pump tubing

9.10 Orange/Orange/Orange pump tubing

9.11 Internal Standard tubing mixing kit

9.12 Autosampler sample probes (TJA #4097-30)

9.13 500 mL graduated cylinder

9.14 Para-ilm

9.15 Repipettors for acids and dilutions.

10.0 REAGENTS AND STANDARDS

10.1 All standards and samples are prepared such that the matrices are matched.

10.1.1 All standards and samples are prepared using 18 $M\Omega cm^{-1}$ ELGA water. The metals lab has an ELGA water system attached to a deionized water system. The ELGA water is

monitored daily by the Wet Chemistry department and maintenance is performed as needed.

10.1.2 All standards are prepared with volumetric flasks, and calibrated Eppendorfs.

10.1.3 All standards and samples are prepared with Trace Metals Grade Nitric and Hydrochloric Acids.

10.1.4 All the standards and samples are prepared in the same matrix containing 6%HNO_3 and 5% HCl by volume.

10.1.5 Standards are prepared as needed, every 3 to 7 days for Calibration Standards.

10.2 Table 22.7 lists all the reagents and stock solutions that are purchased as starting materials. All stock solutions are certified and the certifications are kept for a record. All stock solutions are logged into an incoming logbook that is stored in the lab.

The multi-element calibration standards and other solutions required (except the quality control sample used for ICVs and CCVs) are prepared from stock solutions purchased from ULTRA SCIENTIFIC. The quality control sample used for ICVs and CCVs are prepared from stock solutions purchased from HIGH PURITY. The use of two vendors ensures a second source verification of standards. The ionization buffer $LiNO_3$ is purchased as a solid from MALLINCKRODT.

10.3 There are two types of solutions that are prepared from the purchased stock standards. They are prepared stock solutions and the working standards. Prepared stock solutions are used as intermediate standards for preparing the working standards. Prepared stock solutions are recorded in the standards logbook. They expire in six months or when the original starting stock standards expire, whichever is first. Prepared stock solutions are labeled with their name, the preparation date, the expiration date, and the initials of the analyst preparing the solution.

The working standards are prepared from the purchased stock standards and the prepared stock solutions. The working standards are also recorded in the standards logbook.

Solutions prepared by the analyst are recorded in the standards logbook. The following information is recorded:

- Name or concentration of the solution
- Date prepared
- Initials of analyst preparing the solution
- The manufacturer of the starting stock solution
- The lot number of the starting stock solution
- The name or concentration of the starting stock solution
- The volume of the starting stock solution used
- The final volume of the solution being prepared

- The source of the HNO_3
- The source of the HCl

10.4 Blank solutions contain 6% HNO_3 and 5% HCl in ELGA water. The blank solution is used for the following:

- Calibration blank
- ICB
- CCBs
- Sample dilutions

10.4.1 The Blank Solution is prepared by adding 1200 mL concentrated HNO_3 and 1000 mL concentrated HCl to a 20 liter plastic carboy half filled with ELGA water. Bring up to volume with ELGA water. This procedure may be scaled up or down. Use a 500 mL graduated cylinder to add the acids. Be extremely careful when handling conc. acids in these amounts (work in the fume hood wearing lab coat, gloves and safety glasses).

10.4.2 The instrument rinse is prepared in a 20-liter plastic carboy. The rinse blank is prepared by adding 1200 mL of concentrated HNO_3 to the carboy half filled with ELGA water. Fill the carboy to the 20-liter mark with ELGA water.

10.5 The following stock solutions are prepared:

- Spike 3, containing 10 µg/mL Ag and Spike 4, containing 40 µg/mL Sn
- 100 g/L $LiNO_3$ solution

Begin with ULTRA SCIENTIFIC stock standards.

10.5.1 Spike 3 is prepared by adding 1.0 mL of 1,000 µg/mL Ag to a 100 mL volumetric flask half filled with Blank Solution. Bring up the final volume with Blank Solution. This spike is used for the postspike.

10.5.2 Spike 4 is prepared by adding 4.0 ml of 1,000 ug/ml Sn to a 100 ml volumetric flask filled with blank solution. Bring up the final volume with blank solution. This spike is used for the post-spike.

10.5.3 The 200.0 g/L $LiNO_3$ (0.1%) solution is prepared by weighing out 200.0 g of $LiNO_3$ and dissolving in a 1000 mL volumetric flask half filled with Blank Solution. Dilute to one liter with Blank Solution. This is the buffer for the internal standard.

10.6 The following calibration standards and solutions are to be prepared in the laboratory:

- Std. 1
- Std. 2
- Std. 3

- ICSA
- ICSAB
- CCV
- Internal Standard
- CRI
- NAKCAMG100
- NAKCAMG400

These standards and solutions are prepared from ULTRA SCIENTIFIC stock standards and prepared stock solutions.

10.6.1 Std. 1 is prepared by adding 20 mL of Std. 3 (Section 10.6.3) to a 200 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solutions. See Table 22.8 for concentrations of elements in Std. 1.

10.6.2 Std. 2 is prepared by adding 100 mL of Std. 3 (Section 10.6.3) a 200 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.8 for concentrations of elements in Std. 2.

10.6.3 Std. 3 is prepared by adding 5.0 mL ICUS-575, 5.0 mL ICUS-576, 0.5 mL 1000 µg/mL Sn and 0.5 mL 1000 µg/mL Ag to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.8 for concentrations of elements in Std. 3.

10.6.4 The ICSA is prepared by adding 50.0 mL of ICSA stock solution (ICM-441) to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.9 for concentrations of elements in the ICSA.

10.6.5 The ICSAB is prepared by adding 50.0 mL of stock solution (ICUS-919) to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.9 for concentrations of elements in the ICSAB.

10.6.6 The NAKCAMG100 is prepared by adding 1.0 mL of each 10,000ppm stock solutions Na (ICP-111-5), K (ICP-119-5), Ca (ICP-120-5), Mg (ICP-112) to a 100 mL volumetric flask half filled with Blank Solution. See Table 22.9 for concentrations of elements in the NAKCAMG100.

10.6.7 The NAKCAMG400 is prepared by adding 4.0 mL of each 10,000ppm stock solutions Na (ICP-111-5), K (ICP-119-5), Ca (ICP-120-5), Mg (ICP-112) to a 100 mL volumetric flask half filled with Blank Solution. See Table 22.9 for concentrations of elements in the NAKCAMG400.

10.7. The following standards are prepared from HIGH PURITY

10.7.1. A quality control sample (called the CCV) is prepared from HIGH PURITY stock standards. See Table 22.10 for true values for the CCV. The CCV is prepared by adding 5.0 mL CAL STD. #2 –R Solution A, 5.0 mL CAL STD.#2-R Solution B, 0.5 mL of 1000 µg/mL Ag and 0.5 mL of 1000 µg/mL Sn to a 1000 mL volumetric flask half filled with Blank Solution. Bring up to volume with Blank Solution. Final concentrations can be found in Table 22.10.

10.7.2. The Initial Calibration Verification (ICV) is prepared using the same stock as the CCV. It is prepared by adding 75.0ml of the CCV to a 100ml volumetric flask and bringing it up to volume with Blank Solution. Final concentrations can be found in Table 22.10.

10.8. Yttrium – 5 mg/l: The internal standard/profile is prepared by adding 5 mL of 1,000 µg/mL Y stock solution to a 1000 mL volumetric flask half filled with Blank Solution. Add 50.0 mL of the 200 g/L $LiNO_3$ solution. Bring up to final volume with Blank Solution.

10.9. Low Level Verification Standards:

10.9.1. The CRI is prepared by adding 50 ml ICUS-1241 to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 The maximum holding time for metals samples is 180 days from sample collection. Aqueous samples are preserved with nitric acid to a pH<2. Soil samples do not require additional preservation.

11.2 Soil and total water samples are prepared by a digestion procedure in the digestion lab. The digestates are brought to the instrumental lab by the Digestion analyst. The digestates are stored on a shelf in the instrumental lab. When analysis on the digestates are complete, the digestates are placed in a main sample storage area. The main storage area is located near the digestion lab. The main storage area is used to store the original total samples, digestates, and soluble samples. The main storage area is kept locked when unattended. The digestates are kept for 6 months before they are finally disposed of. For CLP work the digestates are stored for 365 days after delivery of the data package. For all CLP, AFCEE and USACE samples, samples must be refrigerated at 4 degrees C from the time of collection until digestion. CLP samples may be disposed of after 60 days in a manner that complies with all applicable regulations.

11.3 Soluble samples are stored in the main sample storage area with the digestates and the original total samples. All samples taken from the storage area must be logged out in the sample custody logbook that is kept in the digestion lab. Samples are logged back in when complete. The main storage area is kept locked when unattended. The key to the storage area can be obtained from the sample control personnel and returned to them when finished.

11.3.1 **Controlled Access Storage**: CLP, AFCEE and USACE samples require controlled access storage with strict Chain-of –Custody procedures. Digestates for these samples are obtained

from and returned to the cooler custodian. The custodian maintains both the original samples and the digestates in the locked controlled access storage cooler.

11.3.1.1 The original samples are kept for 60 days following delivery of the final report package.

11.3.1.2 Digestates are maintained for 365 days for CLP samples; 60 days for AFCEE and USACE samples.

11.4 Most total and soluble samples have already been preserved by sample control when they were received or in the field when the samples were taken. Preservation is required by the laboratory analyst in cases that samples have not been filtered and preserved. A comment, listing lot numbers of the acid and filter used, is placed in the particular job affected

12.0 QUALITY CONTROL:

Overview: This section provides the guidelines of the quality control that are used to determine if data are useable or not. Depending on the clients' requests and each specific protocol, some QC samples may not be prepared and/or analyzed to each job. Any observed deviations must be documented for future references. If the analyst cannot make a decision about the usability of data, the supervisor must be consulted and the resolution must be documented. If data are unusable, the samples must be redigested and/or reanalyzed depending on the situation. For details on how an actual analytical run is laid out see Section 14.0 - Procedures. For details on any of the calculations which are required in this section see Section 15.0 - Calculations.

12.1 Standards - To insure quality data, all working standards are prepared from high quality certified stock standards. All prepared standards are logged into the standards logbook to insure traceability. Stock solutions are purchased as often as necessary to insure a fresh source.

12.2 Instrument Calibration - The instrument is calibrated daily at the beginning of each analytical run. All relevant information is printed for reference. A blank and 3 levels of standards are used to calibrate each element. A linear plot of each element is produced. The correlation coefficient (which is printed right after the calibration standards have been analyzed) for each element must be 0.995 or greater. If the correlation is less than 0.995 for a particular element, then the data for that element may not be used from that particular analytical run.

12.3 ICL - The ICL is the highest calibration standard that is analyzed after the instrument is calibrated. Results for the ICL must agree within ±5% of the true value of each element. If the ICL is outside the control limits for an element, then the instrument must be re-calibrated or that element cannot be used from that analytical run. See Table 22.8 for the true values of the highest standard (STD. 3). The ICL is called STD 3 Ver on the analytical run.

12.4 ICV - The ICV is prepared from a separate source other than the calibration standards. It is analyzed after the ICL. See Table 22.15 for ICV control limit criteria. If the ICV is outside the control limits for an element, then the instrument must be recalibrated or that element cannot be

used from that analytical run. The measured values must be within +/- 10% of the true value for CLP and method 6010B. The measured values must be within +/- 5% of the true value for method 200.7. See Table 22.10 for the true values of the ICV.

12.5 CCV's - The CCV is prepared from a separate source other than the calibration standards. It is analyzed after every ten samples and at the end of the analytical run. See Table 22.15 for CCV control limit criteria. If the CCV is outside the control limits for an element, the ten samples before and after that CCV should be reanalyzed for that element. See Table 22.10 for the true values of the CCV.

12.6 ICB and CCB's - After analyzing the ICV, analyze an ICB. After analyzing each CCV, analyze a CCB. See Table 22.15 for blank control criteria.

12.7 ICSA – See Table 22.15 for recovery criteria for the ICSA standard. If the ICSA is outside the control limit for an element, that element cannot be used from that analytical run. See Table 22.9 for the true values of the ICSA.

12.8 ICSAB- After analyzing the ICSA, analyze an ICSAB. See Table 22.15 for recovery criteria. If the ICSAB is outside the control limits for an element, that element cannot be used from that analytical run. See Table 22.9 for the true values of the ICSAB.

12.9 Method Blank – For each batch of digestions, one method blank is digested for every 20 samples. Table 22.15 summarizes method blank compliance criteria.

12.10. LFB - For each batch of digestions of water samples, an LFB (Lab Fortified Blank) is prepared. Refer to Table 22.15 for compliance criteria. If the LFB for an element is outside the control limits, then all the samples for that element must be re-digested. See Table 22.6 for the concentrations of each analyte.

12.11. LCS - For each batch of digestions of soil samples, a LCS (Laboratory Control Sample) is prepared. The certified values are different from lot to lot, and the certified values can be found in the binder of the Certificate of Analysis. The acceptance limits are provided by the supplier. If the LCS for an element is outside the control limits, all the samples for that element must be re-digested.

12.11.1. Instead of this LCS, AFCEE and USACE require a water aliquot to be spiked in a similar fashion to the LFB in 12.10. See Table 22.6 for the concentration of each analyte.

12.12 Matrix Duplicate (MD) - For CLP + per client request, one matrix duplicate is performed per digestion batch. See table 22.15 for duplicate compliance criteria. If the RPD is outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or redigest. Generally MD is performed only for CLP digestions.

12.13 Matrix Spike (MS) and Matrix Spike Duplicate (SD) - For each batch of samples, two matrix spikes are performed (one is MS and the other is SD). See Table 22.6 for the concentrations of the matrix spikes for each element. See table 22.15 for criteria for spike recovery and precision. If the RPD is

outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or redigest. If the recovery for an element is outside the control limits, matrix effect is suspected for digestion and/or the determination. Generally SD is performed for SW-8463 and CFR protocols.

12.14 Post Spike - A post digestion spike is performed based on client requirements. It is performed on the base sample that has an MS associated with it. The spike recovery must agree within the limits specified in Table 22.15. If the post spike for an element is outside the control limits, the matrix effect is suspected in the ICP determination.

12.15 Serial Dilution - A serial dilution is performed on the base sample in the batch of 20 that has a matrix spike. If the serial dilution is outside the control limit, the matrix effect is suspected in the ICP determination. The dilution is a 1:5 (one part of the sample to four parts of the blank solution).

12.16 IEC, IDL, AND LINEAR RANGE DETERMINATION

12.16.1 IEC's - The IEC's (interelement correction factors) are determined by first analyzing the following solutions:

- 100 µg/mL Cr
- 100 µg/mL Mn
- 100 µg/mL V
- 1000 µg/mL Fe
- 1000 µg/mL Al
- 1000 µg/mL Ca
- 1000 µg/mL Mg
- 100 µg/mL Tl
- 100 µg/mL Ti
- 100 µg/mL Mo
- 100 µg/mL Co
- 100 µg/mL Cu

Each of the above solutions is prepared from individual 10,000 ug/ml (Ultra Scientific) stock standards at the above concentrations.
Next check to see whether there are any false positive or negative readings for the other elements in each of the solutions. If there are any, then an IEC calculation is necessary.

To calculate the IEC factor, divide the false reading for an element by the actual reading of the interfering element.

Sample Calculation:
The following results are obtained after running a 200.0 µg/mL Fe solution:

Fe = 208.0 µg/mL
Cd = 1.21 µg/mL

The IEC factor for Cadmium would be:

$$\frac{1.21}{208.0} = 0.00582$$

12.16.2 IDLs – For non-CLP protocols, the IDL is determined by analyzing a blank solution 7 times. Calculate the standard deviation of the 7 readings for each element. The IDL is 3 times the standard deviation. For CLP protocol, the IDL is determined by multiplying 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution at a concentration 3x-5x the estimated IDLs, with seven consecutive measurements per day.

12.16.3 Linear Ranges - The linear range is the highest standard the instrument can read which is ±5% of the known value. Analyze a series of standards for each element. The highest that is within ±5% establishes the linear range.

12.17 Additional AFCEE and USACE specific quality control requirements are detailed in Tables 22.18 and 22.19.

12.18 The internal standard counts are monitored for every analysis. The internal standard counts must fall between 50 and 150 percent of the counts of the internal standard in the initial calibration blank. If the internal standard fails to fall between 50 and 150 percent of the initial blank the data from that particular sample may not be used from that analytical run. Recalibrate and reanalyze the sample.

13.0 CALIBRATION AND STANDARDIZATION

13.1 The daily stardization of the 61E Trace analyzer is done automatically at the beginning of each run and approximately every 60 samples. The calibration standards are programmed into the autosampler table. The programming procedure and the standards used are outlined in section 14.

13.2 A Calibration Curvefit is used to calibrate the Na, K, Ca, and Mg wavelengths. This procedure uses an extended calibration with more standards to produce a more linear and precise response over a greater range. The calibration is then fitted to a curve, which can be resloped during a instrument standardization using the low and high standards of the daily calibration. A Calibration Curvefit is preformed quarterly or if significant instrument changes occur. The procedures below explain the steps to run a calibration curvefit

13.2.1 Calibration Curvefit Sample Table Setup and Modification

The following procedure is used to select the elements to be standardized and the concentration of the standards used during a curvefit, or to modify an existing standards table.

- Starting from the main menu, go to "Development".
- Go to "Calibration-Standards".
- Type table name to be create or modified.
- To add a standard press <F1>.
- Add elements to the standard by pressing <F1>.
- To add an element on the periodic table highlight the element to be added and press <F1>. To remove an existing element press <F2>.
- Press <F9> when done.
- Type in the concentration of the each element in the standard.
- Press <F9> when done.
- To modify an existing standard in the table, highlight it, and press <F1>. This will allow you to change the elements and concentrations in the standard by following the instructions above.
- To delete a standard press <F5>.

NOTE: The blank and highest standard of the daily curve must be represented exactly in curvfit table including the same concentrations. If this is not done the fitted curve will not automatically be resloped during daily calibration.

13.2.2 Analysis of the Calibration Curvefit Standards

This procedure is used when analyzing a curve to be fitted.

- Starting from the main menu go to "Development".
- Go to "Calibration –Analysis"
- Enter the standards table name and designate a calibration data file using a one digit run number, three letter month abbreviation, two number day date, and the last two digits of the year (example: 1MAR0705).
- Press <F9> Done to continue.
- To analyze a standard, begin by manually aspirating the standard, then press<F1>Run Standard, and skip comments section by pressing <F1>Run.
- When analysis is complete press <F9>Done.
- Repeat above two steps for each standard in the table.
- When all standards have been analyzed press <F9>Done.

13.2.3 Fitting the Curve

These steps are used to fit the analyzed standards or refit an existing data set.

- Starting at the main menu, go to "Development".
- Go to "Calibration-Curvefit".
- Select or enter the standards table and data file used during analysis.
- Press <F9>Done to continue.
- Select the element/line to be fitted.
- Press <F1>Fit Element.

- Press <F1>Fit Element.
- Under fit type select "Full Fit".
- Under Weight select "Concentration Zero Factor :100.000".
- Press <F9>Done Calc.
- Check that the Correlation Coefficient > 0.995.
- Press <F9>Done.
- Repeat for the rest of the elements to be fitted.

13.2.4 Integrating the Calibration Curvefit into the Instrument Method

The following procedure is used to integrate the Calibration Curvefit into the chosen method. This will allow the Calibration Curvfit to be resloped with the daily curve using the blank and high standard.

- Starting on the main menu, go to "Development".
- Go to "Methods".
- Type the method name.
- Press <F5>Element Info
- Highlight the fitted element. The drop down menu below the line marked "Stdzn Method" should read "2-pt Calib" and the high and low standards names should match both the daily curves high and low standards as well as the two matching standards in the curve fit table. If these are correct check the remaining curvefit elements.
- If any of the criteria are incorrect press <PgDwn> twice.
- Highlight the incorrect item and type correction or press spacebar for drop down.
- Press <F9>Done to save.
- Repeat for the remaining curvefit elements.
- Press <F9>Done/Keep to save changes.
- Next from "Modifying Existing Method" page press <F3>Method Info.
- At the bottom left of the page enter the "Calibration Data File" and the "Calibration Stds" table name.
- Press <F9>Done/Keep.

These steps should have completed Calibration Curvefit standardization and integration into the method. Run a daily curve to confirm.

14.0 PROCEDURES

14.1 The following is a daily checklist for the operation of 61E Trace analyzer. This is only a summary of the basic steps. Each step is followed by a reference for further information.

1. Empty the main drain waste and the autosampler drain waste, if necessary. See Section 14.2.2.1
2. Fill the autosampler rinse, if necessary. See Section 14.2.2.2
3. Refill the internal standard, if necessary. See Section 14.2.2.3.
4. Inspect the pump tubing daily. Either change tubing or change side to side. See section

14.2.2.7.

5. Change the torch, if necessary. The torch should be changed when necessary. See Section 14.2.2.4. The procedure for cleaning the torch is described in Section 14.2.2.5.
6. Check the argon pressure, if necessary. See Section 14.2.2.6.
7. Fill the Argon Saturator if necessary. See Section 14.2.2.12
8. Ignite the plasma. See Section 14.3.
9. Start the peristaltic pump. See Section 14.3.2.
10. Prepare the standards and QC samples. These must be re-poured daily. See Section 10.0.
11. Type the run into an autosampler table and print the table. See section 14.5.
12. Change the data file name for each run. See Section 14.6.
13. Place the standards and QC samples in the autosampler. See Section 14.7.
14. Run a profile before each run. See Section 14.8.
15. Prepare the samples and place them into the autosampler. See Section 14.9.
16. Start the analysis. See Section 14.10.
17. When the analysis is complete, print the data in a condensed format. See Section 14.11.
18. Turn off the plasma unless you want to perform another analysis. See Section 14.12.
19. Empty the samples and standards into a waste receptacle.
20. Perform a validation of the data. See Section 14.13.

14.2 Instrument Layout and Maintenance

This section contains information on the layout of the instrument and any maintenance that the analyst might perform. Any maintenance beyond what is covered here, will be performed by Thermo Jarrell Ash service personnel.

14.2.1 There are two 61E Trace Analyzers at STL Buffalo. They are both set up in a similar manner.

14.2.1.1 See Section 22.11 for a diagram of the main instrument layout.

14.2.1.2 See Section 22.12 for a diagram of the tubing layout.

14.2.1.3 See Section 22.13 for a diagram of the torch/spray chamber/nebulizer assembly.

14.2.1.4 See Section 22.14 for a diagram showing the layout of the autosampler. Rack #1 holds the 28 mL disposable polypropylene sample vials and are used for the calibration standards and quality control standards.

14.2.1.5 Racks #2, #3, #4, and #5 hold the disposable polypropylene culture tubes and are used for the samples.

14.2.2 The following is a list of maintenance topics and problems that need troubleshooting:

- Emptying the waste (See Section 14.2.2.1).

- Filling the autosampler rinse (See Section 14.2.2.2).
- Filling the Internal Standard (See Section 14.2.2.3).
- Filling Argon Saturator (See Section 14.2.2.12).
- Changing the torch (See Section 14.2.2.4).
- Cleaning the torch (See Section 14.2.2.5).
- Check the argon pressure (See Section 14.2.2.6).
- Changing the pump tubing (See Section 14.2.2.7).
- The cooling water level is low (See Section 14.2.2.8).
- The red warning light on the power supply comes on (See Section 14.2.2.9).
- Droplets form on the wall on the inside of the spray chamber (See Section 14.2.2.10).
- The "By Pass" light comes on (See Section 14.2.2.11).

14.2.2.1 Emptying the Waste

14.2.2.1.1 There is one waste container for the 61E Trace Analyzer. One waste line comes from the excess sample draining from the spray chamber (this is called main instrument drain). The other waste line comes from the autosampler rinse station. Both lines run into a central container and are acid wastes and must be disposed of properly. The analyst is responsible for disposal of the acid wastes. Make sure that the 5 gal. waste container is labeled for nitric acid (AN) waste.

14.2.2.1.2 The following steps are used when emptying the instrument waste container:

- Unscrew the instrument waste cap.
- Replace full carboy container with an empty one.
- Replace the cap being sure not to tangle the tubing.

CAUTION: Always wear gloves and safety glasses when handling wastes.

14.2.2.2 Filling the Autosampler Rinse

- Pull the tubing from the 20 liter rinse container.
- Refill the rinse container with reagent blank prepared according to Section 10.4.2.
- Replace the tubing into the rinse container.
- Place a piece of parafilm on the top of the rinse container to keep dust out.

14.2.2.3 Filling the Internal Standard

- Pull the tubing out of the internal standard flask.
- Fill the flask with the internal standard prepared according to Section 10.8.
- Place the tubing back into the internal standard flask.
- Place a piece of parafilm on the top of the flask to keep dust out.

14.2.2.4 Changing the Torch - Refer to the diagram in Section 22.13 to aid in changing the torch. Use the following steps:

- Turn the plasma off.
- Pull the spray chamber out.
- Remove the two screws holding the white cover in place.
- Remove the white cover.
- Pull out the gray collar. Be careful not to bump the quartz end.
- Remove the two argon lines from the gray collar by unscrewing the connectors.
- Remove the black O ring from the end of the gray collar.
- Tip the gray collar until the torch slides out.
- Place a clean torch in the instrument and follow steps 1 through 8 in reverse order.
- Ignite the plasma

14.2.2.5 Cleaning the Torch – Soak the torch overnight in a 1:1 HNO_3 solution (equal parts of HNO_3 acid and ELGA water). Rinse the torch with ELGA water and allow to dry.

Prepare the 1:1 HNO_3 solution (equal parts of HNO_3 acid and ELGA water) by adding 100 mL of ELGA water using a graduated cylinder to a 400 mL beaker. Carefully add 100 mL of conc. HNO_3 acid. Be very careful when working with concentrated acids in this quantity. Work in the fume hood and wear gloves, lab coat, and safety glasses.

14.2.2.6 Checking the Argon Pressure

- The argon regulator is installed on the wall next to the door. Check the pressure to make sure it is 80 psi.

14.2.2.7 Changing the Pump Tubing

There are three types of pump tubing used (use the diagram in Section 22.12 for reference):

- orange/orange/orange - used for the samples.
- orange/green/orange - used for the internal Standard
- red/red/red - used for the autosampler rinse and main instrument waste.

To replace the pump tubing, first pull the tubing from each end of the pump tubing. Insert the tubing into the ends of the new pump tubing.

14.2.2.8 The Cooling Water Level is Low

If the cooling system light comes on, the water level is low. Use the following steps to add water to the cooling system:

- Remove the cover from the top of the cooling system.
- Remove the cap inside.
- Add DI water.
- Replace the inside cap.
- Replace the top cover.

14.2.2.9 The Red Warning light on the Power Supply Comes On

This indicates that the filter on the back of the power supply is dirty. Use the following steps to clean the filter.

- Remove the filter from the power supply.
- Rinse the filter with water.
- Pat the filter dry with a paper towel.
- Allow the filter to dry.
- Replace the filter on the power supply.

14.2.2.10 Droplets Form on the Wall on the Inside of the Spray Chamber

Aspirate a solution of 1:1 Nitric acid (equal parts of HNO_3 acid and ELGA water). Do not aspirate for more than 10 seconds at a time. Prepare the 50% Nitric acid solution by adding 50 mL of conc. Nitric acid to ~50 mL

of ELGA water.

WARNING!: Conc. Nitric acid is extremely hazardous! Work in the fume hood and wear gloves, lab coat, and safety glasses. Pull the hood sash down to protect your face.

14.2.2.11 The "By Pass" Light Comes On

The "By Pass" light comes on if the instrument loses the vacuum due to a power outage or pump failure. If the pump fails, it needs to be fixed first. Use the following steps to regain the vacuum:

- Go to the back of the instrument and turn the switch labeled HV (high voltage) to the off position.
- Dial the Red needle on the vacuum gauge all the way to the right.
- Push the start button.
- When the black needle reads less than 20, dial the Red needle to 20.
- Turn the HV (high voltage) to on.
- Ignite the plasma.

14.2.2.12 Filling the Argon Saturator

If the water level is below "min" line, refilling is necessary with the following steps:

- Lower the neb. pressure to 15 PSI on the plasma screen.
- Turn off neb. pressure.
- Unscrew Argon Saturator cap.
- Fill with Elga Water to the red "Max" line.
- Replace cap
- Turn neb. pressure on.

14.3 Instrument Start-Up

14.3.1 The instrument should always be left on. This is required to maintain the vacuum. If it ever becomes necessary to turn off the instrument, make sure that the high voltage (HV) switch on the back of the instrument is turned to off. If the instrument is not being used on a given day, it is only necessary to turn the computer, monitor, and printer off.

If the computer has been turned off, and if only the C:\ prompt comes up when it is turned on, then type STNRUN and press ⟨Enter⟩ to get into the software. Once in the software the plasma is ready to ignite.

14.3.2 The following steps are used to ignite the plasma and start the peristaltic pump.

- Starting from the main menu. Go to "Setup".
- Go to "Plasma Control Panel".
- Press ⟨Enter⟩.

- Press ⟨F1⟩ for plasma on.
- Press ⟨F9⟩ to continue.
- If the plasma does not ignite, then press ⟨F1⟩ and try again. Make sure there is sufficient argon first and that the valve on the argon tank is open.
- When the plasma ignites, press ⟨Enter⟩ for O.K.
- Press ⟨ESC⟩ to go back to the main menu.
- Go to "Operation".
- Press ⟨Enter⟩.
- Type in the name of the method you want to run.
- Press ⟨Enter⟩. This will start the peristaltic pump. The instrument is now warming up. Allow the instrument to warm up for 30 to 60 minutes.

14.4 Typing an Analytical Run

14.4.1 Each Non-CLP analytical run is typed in the following format:

Calibration: Blk, Std. 1, Std. 2, Std. 3

Std. 3 VER
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB

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CRI	Only for AFCEE / ASP / or Client specific.
ICSA	↓
ICSAB	↓
CCV	↓
CCB	↓

Run a CCV and CCB after every 10 samples and at the end of the analytical run. Run the CRI, ICSA, and ICSAB at the beginning and end of the analytical run.

NOTE: To be compliant with all protocols and clients' particular requests, extensive QC samples are routinely prepared and run. However, not all these QC samples are required for a particular protocol. For example, the ending CRI, ICSA and ICSAB are not required by SW-864 and 40 CFR protocol. Therefore, a particular run may not include ending CRI, ICSA and ICSAB if that procedure only involves standard SW-864 and 40 CFR protocols. This note is also applicable to CLP procedure.

14.4.2 Each CLP analytical batch is typed in the following format:

Calibration
- Blk
- Std. 1
- Std. 2
- Std. 3

Std. 3 VER
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
7 samples
CRI
ICSA
ICSAB
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
7samples
CRI
ICSA
ICSAB
↓

CCV
CCB
CRI
ICSA
ICSAB
CCV
CCB

14.5 Typing the Autosampler Table

Type the analytical run into the autosampler table according to the following steps:

- Starting from the main menu, go to "Exit".
- Go to "ASEDIT.EXE".
- Press ⟨Enter⟩.
- Type in the name of the autosampler table. Use one of the saved templates.
- Press ⟨Enter⟩.
- Press ⟨F1⟩ to edit set.
- Press ⟨F1⟩ to edit samples.
- Use the arrow keys to toggle down to the CCB.
- Holding the ⟨ALT⟩ key down, press:
  - ⟨F1⟩ - to add a sample,
  - ⟨F3⟩ - to add a QC,
  - ⟨F5⟩ - to add calibration standards, or
  - ⟨F7⟩ - to add a blank.
- Type in the ID's of the samples and QC as written in the analytical run log.
- Under the check table column type:
  - ICV - for each ICV,
  - CCV – for each CCV
  - FUCRI – for each CRI,
  - B - for each ICB and CCB,
  - ICSA - for each ICSA
  - ICSAB - for each ICSAB
- After all the samples have been typed in, note the position of the last sample.
- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F5⟩ for modify set.
- Type in the last sample position (from step 12).
- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F9⟩, again, for Done/Keep.
- Press ⟨F2⟩ to print the autosampler table.
- Press ⟨F9⟩ for Done/Keep.

14.6 Entering the Data File Name

Each analytical run requires its own specific data file name. Use the following procedure to enter the data file name prior to the start of the analytical run:

- Starting from the main instrument menu, go to "Development".
- Go to "Methods".
- Press ⟨Enter⟩.
- Type in the method name of the method you are going to run.
- Press ⟨Enter⟩.
- Press ⟨Enter⟩ for method info.
- Use the arrow keys to toggle down to "Analysis Data File".
- Enter the new data file name.

Examples:

For Trace #1

1100500

Key: 1 = run# (1, 2, 3, etc.)
10 = month
05 = day
00= the last two digits of the year

For Trace #2

A100500

Key: A = run # (A,B,C, etc.)
10 = month
05 = day
00 = the last two digits of the year

- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F9⟩, again, for Done/Keep.

14.7 Using the autosampler printout and the autosampler layout diagram (See Section 22.14), set-up the calibration standards and the quality control samples. Use the 28 mL disposable polypropylene sample vials to hold the calibration standards and the quality control samples.

14.8 Instrument Profile

Before each analytical run, a profile of the instrument must be performed. The profile is performed using the Internal Standard, which is a 5 PPM Yitrium solution according to the following steps:

- Starting from the main menu, go to "Operation".
- Go to "analysis".
- Press ⟨Enter⟩.
- Type in the name of the method you are going to run.
- Press ⟨Enter⟩.
- Press ⟨F5⟩ for profile.
- Press ⟨F3⟩ for automatic..
- Press ⟨F1⟩ to run scan.
- When the scan comes up, check the "Peak Position". The "Peak Position" should be between negative 0.05 and positive 0.05.

If yes, then press ⟨F9⟩ for done/keep.
If no, then press ⟨F1⟩ to calc. SS. Check the vernier position on the computer. This should be the same as the vernier position on the instrument. Press ⟨Enter⟩. Dial in the new vernier position.

- Press ⟨F9⟩ for Done/Keep.

14.9 Preparing Samples for the Autosampler

14.9.1 Using the autosampler table printout and the autosampler layout diagram (See Section 22.14), set-up the samples in the autosampler. Use the disposable polypropylene culture tubes. Pour the samples into the culture tubes and place in the autosampler.

14.9.1.1 For 'Total Metals' and 'Soluble Metals', the samples consist of the digestates received from the metals preparation department.

14.9.2 To prepare post spikes, add the following amounts of each spike solution to 9.80 mL of sample:

50 µL - Spike 1 (Section 7.2.2.1)
50 µL - Spike 2 (Section 7.2.2.1)
50 µL – Spike 3 (Section 7.2.2.1)
50 µL – Spike 4 (Section 7.2.2.1)

Mix each post spike thoroughly and place in autosampler.

14.9.3 To prepare the 1:5 serial dilution, add 2.0 mL of sample to 8.0 mL of calibration blank.

14.10 Starting an Analysis

The determination parameters, such as integration time (13 seconds), rinsing time (60 seconds), number of replicates (2), standard concentrations are already entered, and in very rare cases these parameters may be changed. Refer to the instrument operation manual for modifying these values when necessary.

Once the autosampler table has been prepared, the samples, standard and quality control samples have been placed in the autosampler, and the instrument has been profiled, you are ready to begin the analysis. Use the following steps to begin the analysis.

- From the main menu, go to "Operation".
- Go to "Analysis".
- Press ⟨Enter⟩.
- Enter the method name of the method you wish to run.
- Press ⟨Enter⟩.
- Press ⟨F9⟩ for autosampler.
- Type in the autosampler table name under "Sample Name:".

- Press ⟨Enter⟩.
- If you want to have the instrument shut off automatically then press ⟨F7⟩ until the "Terminating Action:" reads "shutdown".
- Press ⟨F1⟩ to start the analysis.

14.11 Printing Data in Condensed Format

The trace instrument will print data during an analysis. A simplified, condensed version of the data may be prepared after the analysis is completed according to the following procedure:

- Starting from the main instrument menu, go to "IMS".
- Go to "Report Writer".
- Press ⟨Enter⟩.
- Enter the file name of the file you wish to print according to the following steps:
  - Press ⟨F6⟩ for a list of files.
  - Press ⟨F2⟩ to deselect all files.
  - Enter the number preceding the file you wish to print.
  - Press ⟨Enter⟩.
  - Press ⟨F9⟩ for Done/Keep.
- Use the arrow keys to go to "Method name".
- Enter the name of the method used to generate the data file.
- Press ⟨Enter⟩.
- Using the arrow keys to toggle back and forth, enter the start date and the end date of the data file you wish to print. You don't need to enter anything for the times.
- Use the arrow keys to go to "Sample type".
- Press the space bar to bring up a list of options.
- Use the arrow keys to toggle up to "All Types".
- Press ⟨Enter⟩.
- Press ⟨F9⟩ to continue.
- Use the arrow keys to toggle down to "Show Internal Standards".
- Change the No to Yes by pressing the "y" key.
- Use the arrow keys to toggle down to "Report format".
- Press the space bar to bring up a list of options.
- Use the arrow keys to toggle down to "vertical".
- Press ⟨Enter⟩.
- Press ⟨F9⟩ for Done/Go to start printing the data file.
- After printing the data file, press ⟨ESC⟩ to return to the main menu.

14.12 Plasma Shutoff

To turn the plasma off, use the following steps:

- From the main menu, go to "Setup".
- Go to "Plasma Control Panel".

- Press ⟨Enter⟩.
- Press ⟨F5⟩.
- Press ⟨Enter⟩.
- After about one minute, press ⟨ESC⟩.

14.13 Validation of the Data

When the analytical run is complete, the data must be checked for compliance with the method. Using Section 12.0 - Quality Control - check all the quality control samples (ICV, ICB, CCVs, CCBs, ICSA, ICSAB, Cal. Standards, ICL, and digested blank and LCS) for compliance. If a quality control sample falls outside the required limits for an element, then that element must be rerun on another analytical run.

Also check the spikes and serial dilution for any matrix effects that might require a diluted sample run.

15.0 CALCULATIONS

15.1 Refer to sections 12.1 through 12.15 and section 14.13 to determine if data are valid for each element. Any sample reading over the linear range must be diluted. Diluted samples must be run on required samples. Analyzing the sample and a series of spiked aliquots of the sample at different known concentrations performs an MSA.

15.2 The following calculations are illustrated:
- Relative Percent Difference (RPD) (See Section 15.2.1).
- Post spike calculation (See Section 15.2.2).
- Method of Standard Addition (MSA) calculation (See Section 15.2.3).

15.2.1 The formula for calculating the relative percent difference is:

$$\%\,RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} X\ 100$$

Where,

RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

Sample calculation: A sample gave a reading of 2.51 µg/mL and the replicate reading was 2.39 µg/mL.

$$\%RPD = \frac{2(2.51-2.39)}{2.51+2.39} X100$$

$$RPD = 4.90\%.$$

15.2.2 The formula for calculating the post spike recovery is:

$$\% \ Recovery = \frac{S_2 - S_1}{SA} X \ 100$$

Where, S_2 = the post spiked sample reading
S_1 = the sample reading
SA = the spike added

Sample Calculations:
A sample gave a reading of 0.250 µg/mL. The sample was post spiked with 2.000 µg/mL and gave a reading of 2.289 µg/mL.

$$\%Recovery = \frac{2.289 - .250}{2.000} X100$$

$$\% \ Recovery = \frac{2.039}{2.000} X \ 100$$

$$\%Recovery = 102.0\%$$

15.2.3 The formula for calculating the simplest version of MSA (single-addition method) is:

Where,

$$C_x = \frac{S_A V_S C_S}{(S_B - S_A) V_x}$$

S_B = the concentration of the spiked sample
S_A = the concentration of the unspiked sample
V_S = volume of spike solution added.
C_S = concentration of spike solution
V_x = volume of sample before adding spike

C_x = the unknown sample concentration

Sample calculation:

A sample gave a reading of 0.792 µg/mL. 50 µL of a 200 µg/mL spike solution was added to 10.0 mL of the sample. The spiked sample reading was 1.512 µg/mL.

$$C_x = \frac{(0.792)(0.05)(200.0)}{1.512 - 0.792)(10.0)}$$

$$C_x = \frac{7.92}{7.20}$$

$$C_x = 1.10ppm$$

16.0 METHOD PERFORMANCE

16.1 This SOP is applicable to digested sample matrices and soluble water samples.

16.2 Extensive quality control is used to insure compliance with method 6010B, 200.7 and CLP protocol.

16.3 Thorough documentation is employed to insure traceability of reagents and standards.

16.4. Approximate detection and reporting limits for Trace #1 and Trace #2 are found in Section 22.2 and 22.3.

16.5. Samples that read above the instrument's linear range must be diluted.

16.5.1. For USACE all samples above the high level calibration standard shall be diluted to within the calibration curve.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 Refer to Table 22.15 for acceptance criteria for QC measurements

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1. If calibration fails, i.e., correlation coefficient is lower than 0.995, ICV and/or ICB are out of control limit, ICSA and/or ICSAB are not recovered quantitatively, the analysis procedure must be terminated, the problems must be solved, and re-calibration must be started over.

18.2. If CCV and/or CCB fail, affected analytes in the 10 samples before and after that CCV/CCB pair must be reanalyzed.

18.3. If LFB or LCS fail, the whole digestion batch must be reanalyzed. If the reanalysis still fails, that whole digestion batch must be reprepared.

18.4. If the Method Blank fails for a analyte, but samples do not contain that analyte higher than the reporting limit or samples contain that analyte higher than 10x the Method Blank, the data is usable and reportable. Otherwise, the digestion batch must be reprepared for that analyte.

18.4.1. For USACE projects, target analytes in the Method Blank must be less than ½ the reporting limit.

18.5. If RPD for SD or MD is out of control limits, the data should be reviewed to determine cause. If redigestion and reanalysis are still out of limits, the sample might be inhomogeneous and the data should be reported with qualification. Refer to table 22.15 for RPD criteria.

18.6 If Post Spike or Serial Dilution fail, matrix effects in determination are suspected.

18.7 If LCS, Post Spike and Serial Dilution are within QC limits but MS fail, matrix interference can be assumed and corrective action is not required.

18.8 For CLP if the percent recovery of the CRI falls outside the control limits of 70-130% (50-150%for Sb, Pb, Tl), the CRI must be re-analyzed for the outlying analytes.

18.8.1 For USACE the control limits are +/- 20% of the true values. If the analysis is not compliant, recalibrate and re-analyze the CRI and all samples associated with it.

18.9 Additional AFCEE and USACE corrective action requirements are detailed in Tables 22.18 and 22.19.

18.10 If the internal standard counts for any analysis fail to fall between 50 and 150 percent of the counts of the internal standard in the initial calibration blank, recalibrate and reanalyze the affected sample/samples.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1. Contingencies for unacceptable data will have to be evaluated on a client-by -client or even by a sample-by-sample basis by the supervisor, the lab manager or the QA manager. Corrective action will be prescribed accordingly.

19.2. In the event acceptable data can not be obtained, a Job Exception Form must be filed with the Project Manager and the client notified.

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20.0 WASTE MANAGEMENT /POLLUTION PREVENTION

20.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

20.2. Waste Streams Produced by the Method: The following waste streams are produced when this method is carried out.

20.2.1. All acidic waste consisting of sample and rinse solution: Dispose of as HNO_3 waste in a "AN" waste container.

21.0 REFERENCES

21.1 Method 6010B, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996.

21.2 ICAP 61E Trace Analyzer Operator's Manual.

21.3 ILM04.1, USEPA Contract Laboratory Program. Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.

21.4 ILM05.2, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.

21.5 Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3.3, 40CFR Part 136, Appendix C, April 1991. (Approved for CWA compliance testing)

21.6 Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Couple Plasma-Atomic Emission Spectrometry", Revision 4.4, US EPA / EMSL, May 1994. (Approved for SDWA compliance testing)

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Elements which are analyzed on ICAP 61E Trace analyzer
22.2. Approximate Water Detection Limits for the ICAP 61E Trace Analyzers
22.3. Approximate Soil Detection Limits for the ICAP 61E Trace Analyzers
22.4. Wavelengths and Background Points Used for Each Element on the ICAP 61E Trace Analyzer
22.5. Approximate Linear Dynamic Range of Each Element on the ICAP 61E Trace Analyzer
22.6. Concentration of each analyte for LFB, LCS, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike.
22.7. Reagents and Stock Solution which are Purchased as Starting Materials for Preparation of Trace

Standards
22.8. Concentration of Calibration Standards
22.9. Values for ICSA and ICSAB
22.10. Values for CCV and ICV
22.11. Main Instrument Layout
22.12. Tubing Layout
22.13. Torch/Spray Chamber/Nebulizer Assembly
22.14. Autosampler Layout
22.15. Blank Page form Analytical Run Log
22.16. Method Summary
22.17. CLP Contract Required Detection Limits (CRDLs)
22.18. Concentration of Each Element in the CRI Standard Solutions
22.19. USACE Requirements
22.20. AFCEE 3.1 Requirements
22.21. Data Review Summary for Metals
22.22. Certificates of Analysis for Custom Blend Standards

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 10.5.3 modified from 100g/L of LiNo3 to 200 g/L and 100g LiNo3 to read 200g LiNo3
23.2 Section 10.8 modified 100g/L to 200g/L
23.3 22.20 Replaced Review Summary form with current form.
23.4 Table 22.15: Revised frequency of CRI and ICSA/ICSAB 200.7 and SW-846 to beginning of analytical run only

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22.1 Elements Which are Analyzed on the ICAP 61E Trace Analyzer:

Aluminum	Al	Magnesium	Mg
Arsenic	As	Manganese	Mn
Antimony	Sb	Molybdenum	Mo (not on Trace 1)
Barium	Ba	Nickel	Ni
Beryllium	Be	Sodium	Na
Boron	B	Potassium	K
Cadmium	Cd	Selenium	Se
Calcium	Ca	Silver	Ag
Chromium	Cr	Thallium	Tl
Cobalt	Co	Vanadium	V
Copper	Cu	Zinc	Zn
Iron	Fe	Tin	Sn
Lead	Pb	Titanium	Ti

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22.2 Approximate Water Detection Limits for the ICAP 61E Trace Analyzers.

Element	Estimated IDL (mg/L)	Estimated MDL (mg/L)	Lab PQL (mg/L)
Al	0.020	0.025	0.2
Sb	0.004	0.005	0.02
As	0.003	0.005	0.01
Ba	0.0002	0.0002	0.002
Be	0.0001	0.0003	0.002
B	0.001	0.005	0.05
Cd	0.00032	0.0006	0.001
Ca	0.014	0.02	0.5
Cr	0.0008	0.001	0.004
Co	0.0008	0.001	0.004
Cu	0.001	0.002	0.01
Fe	0.02	0.030	0.05
Pb	0.002	0.004	0.006
Mg	0.015	0.02	0.2
Mo	0.001	0.002	0.01
Ni	0.00090	0.002	0.01
K	0.052	0.075	0.5
Se	0.003	0.005	0.015
Na	0.250	0.3	1.0
Ag	0.00075	0.0008	0.003
Tl	0.003	0.005	0.02
V	0.001	0.002	0.005
Zn	0.004	0.007	0.02
Sn	0.003	0.004	0.01
Ti	0.00035	0.0008	0.005
Mn	0.0005	0.0003	0.003

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.3 Approximate Soil Detection Limits for the ICAP 61E Trace Analyzers.

Element	Estimated IDL (mg/kg)	Estimated MDL (mg/kg)	Lab PQL (mg/kg)
Al	2.0	2.0	10.0
Sb	0.4	5	15.0
As	0.3	0.5	2.0
Ba	0.02	0.1	0.5
Be	0.01	0.03	0.2
B	0.1	0.5	2.0
Cd	0.03	0.06	0.2
Ca	1.0	2.0	10.0
Cr	0.08	0.1	0.5
Co	0.1	0.1	0.5
Cu	0.1	0.2	1.0
Fe	2.0	3.0	10.0
Pb	0.2	0.4	1.0
Mg	2.0	2.0	20.0
Mo	0.1	0.2	1.0
Ni	0.1	0.2	0.5
K	5.0	8.0	30.0
Se	0.3	0.5	4.0
Na	25	30	140.0
Ag	0.1	0.1	0.5
Tl	0.3	1.0	6.0
V	0.1	0.2	0.5
Zn	0.4	0.7	2.0
Sn	0.3	0.4	2.0
Ti	0.05	0.1	0.5
Mn	0.05	0.05	0.2

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.4 Wavelengths and Background Points Used for Each Element on the ICAP 61E Trace Analyzer.

		Background Points	
Element	Wavelength	Trace #1	Trace #2
Al	3082.15	+10	+10
Sb	2068.38	+10	+10
As	1890.42	-10	-10
Ba	4934.09	+10	+10
Be	3130.42	-10	+10
B	2946.78	+10	+10
Cd	2265.02	-10	-10
Ca	3179.33	-10	-10
Cr	2677.16	-10	+10
Co	2286.16	+10	-10
Cu	3247.53	+10	+10
Fe	2714.41	+10	+10
Pb	2203.53	+10/-10	+10/-10
Mg	2790.78	-10	+10
Mo	2020.30	NA	-10
Ni	2316.04	+10	+10
K	4047.35/7664.35	-28	-28
Se	1960.26	+10/-10	+10/-10
Na	3302.32	+10	-10
Ag	3280.68	-10	-10
Tl	1908.64	+10	-10
V	2924.02	+10	+10
Zn	3062.00	+10	+10
Sn	2899.89	-10	-10
Ti	3372.80	+10	-10
Mn	2576.10	-10	+10

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.5 Approximate Linear Dynamic Range of Each Element on the ICAP 61E Trace Analyzer.

Element	Trace #1 (mg/L)	Trace #2 (mg/L)
Al	500	500
Sb	25	25
As	50	50
Ba	10	10
Be	10	10
B	25	25
Cd	5	5
Ca	500	500
Cr	25	25
Co	5	5
Cu	20	20
Fe	200	200
Pb	10	25
Mg	500	500
Mo	NA	25
Ni	10	10
K	500	500
Se	10	50
Na	500	500
Ag	10	10
Tl	25	50
V	200	200
Zn	200	200
Sn	10	10
Ti	5	5
Mn	50	50

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.6 Concentration of each analyte for LFB, LCS, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike:

Element	LFB, Post-digestion Spike and Non-CLP Matrix Spike (mg/L)	CLP Matrix Spike (4.1) (mg/L)	CLP Matrix Spike (5.2) (mg/L)	Soil Post-digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Estimated Soil LCS (mg/kg) Changes with new lot	CLP Matrix Spike Soil (4.1) (mg/kg)	CLP Matrix Spike Soil (5.2) (mg/kg)
Al	10.0	2.00	2.00	100	6340		
Sb	0.20	0.500	0.100	20	34	20	20
As	0.20	2.00	0.040	20	192	8	8
Ba	0.20	2.00	2.00	20	417	400	400
Be	0.20	0.050	0.050	20	99.9	10	10
B	0.20			20	131		
Cd	0.20	0.050	0.050	20	125	10	10
Ca	10.0			100	3370		
Cr	0.20	0.200	0.200	20	133	40	40
Co	0.20	0.500	0.500	20	56.8	100	100
Cu	0.20	0.250	0.250	20	93.9	50	50
Fe	10	1.00	1.00	100	11600		
Pb	0.20	0.500	0.020	20	160	4	4
Mg	10.0			100	2000		
Mo	0.20			20	62.9		
Ni	0.20	0.500	0.500	20	174	100	100
K	10.0			100	1890		
Se	0.20	2.00	0.010	20	97	2	10
Na	10.0			100	241		
Ag	0.20	0.050	0.050	20	115	10	10
Tl	0.20	2.00	0.050	20	79.1	10	10
V	0.20	0.500	0.500	20	92.7	100	100
Zn	0.20	0.500	0.500	20	246	100	100
Sn	0.20			20	117		
Ti	0.20			20	327		

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Element	LFB, Post-digestion Spike and Non-CLP Matrix Spike (mg/L)	CLP Matrix Spike (4.1) (mg/L)	CLP Matrix Spike (5.2) (mg/L)	Soil Post-digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Estimated Soil LCS (mg/kg) Changes with new lot	CLP Matrix Spike Soil (4.1) (mg/kg)	CLP Matrix Spike Soil (5.2) (mg/kg)
Mn	0.20	0.500	0.500	20	320	100	100

22.7 Reagents and Stock Solutions which are Purchased as Starting Materials for Preparation of Trace Standards.

From ULTRA SCIENTIFIC:

ICUS-575	10,000 µg/mL	Al
ICUS-576	10,000 µg/mL	Sb
ICM-441	10,000 µg/mL	As
ICUS-573	10,000 µg/mL	Ba
ICUS-574	10,000 µg/mL	Be
ICUS-919	10,000 µg/mL	Cd
ICUS-1241	10,000 µg/mL	Ca
1,000 µg/mL Ag	10,000 µg/mL	Cr
1000 ug/ml Y	10,000 µg/mL	Co
1,000 µg/mL Sn	10,000 µg/mL	Cu
CLP-1	10,000 µg/mL	Fe
CLP-2	10,000 µg/mL	Pb
CLP-3	10,000 µg/mL	Mg
	10,000 µg/mL	Mn
	10,000 µg/mL	Mo
	10,000 µg/mL	Ni
	10,000 µg/mL	K
	10,000 µg/mL	Se
	10,000 µg/mL	Na
	10,000 µg/mL	Ag
	10,000 µg/mL	Tl
	10,000 µg/mL	Zn
	10,000 µg/mL	Sn
	10,000 µg/mL	Ti

Certificates of Analysis are attached for the custom blend standards listed as ICUS-(...) above.

From JT-BAKER

Concentration HCl (Trace Metals Grade)
Concentration HNO_3 (Trace Metals Grade)

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

From HIGH PURITY:

Solid $LiNO_3$
10,000 µg/mL Y
1,000 µg/mL Ag
1,000 µg/mL Sn

CAL STD #2-R Solution A
CAL STD #2-R Solution B

Table 22.8 Concentrations of Calibration Standards: (in mg/L)

Element	Std. 1	Std. 2	Std. 3	NAKCAMG100	NAKCAMG400
Al	5.0	25.0	50.0		
Sb	0. 1	0.5	1.0		
As	0. 1	0.5	1.0		
Ba	0. 1	0.5	1.0		
Be	0. 1	0.5	1.0		
Cd	0. 1	0.5	1.0		
Ca	5	25	50	100	400
Cr	0.1	0.5	1.0		
Co	0.1	0.5	1.0		
Cu	0.1	0.5	1.0		
Fe	5	25	50		
Mg	5	25	50	100	400
Mn	0.1	0.5	1.0		
Ni	0.1	0.5	1.0		
Ag	0.1	0.5	1.0		
Tl	0.1	0.5	1.0		
Zn	0.1	0.5	1.0		
V	0.1	0.5	1.0		
B	0.1	0.5	1.0		
Mo	0.1	0.5	1.0		
Ti	0.1	0.5	1.0		

Sn	0.1	0.5	1.0		
Se	0.1	0.5	1.0		
Na	5	25	50.0	100	400
K	5	25	50.0	100	400
Pb	0.1	0.5	1.0		

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.9 Values for ICSA and ICSAB (in mg/L)

Element	ICSAB	ICSA
Al	500.0	500
Ca	500.0	500
Fe	100.0	200
Mg	500.0	500
Ba	0.5	-
Be	0.5	-
Cd	1.0	-
Co	0.5	-
Cr	0.5	-
Cu	0.5	-
Mn	0.5	-
Ni	1.0	-
Pb	0.05	-
V	0.5	-
Zn	1.0	-
Sb	0.6	-
As	0.1	-
Tl	0.1	-
Se	0.05	-
Ag	0.2	-

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.10 Values for CCV and ICV (in mg/L):

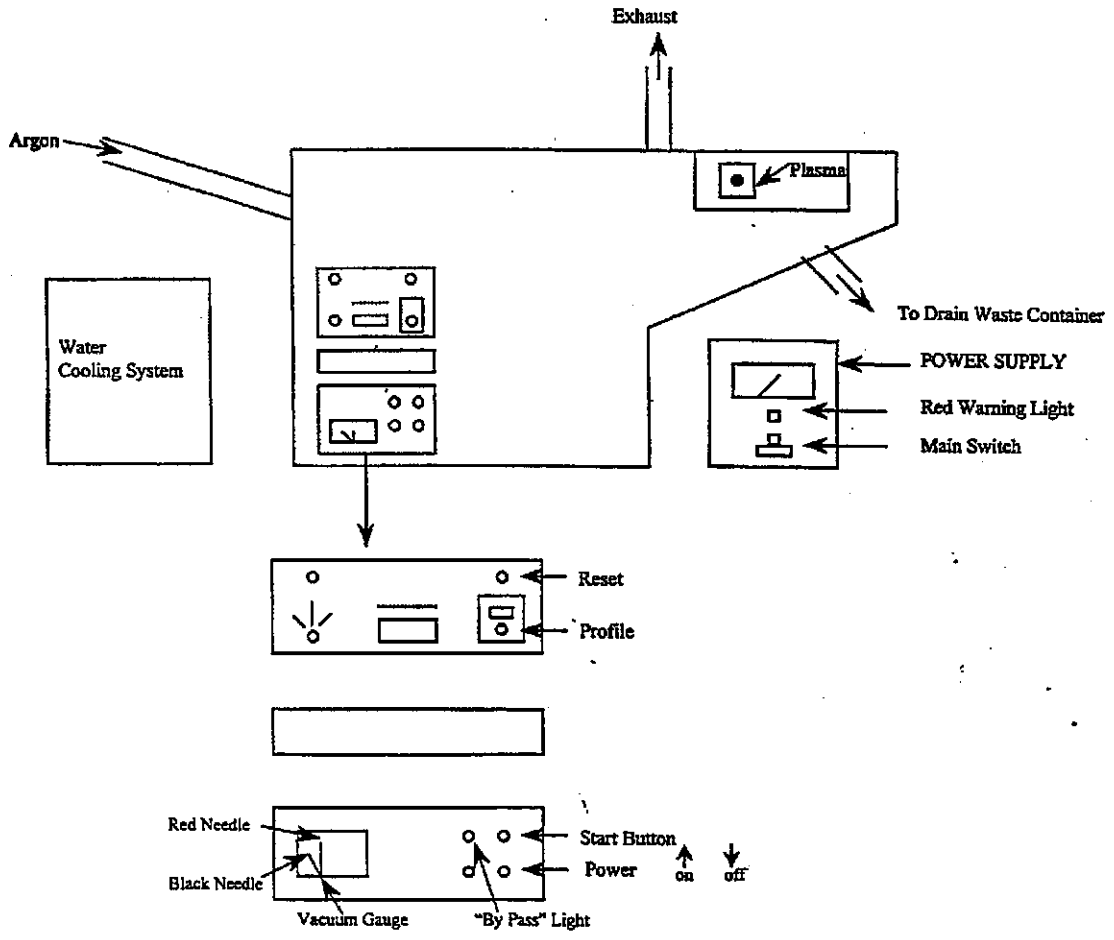
Element	CCV	ICV
Al	25.0	18.75
Sb	0.5	0.375
As	0.5	0.375
Ba	0.5	0.375
Be	0.5	0.375
B	0.5	0.375
Cd	0.5	0.375
Ca	25.0	18.75
Cr	0.5	0.375
Co	0.5	0.375
Cu	0.5	0.375
Fe	25.0	18.75
Pb	0.5	0.375
Mg	25.0	18.75
Mn	0.5	0.375
Mo	0.5	0.375
Ni	0.5	0.375
K	25.0	18.75
Se	0.5	0.375
Na	25.0	18.75
Ag	0.5	0.375
Tl	0.5	0.375
V	0.5	0.375
Zn	0.5	0.375
Sn	0.5	0.375
Ti	0.5	0.375
Mn	0.5	0.375

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.11 Main Instrument Layout:

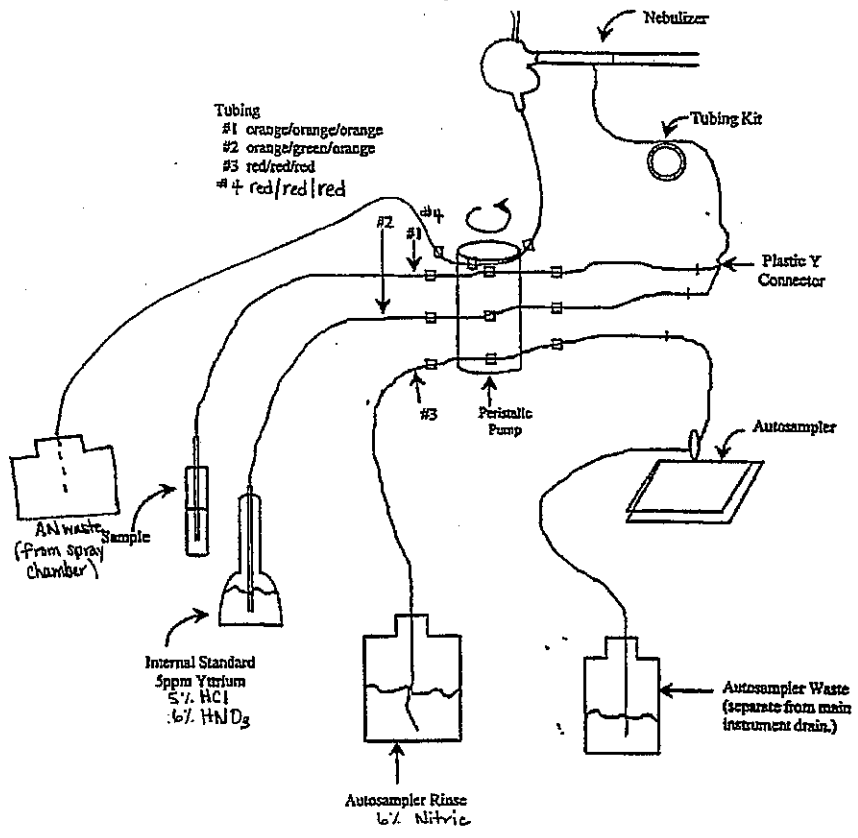
The vacuum pump –not shown- is located behind the instrument. The auto sampler – not shown- is placed in front of the instrument.



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

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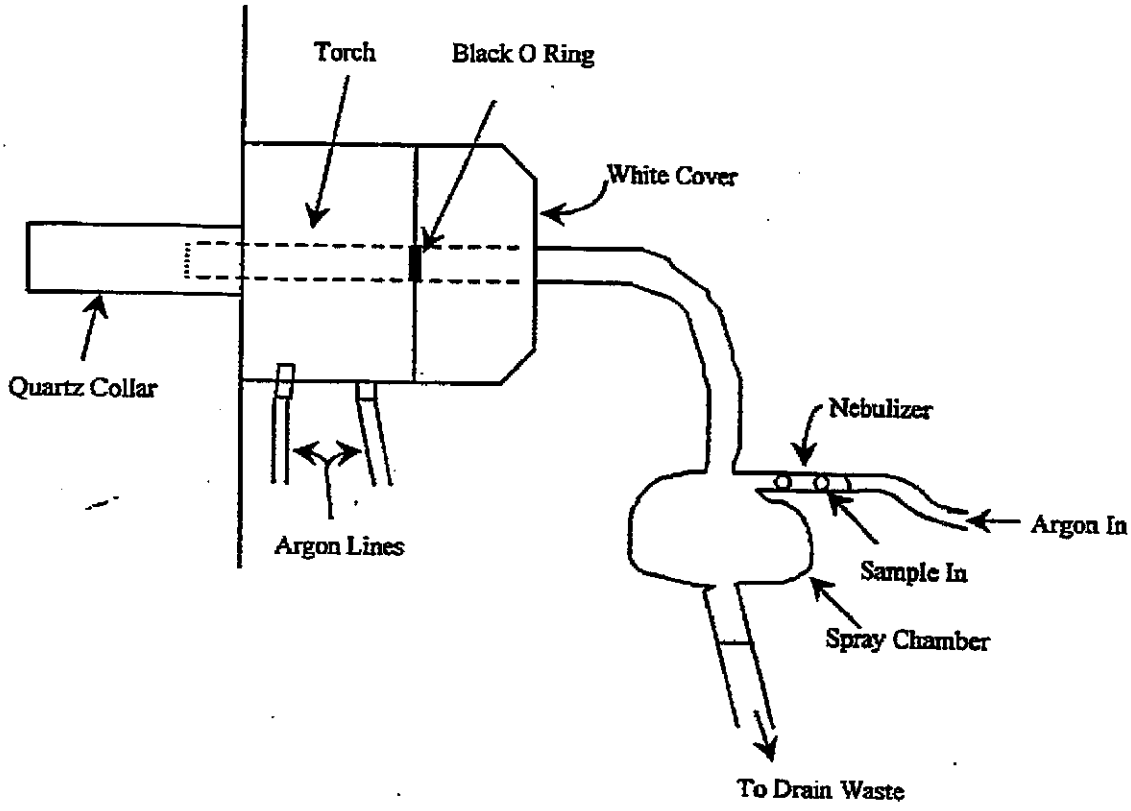
22.12 Tubing Layout:



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

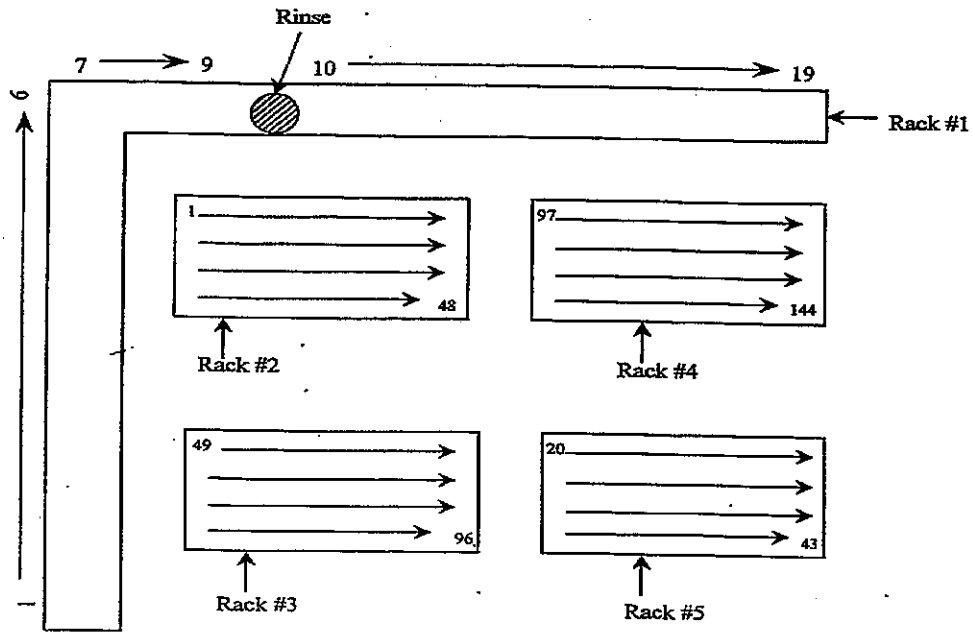
22.13 Torch/Spray Chamber/Nebulizer Assembly:



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.14 **Autosampler Layout:**



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.15: Method Summary

Method ⇒ / Parameter ⇓	EPA Series Method 200.7	SW-846B Method 6010B	CLP
Method Validation (2)	Initial demonstration of performance: 1. Determination of the linear dynamic range. Verify linear calibration range limit by analyzing a high concentration standard. Results must be within 5% of the true value. 2. Analyze LCS within ±10% of stated value. 3. Establish MDLs using Laboratory fortified blanks. MDLs must meet regulatory levels.	Same	Same
QC Check Standards/Samples (ICV)	Verify each element calibration with a Control Sample (ICV) prepared from a source different than the calibration standards at the following concentrations: Silver at a maximum of 0.5mg/L, other elements ≥1.0 mg/L. The ICV must be within 3% RSD. %R = 95-105	Verify each element calibration with an Instrument Check Standard prepared from a source different from the calibration standards at a concentration equivalent to the midpoint of the calibration curves. The ICV must be within 5% RSD. %R=90-110	Initial calibration verified with independent standard. %R=90-110
Method Detection Limit	IDL's are determined quarterly. MDLs are determined annually	IDL's are determined quarterly. MDLs are determined annually	IDL's are determined quarterly. MDLs are determined annually.
Standard Solution Expiration(3)	Stocks: yearly or specified by supplier Intermediate: 6 mos. Working: 3 days to 7 days	(same)	(same)
Initial Calibration	Per instrument manufacturer's specification. Minimum of a blank and one standard.	Per instrument manufacturer's specifications (should consist of 3 levels and a blank). The criteria is that the RSD <3%.	Per instrument manufacturer's specifications. Minimum of a blank and one standard.
Continuing Calibration	Analyze instrument performance check (ICV) solution immediately following calibration, after every 10 samples and at the end of the run. For initial analysis, %R=95-105, for subsequent analyses of standard. %R=90-110.	Analyze instrument check standard after every 10 samples and at the end of the run. %R=90-110.	Calibration checked after every 10 samples or 2 hours whichever is more frequent with a mid-range calibration standard. %R=90-110.

STL BUFFALO
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TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

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Method ⇒ / Parameter ⇓	EPA Series Method 200.7	SW-846B Method 6010B	CLP
Accuracy/Precision	-One MS/MD per 10 samples or each batch %R=75-125%. %RPD < 20% - A laboratory fortified blank (LFB) prepared with each batch of samples digested. %R=85-115.	-One MS/MD per 10 samples or each batch %R=75-125%. %RPD < 20% - A laboratory fortified blank (LFB) prepared with each batch of samples digested. %R=80-120	One MS and one duplicate per sample delivery group or per similar matrix type.%R=75-125. %RPD<20. Analyze a post-digestion spike if the pre-digestion spike recovery is outside control limits and the sample result does not exceed 4 times the spike added.
Blanks	One method blank with each batch of samples. When values constitute 10% or more of the analyte level or are 2.2 times the analyte MDL, whichever is greater, the entire preparation and analysis is repeated. Analyze calibration blank after each instrument check standard (CCV). Blank < 3x IDL.	One method blank per batch of samples processed at the same time. Analyze calibration blank after each Instrument Check Standard (CCV). Blank < 3x IDL.	One method blank per sample delivery group or per sample process batch, whichever is more frequent. Analyze calibration blanks after initial and continuing calibration verification or every 10 samples. If absolute value of blank for any analyte >CRDL, terminate analysis, correct problem, and reanalyze all samples since last compliant blank.
Interference Check Standard	Interference Check Solutions containing known concentrations of interfering elements and the elements of interest are analyzed at the beginning of each analytical run. %R=80-120. Reanalyze highest standard after calibration for checking (not required by the method). %R=95-105.	Interference Check Solutions containing known concentrations of interfering elements and the elements of interest are analyzed at the beginning of each analytical run. %R=80-120. Reanalyze highest standard after calibration for checking (not required by the method). %R=95-105.	Analyze ICSA and ICSAB solutions at a frequency of not greater than 20 analytical samples. Must be followed immediately by CCV/CCB pair. ICSA: For target analytes with CRDL ≤10 µg/L, results should fall within ± CRDL of the analyte's true value, otherwise use alternate method to quantitate results for affected analytes. ICSAB: %R=80-120
IDL Standard (CRI)	Run CRI Standard at the beginning of each analytical run directly following ICB. %R=50-150. Not required per method..	Run CRI Standard at the beginning of each analytical run directly following ICB.. %R=50-150. Not required per method.	Run CRI Standard every 20 analytical samples and at the beginning and end of each analytical run. %R=50-150 For Antimony, Lead, and Thallium %R=70-130 All other elements
Serial Dilution	1:5 Dilution on each new matrix per job. % Difference=10%	1:5 Dilution on each new matrix per job. % Difference=10%	1:5 Dilution on each new matrix per SDG. % Difference=10%
Matrix Spike	%R= 75-125	%R=75-125	%R=75-125
Post Digestion Spike	%R=85-115	%R=75-125	%R=75-125

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Method ⇒ / Parameter ⇓	EPA Series Method 200.7	SW-846B Method 6010B	CLP
Holding Time (4)	180 days from collection	180 days from collection	180 days from receipt (VTSR)

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.16 Contract Required Detection Limits (CRDL)

Analyte	CRDL (4.0) (ng/mL)	CRDL (5.0) (ng/mL)
Aluminum	200	200
Antimony	60	5
Arsenic	10	5
Barium	200	20
Beryllium	5	1
Cadmium	5	2
Calcium	5000	5000
Chromium	10	5
Cobalt	50	5
Copper	25	5
Iron	100	100
Lead	3	3
Magnesium	5000	5000
Manganese	15	10
Mercury	0.2	0.1
Nickel	40	20
Potassium	5000	5000
Selenium	5	5
Silver	10	5
Sodium	5000	5000
Thallium	10	5
Vanadium	50	10
Zinc	20	10

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.17 Concentration of each element in the CRI standard solution and reading on the instrument.

Analyte	CRI Stock Std	CRI
Aluminum	2.0	0.2
Antimony	0.2	0.02
Arsenic	0.1	0.01
Barium	0.02	0.002
Beryllium	0.02	0.002
Boron	0.5	0.005
Cadmium	0.01	0.001
Calcium	5.0	0.05
Chromium	0.04	0.004
Cobalt	0.04	0.004
Copper	0.1	0.001
Iron	0.5	0.05
Lead	0.05	0.005
Magnesium	2.0	0.02
Manganese	0.03	0.003
Nickel	0.1	0.01
Potassium	5.0	0.5
Selenium	0.15	0.0015
Silver	0.03	0.003
Sodium	10.0	1.0
Thallium	0.2	0.02
Tin	0.1	0.01
Vanadium	0.05	0.005
Zinc	0.2	0.02

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.18 USACE Requirements

EM 200-1-3
1 Feb 01

Table I-1
Summary of Measurement quality objectives for Method 6010 Inductively Coupled Plasma (ICP) Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (I.9.2.1.1)	Option 1- 1 std and blank, and a low-level check standard at MQL Option 2- 3 stds and blank	Daily	Option 1- Low-level check standard ± 20% Option 2- r • 0.995
Instrumental Precision (I.9.2.1.1)	%RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	%RSD < 5%
Initial Calibration Verification (ICV) (I.9.3)	Midlevel (2nd source) verification	After initial calibration	%Recovery ± 10%
Initial Calibration Blank (ICB) (I.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL
Interelement Check Standards (ICS) (I.8.1)	ICS-A - interferents only ICS-B - interferents and target analytes	Beginning of analytical sequence	%Recovery ± 20% for target analytes
Continuing Calibration Blank (CCB) (I.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL
Continuing Calibration Verification (CCV) (I.9.5 / I.9.5.1)	Midlevel verification	Every 10 samples and at end of analytical sequence	%Recovery ± 10%
Method Blank (MB) (I.10.2.1 / I.11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < one-half MRL
Laboratory Control Sample (LCS) (I.10.2.2 / I.11.4.2)	Interference-free matrix containing all target analytes	1 per sample batch	%Rec = 80% - 120% Sporadic marginal failures[1]: %Rec = 60% - 140%
Matrix Spike (MS) (I.10.2.3 / I.11.4.3 / I.11.4.3.1)	Sample matrix spiked with all/subset of target analytes prior to digestion	1 per sample batch	%Rec = 75% - 125%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (I.10.2.4 / I.11.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD • 25%
Post Digestion Spike (PDS) (I.10.3.1 / I.11.4.6)	Sample digestate spiked with all/subset of target analytes	1 per sample batch on MS sample	%Rec = 75% - 125%
Serial Dilution (SD) (I.10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results ± 10%
Method of Standard Additions (MSA) (I.12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	r • 0.995

[1] The number of Sporadic Marginal Failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if between 7 to 15 metals are reported from the ICP analysis, one (1) SMF is allowed to the expanded criteria presented. If greater than 15 metals are reported from the ICP analysis, two (2) SMFs are allowed.

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TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

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22.19 AFCEE 3.1 Requirements

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Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	79-120	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	79-120	≤20	80-120	≤30
	Nickel	80-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	75-120	≤30
	Sodium	80-120	≤20	80-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

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Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.05	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

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Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	75-125	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	75-125	≤20	80-120	≤30
	Nickel	80-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	75-125	≤30
	Sodium	80-120	≤20	80-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.06	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

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SUPERCEDES: Revision 15

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Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	79-120	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	79-120	≤20	80-120	≤30
	Nickel	80-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	75-120	≤30
	Sodium	80-120	≤20	80-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

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Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.05	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

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Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	79-120	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	80-120	≤20	80-120	≤30
	Nickel	79-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	80-120	≤30
	Sodium	80-120	≤20	75-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

AFCEE QAPP Version 3.1 August 2001 Page 7-105

Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.05	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.20 Example of a Data Review Summary Form for Metals

Date: 05/31/2006
Time: 15:55:36

STL Buffalo
Metals Job Summary
Analysis Type: TOTAL METALS (WATER)

Page: 2
Rept: AM1500

Job No: AC6-5562
Project/Task: MSAS860210 7

Lab Due Date: 05/30/2006
Client Due Date: 06/08/2006

Test No	Description	CASN	Protocol	Method	Mtx	ICLP	Holding To	Holding Tcln	Holding Exst	Holding Anyt	Prep Type	Unit Meas	Unit Type	Detect Limit Value	Spikes Code	Spikes Amount	Spikes Conc	QC Limits	RPD
CCA001566 TOTAL METALS-6/AA-1753 M_REMAND1.A																			
CTA05201 AG	- WHI - SILVER BY ICP(0.010)-TOT		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.01000	A00267	0.25 ML	10.00 UG/ML	(75-125)	20.0
CTA05737 AS	- WHI - ARSENIC-ICP TJA - TOTAL		SW8463	6010	Water	N	S	0	720	180	D	MG/L	CDL	0.01000	A00267	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05195 BA	- WHI - BARIUM BY ICP(0.020)-TOT		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.02000	A00267	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05203 BE	- WHI - BERYLLIUM BY ICP-TOTAL(0		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.00500	A00267	1.00 ML	10.00 UG/ML	(85-120)	20.0
CTA02612 CD	- WHI - CADMIUM BY ICP(0.0050)-T		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.00500	A00267	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05444 CO	- WHI - COBALT - TOTAL - U WITH RL= 0.		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.02000	A00267	1.00 ML	10.00 UG/ML	(92-120)	20.0
CTA05725 CR	- WHI - CHROMIUM BY ICP(0.010)-T		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.01000	A00267	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05446 CU	- WHI - COPPER - TOTAL - U - WITH RL =		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.01000	A00267	1.00 ML	10.00 UG/ML	(82-120)	20.0
CTA05816 NI	- WHI - NICKEL BY ICP(0.040)-TOT		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.04000	A00267	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA02734 PB	- WHI - LEAD-ICP TJA - TOTAL		SW8463	6010	Water	N	S	0	720	180	D	MG/L	CDL	0.00500	A00267	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05872 SB	- WHI - ANTIMONY-ICP TJA - TOTAL		SW8463	6010	Water	N	S	0	195	180	D	MG/L	CDL	0.02000	A00267	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05882 V	- WHI - VANADIUM BY ICP-TOTAL10.		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.05000	A00267	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA05455 ZN	- ZINC - TOTAL - U - WITH RL= 0.		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.05000	A00267	1.00 ML	10.00 UG/ML	(80-120)	20.0

Comments: _____

Criteria:

Initial Calibration/Second Source Criteria Met? Y / N
CCV/CCB Criteria Met? Y / N
Method Blank Criteria Met? Y / N
LCS Criteria Met? Y / N
MS/MD Criteria Met? Y / N

Analyst Approval: _____ Date: _____
Data Processor: _____ Date: _____
Data Reviewer: _____ Date: _____

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.21 Certificates of Analysis for Custom Blend Standards

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

MDL-1-27

Certificate of Analysis

Custom Inorganic Standard

Catalog Number: ICUS-575

Lot Number: D00396

Expiration Date: 08/2004

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
antimony	100.0	µg/mL	gravimetric
arsenic	100.0	µg/mL	gravimetric
beryllium	100.0	µg/mL	gravimetric
cadmium	100.0	µg/mL	gravimetric
chromium	100.0	µg/mL	gravimetric
cobalt	100.0	µg/mL	gravimetric
copper	100.0	µg/mL	gravimetric
lead	100.0	µg/mL	gravimetric
manganese	100.0	µg/mL	gravimetric
molybdenum	100.0	µg/mL	gravimetric
nickel	100.0	µg/mL	gravimetric
selenium	100.0	µg/mL	gravimetric
thallium	100.0	µg/mL	gravimetric
titanium	100.0	µg/mL	gravimetric
vanadium	100.0	µg/mL	gravimetric
zinc	100.0	µg/mL	gravimetric
calcium	4999.7	µg/mL	gravimetric
iron (99.999%)	5000.0	µg/mL	gravimetric
magnesium	5000.0	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

ISO-9001:2000 Registered by PRO Reg. No. 00-R1192rev.1

ISO 17025 Certified by Cert. No. 0851-01

John E. Russo, Chem. Eng.
Quality Control Manager

Certificate of Analysis MDL-2-27

Custom Inorganic Standard

Catalog Number: ICUS-576

Lot Number: D00385

Expiration Date: 08/2004

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
barium	100.0	µg/mL	gravimetric
boron	100.0	µg/mL	gravimetric
aluminum	5000.0	µg/mL	gravimetric
potassium	5000.0	µg/mL	gravimetric
sodium	5000.0	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis

27-NOV-15

CLP ICP Interference Check Standard I

Catalog Number: ICM-441

Lot Number: D00226

Expiration Date: 05/2006

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method	NIST SRM#
aluminum	5000.0	µg/mL	ICP	3101a
calcium	5000.0	µg/mL	ICP	3109a
iron	2000.0	µg/mL	ICP	3126a
magnesium	4999.8	µg/mL	ICP	3131a

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager

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TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis

MDL-7-26

Custom Inorganic Standard

Catalog Number: ICUS-574

Lot Number: D00347

Expiration Date: 08/2005

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
barium	40.0	µg/mL	gravimetric
boron	40.0	µg/mL	gravimetric
aluminum	2000.0	µg/mL	gravimetric
potassium	2000.0	µg/mL	gravimetric
sodium	2000.0	µg/mL	gravimetric

Matrix: 5% nitric acid and water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager

ULTRA Scientific

Certificate of Analysis

Custom Inorganic Standard

Catalog Number: ICUS-919
Lot Number: E00155
Job Number: J00003691
Expiration Date: 06/2005

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard The standard uncertainty is ± 0 5% relative, unless otherwise specified

Analyte	True Value		Analytical Method
silver	2.0	mg/L	gravimetric
arsenic	1.0	mg/L	gravimetric
barium	5 0	mg/L	gravimetric
beryllium	5 0	mg/L	gravimetric
cadmium	10 0	mg/L	gravimetric
cobalt	5.0	mg/L	gravimetric
chromium	5 0	mg/L	gravimetric
copper	5 0	mg/L	gravimetric
manganese	5 0	mg/L	gravimetric
nickel	10 0	mg/L	gravimetric
lead	0 5	mg/L	gravimetric
antimony	6 0	mg/L	gravimetric
selenium	0 5	mg/L	gravimetric
thallium	1 0	mg/L	gravimetric
vanadium	5 0	mg/L	gravimetric
zinc	10 0	mg/L	gravimetric
aluminum	5000 0	mg/L	gravimetric
calcium	5000 0	mg/L	gravimetric
iron	1000 0	mg/L	gravimetric
magnesium	5000 0	mg/L	gravimetric

Matrix. 5% nitric acid in water

All weights are traceable to NIST traceable weights

ISO-9001:2000 Registered by
Reg No 00-R1192rev.1

ISO 17025 Certified by
Cert. No 0851-01

John E Russo, Chem. Eng
Quality Control Manager

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-1241
Lot Number: E00337
Job Number: J00003929
Expiration Date: 08/2005

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard The standard uncertainty is ± 0 5% relative, unless otherwise specified

Analyte	True Value		Analytical Method
aluminum	2 0	µg/mL	gravimetric
antimony	0 2	µg/mL	gravimetric
arsenic	0 1	µg/mL	gravimetric
barium	0 02	µg/mL	gravimetric
beryllium	0 02	µg/mL	gravimetric
boron	0 5	µg/mL	gravimetric
cadmium	0 01	µg/mL	gravimetric
calcium	5 0	µg/mL	gravimetric
chromium	0 04	µg/mL	gravimetric
cobalt	0 04	µg/mL	gravimetric
copper	0 1	µg/mL	gravimetric
iron	0 5	µg/mL	gravimetric
lead	0 05	µg/mL	gravimetric
magnesium	2 0	µg/mL	gravimetric
manganese	0 03	µg/mL	gravimetric
molybdenum	0 1	µg/mL	gravimetric
nickel	0 1	µg/mL	gravimetric
potassium	5 0	µg/mL	gravimetric
selenium	0 15	µg/mL	gravimetric
silver	0 03	µg/mL	gravimetric
sodium	10 0	µg/mL	gravimetric
thallium	0 2	µg/mL	gravimetric
tin	0 1	µg/mL	gravimetric
titanium	0 05	µg/mL	gravimetric
vanadium	0 05	µg/mL	gravimetric
zinc	0 2	µg/mL	gravimetric

Matrix: 5% nitric acid in water

ISO-9001:2000 Registered by
All weights are traceable to NIST traceable weights
ISO 17025 Certified by

Reg No 00-R1192rev 1

Cert No 0851-01

John E. Russo, Chem. Eng
Quality Control Manager

P.O. Box 41727
Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-606-044 (CAL STD. #2-RR)

Solution A

Lot # 417517

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals Salts or Oxides	99.99+%	HNO_3, 5%	µg/mL ± 0.5% See elements listed on back

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp. Date JUL 0 5

MSDS ATTACHED

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

P.O. Box 41727
Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-606-044 (CAL STD. #2-RR)

Solution B

Lot # 417517

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals Salts or Oxides	99.99+%	HNO_3, 5% + Tr HF	100 µg/mL ± 0.5% Antimony Molybdenum Titanium

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

JUL 0 5
Exp. Date
MSDS ATTACHED

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-1370

Lot Number: F00019

Job Number: J00004351

Expiration Date: 02/2006

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value				Analytical Method
antimony	40.00	±	0.20	µg/mL	gravimetric
arsenic	40.00	±	0.20	µg/mL	gravimetric
beryllium	40.00	±	0.20	µg/mL	gravimetric
cadmium	40.00	±	0.20	µg/mL	gravimetric
chromium	40.00	±	0.20	µg/mL	gravimetric
cobalt	40.00	±	0.20	µg/mL	gravimetric
copper	40.00	±	0.20	µg/mL	gravimetric
lead	40.00	±	0.20	µg/mL	gravimetric
manganese	40.00	±	0.20	µg/mL	gravimetric
molybdenum	40.00	±	0.20	µg/mL	gravimetric
nickel	40.00	±	0.20	µg/mL	gravimetric
selenium	40.00	±	0.20	µg/mL	gravimetric
thallium	40.00	±	0.20	µg/mL	gravimetric
vanadium	40.00	±	0.20	µg/mL	gravimetric
zinc	40.00	±	0.20	µg/mL	gravimetric
titanium	40.00	±	0.20	µg/mL	gravimetric
calcium	2000	±	10	µg/mL	gravimetric
iron	2000	±	10	µg/mL	gravimetric
magnesium	2000	±	10	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851-01

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STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES
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REVIEWED & APPROVED BY:	Signature	Date
Verl Preston, Quality Manager	Verl D. Preston	9/26/06
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1.0 IDENTIFICATION OF TEST METHODS

1.1 This SOP outlines the method for the determination of mercury (Hg) in aqueous and solid environmental samples by Method 7470A, 7471A, 245.1.

2.0 APPLICABLE MATRIX

2.1 Groundwater, surface water, TCLP's, leachates, drinking water, filtered collection wastes, sand, rock, concrete, soil and sludge samples

3.0 METHOD DETECTION LIMITS

3.1 The method detection limit (MDL) is the lowest concentration that can be measured for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". The laboratory maintains MDL studies for analyses performed, and are verified annually.

3.2 Instrument Detection Limits (IDLs) are generated for mercury quarterly for each instrument as specified in CLP. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance, without the introduction of method variance.

3.3 Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error, client requirements, values specified by the EPA methods or other project and client requirements. Wherever possible, reporting is limited to values approximately 3 – 5 times the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement. The reporting limit for mercury analysis in an aqueous matrix is 0.2 ug/L while the reporting limit for solid samples is typically 0.025 µg/g.

4.0 SCOPE AND APPLICATION

4.1 This method is used for the determination of Hg in aqueous and solid samples. This procedure is used to analyze organic and inorganic mercury in drinking water, surface water, waste, and saline waters, both domestic and industrial wastes.

4.2 This method is based upon SW-846, 3rd edition method 7470A/7471A and also conforms to the EPA Environmental Methods Management Council's "Guidelines and Format for Methods to Be Proposed at 40 CFR, part 136" (Method 245.1).

4.3 This method is for the determination of Hg in the range of 0.2 ppb to 10.0 ppb and may be extended to higher levels by selection of a smaller sample size or serial dilution on existing samples.

4.4 This method is "performance based." The laboratory is permitted to modify the Method to overcome interferences or lower the cost of measurements if all performance criteria are met. On occasion, clients request slight modifications to the SOP. These modifications are addressed on a case-by-case basis and would be written into a Quality Assurance Plan (QAP).

4.5 This method is used only by analysts experienced in the use of the chemical principles outlined in this SOP and who are trained thoroughly in the sample handling and instrumental techniques described in this method.

5.0 SUMMARY OF THE TEST METHOD

5.1 Mercury in samples is first oxidized to the Hg^{2+} oxidation state under strongly acidic conditions and near boiling temperatures. Potassium permanganate ($KMnO_4$) is added to eliminate the possible interference from sulfide. Potassium permanganate is reduced with hydroxylamine hydrochloride ($NH_2OH{\bullet}HCl$) later.

5.2 This cold-vapor atomic absorption method is based on the absorption of radiation at the 253.7-nm wavelength by Hg vapor. The Hg is reduced to the elemental state and aerated from solution on-line. The Hg vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Absorption is measured as a function of Hg concentration.

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5.3 Quality is assured through the use of preparation blanks, laboratory control sample/laboratory fortified blank, serial dilution, replicate samples and matrix spike samples.

6.0 DEFINITIONS

6.1 Total Mercury
All oxidizable mercury forms and species found in an unfiltered aqueous solution. This includes, but is not limited to, Hg (II), Hg (0), strongly organo-complexed Hg (II) compounds, adsorbed particulate Hg, and several tested covalently bound organo-mercurials.

6.2 Dissolved (Soluble) Mercury
All oxidizable mercury forms and species found in the filtrate of an aqueous solution that has been filtered through a 0.45 micron filter and acidified to a pH < 2.

6.3 Any other definitions contained within this document are standard definitions as defined by the STL Buffalo Laboratory Quality Manual.

7.0 INTERFERENCES

7.1 **Contamination Control:** Any object or substance that contacts the sample is mercury free and free from any material that may interfere with the analysis of mercury. Although contamination control is essential, personal health and safety remain the highest priority. Section 8.6 of this method gives suggestions and requirements for personal safety.

7.1.1 **Avoiding Contamination:** The best way to control contamination is to completely avoid exposure of the sample to contamination in the first place. Avoiding exposure means performing operations in an area known to be free of any traces of mercury. Two of the most important factors in avoiding and/or reducing sample contamination are (1) an awareness of potential sources of contamination and (2) strict attention to the work being done.

7.1.2 **Minimize Exposure:** The apparatus and/or glassware that will come into contact with the samples, blanks, or standard solutions are to be opened or exposed only in a clean area of the lab. When any relevant materials, glassware or instruments are not being used, cover with a plastic liner or remove from the area of analysis to avoid accidental exposure.

7.1.3 **Clean Work Surfaces:** Before a given batch of samples is processed, the analyst makes certain that all work surfaces in the hood, the bench and other areas are clean.

7.1.4 **Wear Gloves:** Sampling personnel wear clean, nontalc gloves during all operations involving handling of any instrument, glassware, samples or blanks. Only clean gloves may touch the instruments. If another object or substance is touched, the gloves must be changed before resuming work on the instrument. If it is suspected that gloves have become contaminated, work must be halted, the contaminated gloves removed, and a new pair put on.

7.1.5 **Use Mercury-Free Materials:** All materials used for the preparation and analysis of mercury at ambient water quality criteria levels must be non-metallic, free of material that may contain metals, or both.

7.1.6 **Containers:** Each new container type is tested before use, because Mercury vapors can diffuse in or out from certain type of materials, resulting in results that are biased high or low.

7.1.7 **Contamination by Carryover:** Contamination may occur when a sample containing a low concentration of mercury is processed immediately after a sample containing a high concentration of mercury. When an unusually concentrated sample is encountered, the cleaning time is extended before proceeding with next sample. To avoid this, samples that are known, or at least suspected, of having the lowest mercury content will be analyzed first.

7.1.8 **Contamination by Samples:** Significant laboratory or instrument contamination may result when untreated effluents, in-process waters, landfill leachates and other undiluted samples containing concentrations of mercury greater than 100 ppb are processed and analyzed. Samples known or suspected to contain Hg concentrations greater than 100 ppb will be diluted prior to bringing them into the laboratory, when possible.

7.1.9 **Contamination From Reagents:** Contamination can be introduced into samples from the method reagents used during processing and analysis. Reagent blanks are monitored using method blanks in each batch. When a reagent is suspected to be impure, it will be analyzed. If the blank is lower than MDL, that reagent can be used.

7.2 **Interferences**

7.2.1 The main source of chemical interference comes from the presence of high levels of sulfides. This is removed by the addition of potassium permanganate. Other chemical interferences include chlorides, gold, iodides and certain organic solvents, such as acetone and hexane. Also copper interferes at levels > 10 ppm.

7.2.2 Physical means of interference include damaged optic tube (including, but not limited to cracks, and moisture), and air bubbles trapped in the samples. Any of these can result in skewed reading and incorrect measurements.

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8.1. **SAFETY**

8.2. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.3. The toxicity or carcinogenicity of each chemical used in this method has not been precisely determined; however, each compound is treated as a potential health hazard. Exposure to these compounds is reduced to the lowest possible level.

8.4. The laboratory is responsible for maintaining a current awareness file of OSHA regulations for the safe handling of the chemicals specified in this procedure. OSHA rules require that a reference file of material safety data sheets (MSDSs) are made available to all personnel involved in these analyses. All MSDS's may be viewed on our internet under Quick Links.

8.5. **SPECIFIC SAFETY CONCERNS OR REQUIREMENTS**

8.5.1. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH may react violently when acids are added.

8.6. **Personal Safety**

8.6.1. Chronic mercury exposure may cause kidney damage, muscle tremors, spasms, personality changes, depression, irritability and nervousness. Organo-mercurials may cause permanent brain damage. Because of the toxicological and physical properties of Hg, only highly trained personnel thoroughly familiar with handling mercury standards should handle pure standards.

8.6.2. As recommended, the laboratory purchases a dilute standard of Hg so that its use won't compromise the health and safety of the analyst. When samples known or suspected of containing high concentrations of mercury are handled, all operations are performed in a controlled area of the laboratory, preferably in a glove box or fume hood with adequate airflow and ventilation.

8.6.3. Disposable plastic gloves, apron or lab coat, safety glasses or mask and a fume hood adequate for the digestion of acidic mediums is used.

8.6.4. Hands and forearms must be washed thoroughly after each manipulation and before breaks.

8.6.5. Mercurial vapors pass through a column of activated carbon or a filter trap containing gold or sulfur in order to sequester mercury vapors away from the analyst.

8.6.6. Broken glassware is immediately cleaned up and discarded in the proper waste container.

8.6.7. All acids are to be handled with extreme care.

8.7. Perform Under a Hood

8.7.1. Whenever samples are opened, dispensed, or diluted, the analyst works under a hood suitable for the task at hand. The hood is periodically monitored to ensure its proper functioning and airflow requirements. All digestion of acidified samples is conducted inside of the hood.

8.8. Spill Response and Decontamination

8.8.1. Personnel can use mild soap with plenty of scrubbing in order to decontaminate skin. In the case of open wounds, professional help is to be sought immediately.

8.8.2. All glassware, tools and surfaces are cleaned with sulfur powder in order to reduce any mercury present to non-volatile mercury sulfide. Washing the surface with reagent water will complete the cleansing process.

8.8.3. The laboratory contains a mercury spill kit in case of serious mercury exposure. The kit is located in an area familiar to all that work in the laboratory, in the cabinet under the sink.

8.9. Special Attention of Instruction

8.9.1. While this procedure does call for the trace analysis of mercury in extremely small levels, it is still possible to be exposed to extremely toxic levels of mercury during normal laboratory conditions. Mercury is at it most toxic when it is allowed to enter the bloodstream, therefore, any analyst who has an open wound or other such injury should take special care in avoiding mercury exposure.

8.9.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

8.10. **Primary Material Used**

8.10.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling(Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and

			permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

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9.1 All equipment and supplies will be free of trace mercury, or at least at a level well below the MDL of the method. All reusable equipment and supplies are soaked in a dilute (10%) nitric acid solution overnight prior to use and rinse with the same after their use.

9.2 Supplies for Digestion of Samples

9.2.1 50 mL Digitubes with graduated marks at 10 mL, 20 mL, 25 mL, 30 mL and 50 mL. Caps are required as well. 30 mL mark must be verified per lot since it is not certified by manufacturer. Verification is accomplished by taking five tubes and filling each with reagent water to the 30 mL mark by eye level. The average of all five by weight must be within 1%. This is logged into a spreadsheet and placed into a binder in the Mercury laboratory.

9.2.2 Hot Block digestion center capable of maintaining temperatures exceeding 95°C. Digestion tube racks are needed in order to store and remove the samples from the hot block.

9.2.3 500 mL volumetric flasks.

9.2.4 2000 mL volumetric flasks.

9.2.5 Eppendorf pipets. Preferable one pipet for each standard calibration to be run. Minimally, a 0.05 - .2 mL, 0.1 – 1.0 mL, 0.5 – 2.5 mL and a 2.0 – 10.0 mL are needed. The pipets should be verified weekly and calibrated quarterly (AGP-Pipet-01).

9.2.6 Timer

9.2.7 Analytical Balance for weighing soil samples – to be calibrated before each use (Balance is accurate to ± 0.1 mg)

9.2.8 Weighing boats and spatulas for soil samples

9.3 Supplies for the Analysis of Samples

9.3.1 15 mL glass sample tubes for analysis

9.3.2 Leeman AP/PS200II Mercury Analysis System Auto-Analyzer

9.3.3 Computer and printer for analysis and report writing

10.0 REAGENTS AND STANDARDS

10.1 Reagents:

10.1.1 Reagent Water: Deionized water starting from a purified source. Water will be monitored for Hg, especially after ion exchange beds are changed.

10.1.2: Silicon (IV) Oxide

10.2 Stock Acids

10.2.1 Nitric Acid
Concentrated trace metal grade.

10.2.2 Sulfuric Acid – Water Samples Only
Concentrated trace metal grade.

10.2.3 Hydrochloric Acid – Solid Samples Only
Concentrated trace metal grade.

10.3 Prepared Reagents and Prepared Stock Standards

10.3.1 All reagents are labeled accordingly at the time of preparation. This label must include the reagent name, the date it was made, the analyst who made it and its expiration date. All reagents made must be noted in the regent logbook containing all the pertinent information listed above. All expiration dates must conform to the earliest date of any of the chemicals used in the preparation of the reagent.

10.3.2 5% Potassium Permanganate ($KMnO_4$) Solution
Prepare by dissolving 100 grams of $KMnO_4$ in 2000 mL of reagent water. The solution has a shelf life of six months.

10.3.3 5% Potassium Persulfate ($K_2S_2O_8$) Solution – for aqueous samples
Prepare by dissolving 100 grams of $K_2(SO_4)_2$ in 2000 mL of reagent water. This solution has a shelf life of six months.

10.3.4 Sodium Chloride-Hydroxylamine Hydrochloride ($NaCl\text{-}NH_2OH\bullet HCl$) Solution
Prepare by dissolving 240 grams of NaCl and 240 grams of NH_2OH-HCl in 2000 mL of reagent water. This solution has a shelf life of six months.

10.3.5 Stannous Chloride ($SnCl_2$)
Prepare by dissolving 100 grams of Stannous Chloride in 10% Hydrochloric acid. Dilute to 1000 mL mark. The solution has a shelf life of one month. It is stored in a tightly closed container so that exposure to the air is kept at a minimum. This solution should also be kept away from any mercury standard, reagent used in digestion or field sample.

10.3.6 10% HCl

Measure 2500 mL of concentrated HCl to a 25.0 L carboy half-filled with reagent water and bring to the mark with reagent water.

10.3.7 Matrix Blank Solution
Measure 40 mL of concentrated HNO_3 , 80ml of concentrated H_2SO_4, 200 ml of $KMnO_4$, 80 ml of $K_2S_2O_8$ and 40 ml of Hydroxlyamine Hydrochloride to a 2000 mL container half-filled with reagent water and bring to 2000 mL with reagent water.

10.3.8 20,000 ng/mL of Hg #1 stock standard (Intermediate Standard # 1)
Measure 10 mL of concentrated HNO_3 to a 500 mL volumetric flask half-filled with reagent water. Measure 5.0 mL of 1000 µg/mL Hg #1 purchased standard to this flask and bring to the mark with reagent water. This standard expires in 6 months or when the original purchased standard is expired, whichever comes first.

10.3.9 20,000 ng/mL of Hg #2 stock standard (Intermediate Standard # 1)
Measure 10 mL of concentrated HNO_3 add to a 500 mL of volumetric flask half-filled with reagent water. Measure 5.0 mL of 1000 µg/mL Hg #2 purchased standard add to this flask and bring to the mark with reagent water. This standard expires in 6 months or when the original purchased standard is expired, whichever comes first.

Note: Hg#1 and Hg#2 are purchased from two separate quality sources with certificates of analysis kept on file in the Mercury laboratory.

10.4 Working Standards: Working Standards are prepared daily

10.4.1 Intermediate Working Standards: 2000 ppb #1

Add 1.0 mLs of concentrated HNO_3 a 50 mL Volumetric Class A glass flask half-filled with reagent water. Measure 5.0 mL of "20,000 ppb #1" standard solution to the digestion cup and dilute to the 50 mL mark with reagent water.

10.4.2 Intermediate Working Standards: 2000 ppb #2
Same as 10.4.2 except that "20,000 ppb #1" should be replaced with "20,000 ppb #2".

10.4.3 Working Standard #1 (100 ppb #1)

10.4.3.1 Add 1.0 mLs of concentrated HNO_3 a 50 mL Volumetric Class A glass half-filled with reagent water. Measure 2.5 mL of "2000 ppb #1" standard solution to the cup and dilute to the 50 mL mark with reagent water.

10.4.3.2 This solution is to be used for the preparation of the standard curve for calibration of the instrument, and for the addition of the mercury spikes to the samples and laboratory fortified blanks (LFB) and the aqueous laboratory control sample (LCS).

10.4.4 Working Standard #2 (100 ppb #2)

10.4.4.1 Same as 10.4.3 except that "2000 ppb #1" should be replaced with"2000 ppb #2".

10.4.4.2 This standard is to be used for the initial calibration verification solution (ICV) and the continuing calibration verification solution (CCV).

10.5 TCLP Spike: Prepared every six months or when 20,000ppb (IS #1) expires, whichever comes first.

Add 5.0 mLs of concentrated HNO_3 into a 100 mL Volumetric Class A glass half-filled with reagent water. Measure 6.675 mL of 20,000ppb (IS#1) standard solution to the volumetric and dilute to the 100 mL mark with reagent water. The final concentration will be 1335 ppb.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 All aqueous samples are preserved with nitric acid to pH<2 upon collection. Non-aqueous samples are stored at 4°C and are analyzed as soon as possible. All non-CLP samples have a maximum holding time of 28 days. CLP samples have a maximum holding time of 26 days. Samples must be analyzed within these holding times in order to ensure accuracy of reportable results.

12.0 QUALITY CONTROL

Refer to STL's Corporate Quality Assurance Plan for general information and more specific detail. Often project-specific quality assurance documents will provide overriding criteria to that presented below. Those criteria depending on project-specific data quality objectives may be more or less stringent than STL's QAP or the following criteria. The following criteria are subsequently presented as the minimum criteria of those criteria deemed applicable in the absence of project-specific DQO's.

12.1 Method (Preparation) Blank
One method blank per preparation batch (≤ 20 samples) must be prepared and analyzed. This is to show that the reagents, supplies and the sample preparation procedure are free

from contamination. Also, TCLP batches will have an associated EBLK (extractor blank) that will accompany a set of TCLPs. This must also be analyzed as a sample.

12.1.1 A water MBLK is prepared by adding 30 mls of reagent water to a digitube.

12.1.2 For a soil MBLK, weigh 0.6 grams of Silicon (IV) Oxide (used as a soil substitute matrix) and place in a 50 ml digitube. Add approximately 20 mls of reagent water. Carry the MBLK through the entire digestion process (i.e.: treat as a sample).

12.2 Laboratory Control Sample (LCS)

12.2.1 One LCS per preparation batch must be prepared and analyzed. The LCS is used to check for the accuracy of the preparation and analysis.

12.2.2 Aqueous LCS (including AFCEE and USACE) is prepared by fortifying a blank solution with a known amount of Hg (1.0 mL of 100 ppb #1 into the digestion with a final volume of 50 mL) to have a final concentration of 2.0 ppb. For TCLP samples, spike 400 mL of the Extractor Blank with TCLP mercury spike for a final concentration of 4.0 ppb.

12.2.3 Soil LCS is prepared by weighing out a set amount per the manufacturer of the certified reference soils. (The amount of sample that is needed might be changed from lot to lot). The check standard expected value is whatever amount of mercury that is put into the sample container. For example, if the standard is known to have a value of 2170 ng/g of mercury and 0.0953 g is added into a final volume of 50 mL. 2170 ppb times 0.0953 grams divided by 50 grams (mL) gives a final concentration of 4.14 ppb. The goal is to have the LCS set at the same value as MS/SD, 4.00 ppb. The certified value of Hg in soil is provided by the manufacturer and the certificate of analysis is kept in lab. . For AFCEE and USACE a water matrix spike is used with the usage of Silicon oxide weighed out to .6 g. The concentration is set at 2.0 ppb. (1.0 mL of 100 ppb #1 into the digestion with a final volume of 50 mL)

12.3 Matrix Spike (MS) and Spike Duplicate (SD)
One sample per preparation batch must be chosen to have a matrix spike and a spiked duplicate. This group comprises a set of three samples, all having the same base sample. The MS or SD is merely another equal aliquot of the sample fortified with a known amount of mercury – 4.0 ppb (use 2.0 mL of 100 ppb # 1). For AFCEE and USACE the concentration is set at 2.0 ppb (1.0 mL of 100 ppb #1 into the digestion with a final volume of 50 mL). Analysis of these samples is also to demonstrate the accuracy and precision and to monitor matrix interferences. TCLP batches do not require any method duplicates but do require that every batch have a spiked sample associated with it.

12.4 Serial Dilution

For each analytical batch, select one typical sample for serial dilution. Dilution test is used to evaluate the matrix effects in determination (1:3 dilution).

12.5 Matrix Duplicate (MD)
When requested by the clients, MD will be performed. MD is simply another aliquot of the sample that is prepared and analyzed in the same manner as the sample.

12.6 Laboratory Fortified Blank (LFB)
This needs to be done only when preparing a batch of 40 CFR 136 samples. The LFB is a reagent blank fortified with a known amount of mercury – 2.0 ppb (use 1.0 mL of 100 ppb #1).

12.7 CRA Preparation Sample
This sample is used to check the linearity at the low end of the calibration curve. It is a reagent blank fortified with a known amount of Hg (use 0.1 mL of 100 ppb #1 in order to have a final concentration of 0.2 ppb).

12.8 Initial / Continuing Calibration Verification (ICV / CCV)
The ICV must be prepared and analyzed before any other sample is analyzed. The CCV is analyzed every 10 samples and at the end to ensure the instrument is still calibrated correctly. Both solutions are reagents blanks fortified with a known amount of Hg (1.0 mL of 100 ppb #2) in order to give a concentration of 2.0 ppb for the CCV's and 1.5 ml of the 100 ppb #2 in order to give a concentration of 3.0 ppb for the ICV.

12.9 Initial / Continuing Calibration Blank (ICB / CCB)
The ICB must be analyzed directly after the ICV and the CCB must be analyzed directly after the CCV. It is simply a reagent blank used to verify that no carryover contamination had accumulated over the analysis of the last ten samples.

13.0 CALIBRATION AND STANDARDIZATION

13.1 A standard curve must be generated each day the analysis is to be done and each of these six standards are digested the same as the samples. Ideally, a new standard curve should be made after twenty-four hours of instrumental use as well.

13.2 Preparation of the Standard Curve
The standard curve must consist of six known mercury standards. Standards are prepared by the addition of aliquots of Hg from working standard 100 ppb #1 directly into sample blanks.

The six standard solutions are to be prepared as follows:

Standard	Final Conc. (ppb)	mLs of 100 ppb #1	Final Volume (mL)	Final Conc. In Aqueous Samples	Final Conc. In Soil Samples

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				(mg/L)	(mg/kg)
1	0	0	50	0	0
2	0.2	0.1	50	0.33	0.017
3	1.0	0.5	50	1.67	0.083
4	2.0	1.0	50	3.33	0.17
5	5.0	2.5	50	8.33	0.42
6	10.0	5.0	50	16.67	0.83

14.0 PROCEDURE

14.1 Once the samples are divided into their individual batching reports and the order to which they will be analyzed has been assessed, sample preparation can begin. The samples, calibrations and standards are mapped out in the logbook for the autoanalyzer. A place for the identification of the sample must clearly be marked in the logbook and referred to every time a sample is about to be prepped. The solid samples cannot be run with the aqueous samples. Each of these runs must be carried out and analyzed independently from the other type of sample and test.

14.2 Once the identification markings of each sample and QC have been made, a Digitube for each sample and QC will be made corresponding to each identification number. All the standards for the calibration curve, blanks, ICV, CCV, CCB, ICB, LFB, CRA, and LCS are prepared by first adding 30 mL of reagent water to their Digitubes. The appropriate amount of spike from the appropriate working solution is added (see Section 12.0 for the amounts of spiking solution to be used).

14.3 All the aqueous field samples are prepped in a similar manner. Pour 30 mLs of each sample into the appropriate Digitube. Be sure to invert the sample bottle several times before pouring off the sample. Also, be sure to use the appropriate bottle when dealing with sample, MD, MS, SD groups. Some clients sent specific bottles for each of the samples in this group. However, most of the time, the analyst will have to use the same sample bottle for all three.

14.4 All of the solid field samples are prepped in a similar manner. Pour about 20 mLs of reagent water into each Digitube. Empty the solid sample in a clean container. Quarter the sample twice and homogenize the sample. Weigh out a total of 0.6 grams. Place the total amount of soil into the appropriate Digitube for digestion. Also, be sure to use the appropriate bottle when dealing with sample, MD, MS, and SD groups. Some clients sent specific bottles for each of the samples in this group. However, most of the time, the analyst will have to use the same sample bottle for all three.

14.5 Next, spike the appropriate MS and SD samples.

14.6 To all the aqueous samples, all mercury standards and each QC sample for the aqueous batch add 2 mLs of concentrated sulfuric acid, 1 mL of concentrated nitric acid, 5 mLs of the 5% potassium permanganate solution and 2 mLs of the potassium persulfate solution.

14.6.1 For soil samples, add 3.0 mL of hydrochloric acid and 1.0 mL of nitric acid. At this time, you place the soil samples and their respective QC on the hot block for two minutes. After this is complete, you proceed with the addition 10 mL of potassium permanganate. Note: no potassium persulfate is needed for the digestion of soil samples.

14.7 The sample, standards and QC should remain a purple color for at least 15 minutes after adding the potassium permanganate. If the sample turns clear, add an additional 5 mL of the potassium permanganate. If this is required, you must add an additional 5 mLs of potassium permanganate to every sample, including QC. If the purple color fades once again, re-prep the sample using a lower amount of sample and note the dilution required.

14.8 Cap the Digitubes loose enough so that any pressure build-up can be evacuated but also tight enough so that mercury lose due to the digestion process will be minimized.

14.9 Place the racks of Digitubes on the hot block set at 110°C for two hours for aqueous samples and thirty minutes for soils.

NOTE: The actual temperature in digestion cups is 95°C (±3°C) although the setting is approximately 110°C. This is verified.

14.10 Remove the racks and let cool to room temperature (approximately 30 minutes, however, a fan may be used to speed up the process).

14.11 One at a time, uncap each sample/QC and add 2 mL of the sodium chloride-hydroxylamine hydrochloride solution to the Digitube. Allow the bubbling to subside and add enough reagent water to equalize the volume at 50 mLs exactly.

14.12 Cap the Digitube tightly and shake vigorously for two to three seconds. Vent the Digitube. Uncap if the samples are to be run immediately or leave capped if the analysis will be in the future.

14.13 A brown residue of manganese dioxide may remain on the bottom or sides of the Digitube. It is of no concern.

14.14 Analysis must be performed within 24 hours of digestion or the sample will need to be re-digested.

14.15 Some aqueous samples and most solid samples will contain sediments that may interfere with the analysis. These samples are filtered.

14.16 The following steps are used in the direct analysis of the mercury content and may be done at a later time after digestion. (Within 24 hours the latest).

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14.17 Note: This procedure outlines the basic steps needed in order to carry out the analysis of mercury in the field samples. For specific details as to the maintenance and operations of the Leeman analyzer, please refer to the user's guide manual. For this SOP it will be assumed that all the software is configured to the way the analyst will want the computer to run and generate reports. Also, it is assumed that the calibration standards spike quantities and check standard values along with their percent deviations are entered according to the user's needs. Suggestion: Check Standard #1 must be a blank, with an acceptance below the EQL/PQL and can be used for the ICB, CCB and Prep Blank. Check Standard #2 can be used for the ICV with its value at 3.0 ppb with an acceptance of ±5% of the true value. (according to all 40 CFR samples). Check Standard #3 can be set to be the CCV with its value at 2.0 ppb with an acceptance of ±10% of the true value. Check standard #4 can be the CRA with its value at 0.2 ppb with an acceptance of ±50% of the true value.

14.18 Before proceeding, be sure the wash basin is clean and filled with 10% hydrochloric acid solution. Make sure that the argon gas tank valve is open and the stannous chloride bottle is connected to the analyzer. The stannous chloride reduces the mercuric ion to liberate elemental mercury vapor. The stannous chloride is added automatically through instrument tubing and is continuously flowing throughout the entire run at the pump rate of 7 ml/min. From windows, click on the Win Hg icon to bring up the Win Hg Runner. First, select 'perform maintenance' from the 'utility' menu and follow the instructions to perform any maintenance required. Click on the 'Control' tab. Turn the lamp, pump, and gas on. Allow the system to clean itself and warm up during the following steps.

14.19 Go to the 'File' menu and select 'New Dataset'. Enter a desired name for the run file. This is the file that will contain all the data needed when you wish to generate a report of your work.

14.20 Sample Data Entry

14.20.1 Select the rack editor icon from the task bar and create the name of the rack(s) you will need for this run. Here you will enter in all the data needed for the current run (e.g. sample ID's). All the samples go into the sample cups tray, while all the check standards have a place for them in the standards rack next to the calibration standards. If the check standards are listed as suggested in step 14.17, then the following order of sample arrangement and macro layout will apply:

14.20.2 Cup One: Sample 1 -- Macros: C2 C1C4 C3 C1 CP -- this will tell the computer to run the ICV, ICB, CRA, CCV, and CCB before running the first sample.

14.20.3 Cups 2 – 10 are for samples with cup 10 having the macros: C3 C1, which will instruct the analyzer to read the CCV and CCB after the first ten samples have been analyzed

14.20.4 Cups 11-20 samples can be added to the rack. If the LCS/LFB falls on any tenth the macros are: S P C3 C1 (for a water / soil run).

14.20.5 Continue as written: Ten samples with the tenth sample having the macros: C3 C1.

14.20.6 Every batch must have an LCS/LFB. A batch can consist of up to twenty samples. Macro for LCS/LFB is S P.

14.20.7 The last sample in the batch must have the macros: C3 C1.

14.20.8 If dilutions are required then add them to the end of the rack (making sure to include any check standards as appropriate). It is a good practice to indicate the dilution factor in the 'Extended ID' column of the rack editor.

14.20.9 Example of a run with sample and QC layout.

ICV
ICB
CRA
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
7 samples
↓
Dilution (Digestion ID) L (1:3)
LCS/LFB (can fall anywhere in the batch depending on the # of samples)
MBLK
CRA (only in CLP or upon client request)
CCV
CCB

14.21 Next click on the 'Sample' tab in the Win Hg Runner. In the 'Autosampler Run' section select the appropriate rack setup by selecting the name of the rack(s) being used for the run. Specify the beginning and ending cup for the rack(s).

14.22 Pour off the samples and QC into the appropriate sample holders. To run the calibration curve click on the 'Standards' tab. Select the desired standards (typically 1-6) and the number of repetitions wanted (typically 1-3) and click on 'Stnd Auto' to run the standards.

To see the calibration curve you need to click on the Database icon on the task bar. This will open the Win Hg Database program. From here, click on the 'Calibration Curve' tab to view the curve. The curve must have a correlation coefficient ("rho" value) of no less than 0.995, but it is recommended that you strive to get a value of greater than 0.9995. To accept the curve for use, click on the 'Accept' button.

14.23 Return to the Win Hg Runner. Check to make sure the desired dataset for the run is selected in the 'Dataset' field. Go to the 'Sample' tab and click 'Run Auto'. This will begin the analysis.

14.24 A full rack of 88 cups including all QC will take about 2.5 – 3.0 hours.

14.25 Once the run is complete go to the Win Hg Database program. Click on the 'Report' tab. Select the appropriate data for the report by clicking a combination of the 'Batch List' and 'Records List' sections. Then click 'Generate Report'. Next, reports can be generated on screen (viewable via the 'Viewer' tab), to the printer, or to a disk file.

14.26 Verify that every QC and duplicate is in range. The following table lists the acceptable ranges:

QC cup	3rd Edition	40 CFR	USACE
Prep Blank	< Reporting Limit	< MDL	< ½ Reporting Limit
ICV	±10%	±5%	±5%
CCV	±20%	±10%	±10%
ICB/CCB	< Reporting Limit	< Reporting Limit	< MDL
LCS-Water	±20%	N/A	±20%
LCS-Soil	Industry specs	Industry specs	Industry specs
LFB	N/A	±15%	N/A
CRA	±50%	±50%	N/A
Dilution Test	±10%	±10%	±10%
Spiked Sample	±20%	±20%	±20%
Duplicate Sample	±30%	±30%	±20%

The computer's software will inform the analyst if any of the check standards values is out of control.

14.27 If any of these values are out of control, see sections 18.0 and 19.0 for corrective actions. To be on the safe side, do not dispose of any sample or QC until the run has finished and the analyst is sure that all the QC standards are in the ranges required.

14.28 If all the samples are in range of required parameters, then the samples may be discarded in the appropriate waste containers. Allow the instrument to flush itself out with the 10% HCl wash solution for at least ten minutes. After this time period, in the Win Hg Runner, go to

the 'Sample' tab. In the 'Move Tip' section click on the 'To Air' button to remove the tip from the 10% HCl wash solution. Unscrew the top of the stannous chloride solution and refit the bottle with a standard closed-top cap. Allow the pump and gas to run for at least ten minutes in order to flush out the stannous chloride and HCl wash solutions from the system.

14.29 After the cleaning has finished select the 'Control' tab, in the Win Hg Runner, and turn off the gas, pump and mercury lamp.

14.30 Close both the Win Hg Runner and Win Hg Database. This will return the computer to the windows desktop. Shut down the computer as normal. Turn off the monitor and printer.

14.31 Turn off the argon gas value.

14.32 Release the five pump latches at the front of the analyzer.

14.33 Complete paperwork

15.0 CALCULATIONS

15.1 Wet-weight basis:

Sample Concentration (mg/kg)= $C \times V/W$
Where: C = concentration in extract (mg/L)
V = Volume of the digestate (L, 50 mL = 0.05 L)
W = Weight of sample aliquot (not dried) extracted (g × 0.001 = kg)

15.2 To report percent solids in solid samples, calculate as follows:

% Solid (S) = $DW/WW \times 100$
Where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

15.3 Dry-weight basis:

Sample Concentration (mg/kg)= $(C \times V)/(W \times S)$, *or*
Sample Concentration (mg/kg)= $C \times V/WW$

15.4 The following calculations are illustrated:

15.4.1 Matrix Spike calculation

The formula for calculating the post spike recovery is:

Recovery (%) = $(S_2-S_1)/SA \times 100$

Where,
S_2 = the spiked sample reading
S_1 = the sample reading
SA = the spike added

Sample Calculations:

A sample gave a reading of 50.0 µg/L. The sample was spiked with 50 µg/L and gave a reading of 95 µg/L. The recovery is *(95-50.0)/50×100%=90%*

15.4.2 Matrix Duplicate calculation

The formula for calculating the relative percent difference (RPD) is:

$RPD\ (\%) = 2\times|D_1-D_2|/(D_1+D_2)\times100$
Where,
D_1 = first sample value
D_2 = second sample value

Sample Calculations:

A sample gave a reading of 50.0 µg/L. The MD gave a reading of 40 µg/L. The RPD is *2×|50-40|/(50+40) ×100%=22%*

The other calculations necessary for this procedure are automatically done by the system's software.

16.0 METHOD PERFORMANCE

16.1 Method Detection Limit Studies (MDLs) are performed on an annual basis. Instrument Detection Limit Studies (IDLs) are performed quarterly. See STL SOP AQA-MDL-20 for the criteria used to perform the MDL studies.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 See section 14.26 for a table listing all the required ranges for the values of the QC samples.

18.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL DATA

18.1 If any one CCV falls out of range, the ten samples before it and the ten samples after must be reanalyzed. Possible redigestion may be needed if further analysis shows the QC to be at unacceptable limits. If the LCS falls out of range, the batch associated with the LCS must be reanalyzed. Again, redigestion may be necessary. A new standard curve will be

analyzed if the ICV falls out of control or any one of the QC samples are unable to be analyzed within the acceptable control limits.

18.2 As always, check for any sources of contamination as this may cause QC samples to be too high.

18.3 Also, be sure to keep instrument maintenance logbooks up to date as a worn tube or faulty optics lens will cause erroneous readings.

18.4 A job exception report form may need to be filed if extensive problems are noted within any one run. The analyst performing the run completes these forms.

18.5 When an out of control situation occurs, the analyst must use his/her best judgment and use any available resources to determine the corrective action to be taken. The analyst may need to seek immediate assistance from the supervisor, laboratory director, project manager, QA personnel or other experienced members of the staff if he/she is uncertain of the proper course of action. The test may need to be stopped until the problem is corrected since the problem may be instrumental and not chemical. Out of control data will never be released without the approval of the Supervisor, QA Manager, or Laboratory Director.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

19.1.1. Holding times exceeded

19.1.2. Insufficient sample volume for re-digestion

19.1.3. Re-digestion required due to method blank or MSB/LCS failure

19.1.4. In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

20.0 WASTE MANAGEMENT / POLLUTION PREVENTION

20.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention." All samples, reagents and laboratory wastes must be handled with caution. Appropriate safety measures are employed in detail in STL Buffalo's Laboratory Safety Manual and Chemical Hygiene Plan.

20.2 Waste Streams Produced by this Method

- Acid Waste – The waste generated will have nitric acid. Therefore, it will be disposed of as "AN" waste.

21.0 REFERENCES

21.1 Method 7470A – Mercury in Liquid Waste

21.2 Method 7471A – Mercury in Solid Waste

21.3 40 CFR Part 136 (Revision B), "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act" U.S. Environmental Protection Agency.

21.4 "Method 1631, Revision B: Mercury in Water by Oxidation" (40 CFR 136, Revision B) U.S. Environmental Protection Agency, May 1999.

21.5 EPA 600/4-79-020 Methods 245.1, Revision B; SW-846, 3rd Edition, Method 7470A

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Exhibit A: Possible positioning of the computer/analyzer setup.

22.2 Table 1: Water Digestion Batch Sheet.

22.3 Table 2: Soil Digestion Batch Sheet.

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 10.1.2: Added Silicon (IV) Oxide

23.2 Section 12.1.1 & 12.1.2: Added water and soil MBLK preparation instructions

23.3 Section 19.0: Added more specific information for handling Out-of-Control Data

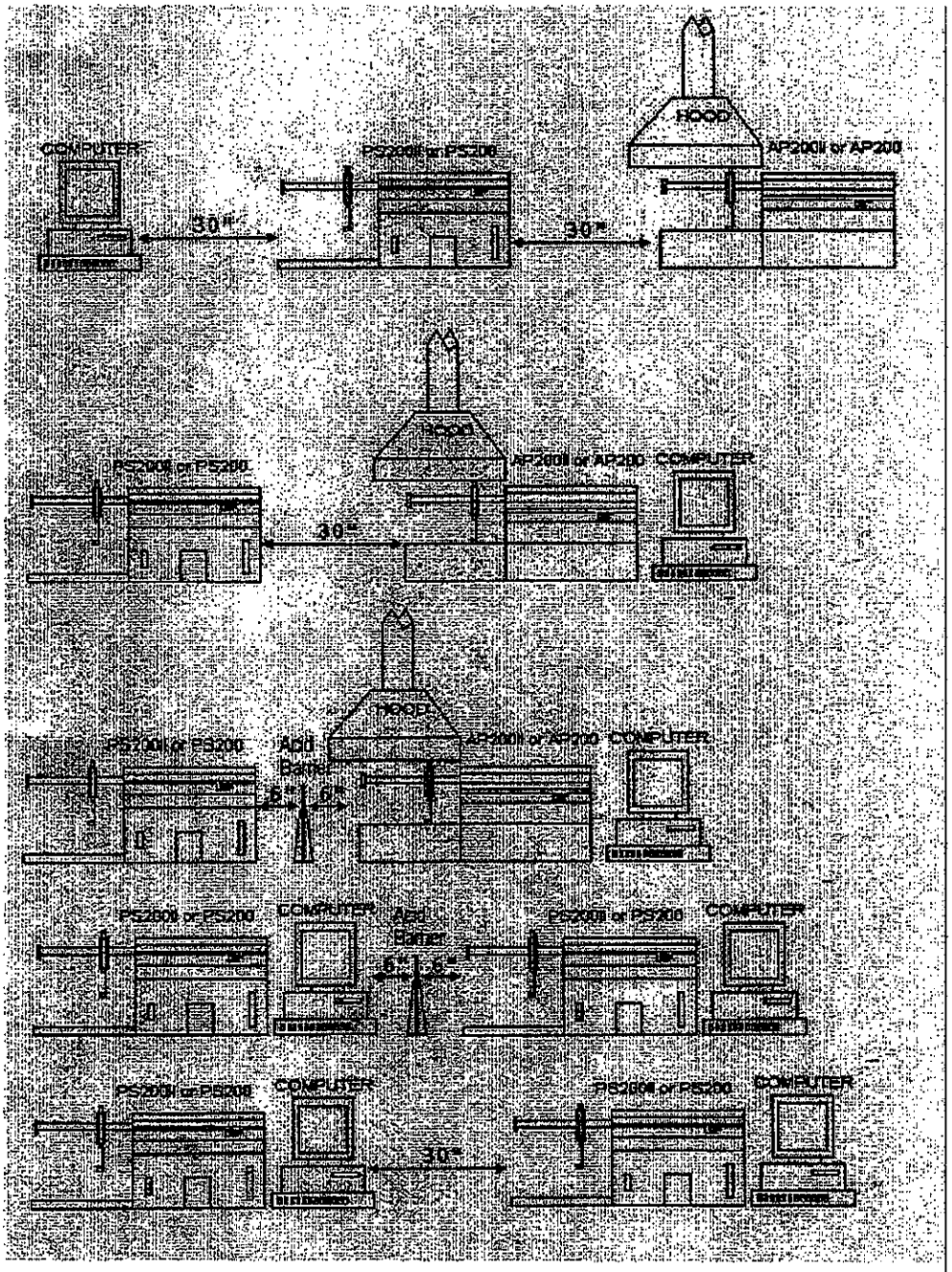
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Exhibit A: Possible positioning of the computer/analyzer setup.



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Table 1: Water Digestion Batch Sheet

STL Buffalo
Date: 09/30/2003
Time: 09:21:15

METALS DIGESTION LOG
A3B10166 - 09/10/2003 NON-CLP WATER Hg Wl (Closed)
AQUEOUS

Page: 1
Rept: AN0764

Date	Time	Dig Emp	Johno	Sample ID	Bot ID	Sample Type	Digest ID	Vl	Analysis Type	Initial Vl (ml)	Final (ml)	Color Before/After	Clarity Before/After	Textur
09/10/03	08:00	AJY	A03-8568	A3856801	A	FS	AD343607	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8580	A3858001	A	FS	AD343608	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8591	A3859101	A	FS	AD343609	A	MERCURY	10.00	50.00			
09/10/03	08:00	AJY	A03-8573	A3857354	A	FS	AD343610	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8573	A3857355	A	FS	AD343611	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8574	A3857454	A	FS	AD343612	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8562	A3856201	A	FS	AD343613	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8562	A3856201MS	A	MS	AD343614	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8562	A3856201SD	A	SD	AD343615	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8562	A3856209	A	FS	AD343616	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8567	A3856701	A	FS	AD343617	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8567	A3856702	A	FS	AD343618	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8567	A3856703	A	FS	AD343619	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8567	A3856704	A	FS	AD343620	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8567	A3856705	A	FS	AD343621	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8567	A3856706	A	FS	AD343622	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY	A03-8569	A3856901	A	FS	AD343623	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY		A3B1016601	A	LFB	AD343624	A	MERCURY	30.00	50.00			
09/10/03	08:00	AJY		A3B1016602	A	MBLK	AD343625	A	MERCURY	30.00	50.00			

Comments: Comments: EPPENDORF USED TO ADD SPIKES;
MA-03A-1404E 2ml
EPPENDORFS USED TO DISPENSE SET VOLUMES:
MA-03C-2304E .1ml
MA-03C-2303E .2ml
MA-03C-2302E .5ml
MA-03C-2301E 1ml
MA-03C-2204E 1.5ml
MA-03C-2203E 2ml

Color: Black Gray Red Yellow
Blue Green Violet Colorless
Brown Orange White
* Redigestion

Clarity: Clear
Cloudy
Opaque

Texture: Fine (powdery)
Medium (sand)
Coarse (large crystals or rocks)

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STL Buffalo
Date: 09/30/2003
Time: 09:21:15

METALS DIGESTION LOG
A3B10166 - 09/10/2003 NON-CLP WATER Hg W1 (Closed)
AQUEOUS

Page: 2
Rept: AN0764

Date	Time	Dig Emp	Jobno	Sample ID	Bot ID	Sample Type	Digest ID	Vl	Analysis Type	Initial Vl (ml)	Final (ml)	Color Before/After	Clarity Before/After	Textur
			MA-03C-2202F 5ml											
			MERCURY BATCH ADDITIONS											
			1.) HgLCS (W) 11-HGL-6											
			2.) Potassium Persulfate 180-0707-K2S2O8											
			3.) Potassium Permanganate 190-0721-KMnO4											
			4.) Stannous Chloride 191-0722-SnCl2											
			5.) Hydroxylamine Hydrochloride 180-0707-HyHy											
			6.) HgLCS (S) 10-HGL-09											
			CONC. NITRIC ACID - J.T. BAKER LOT# 7697-37-2											
			CONC. HCL ACID - J.T. BAKER LOT# X44A17											
			CONC. SULFURIC ACID - MALLINKRODT LOT#5557 X35A10											
			HOT BLOCK TEMP - 95(+-)3 DEG C											
			PREPPED AND SPIKED BY:											
			ANALYZED BY:											

Color: Black Gray Red Yellow
Blue Green Violet Colorless
Brown Orange White
* Redigestion

Clarity: Clear
Cloudy
Opaque

Texture: Fine (powdery)
Medium (sand)
Coarse (large crystals or rocks)

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Table 2: Soil Digestion Batch Sheet

STL Buffalo
Date: 09/30/2003
Time: 09:21:36

METALS DIGESTION LOG
A3B0864 - 8/8/2003 NM-CLP SOIL Hg E1 (Closed)
SOLIDS

Page: 1
Rept: AN0764

Date	Time	Dig Emp	Jobno	Sample ID	Bot ID	Sample Type	Digest ID	Vl	Analysis Type	Initial Wgt (g)	Final (ml)	Color Before/After	Clarity Before/After	Textur
08/08/03	13:00	JMB	A03-7554	A3755401	A	FS	AD337289	A	MERCURY	0.6040	50.00			
08/08/03	13:00	JMB	A03-7554	A3755402	A	FS	AD337290	A	MERCURY	0.6389	50.00			
08/08/03	13:00	JMB	A03-7554	A3755403	A	FS	AD337291	A	MERCURY	0.6537	50.00			
08/08/03	13:00	JMB	A03-7554	A3755404	A	FS	AD337292	A	MERCURY	0.5885	50.00			
08/08/03	13:00	JMB	A03-7554	A3755405	A	FS	AD337293	A	MERCURY	0.5713	50.00			
08/08/03	13:00	JMB	A03-7554	A3755406	A	FS	AD337294	A	MERCURY	0.6526	50.00			
08/08/03	13:00	JMB	A03-7554	A3755407	A	FS	AD337295	A	MERCURY	0.6087	50.00			
08/08/03	13:00	JMB	A03-7554	A3755408	A	FS	AD337296	A	MERCURY	0.6193	50.00			
08/08/03	13:00	JMB	A03-7554	A3755408MS	A	MS	AD337297	A	MERCURY	0.6131	50.00			
08/08/03	13:00	JMB	A03-7554	A3755408SD	A	SD	AD337298	A	MERCURY	0.5996	50.00			
08/08/03	13:00	JMB	A03-7554	A3755409	A	FS	AD337299	A	MERCURY	0.6086	50.00			
08/08/03	13:00	JMB	A03-7554	A3755410	A	FS	AD337300	A	MERCURY	0.5674	50.00			
08/08/03	13:00	JMB	A03-7554	A3755411	A	FS	AD337301	A	MERCURY	0.6696	50.00			
08/08/03	13:00	JMB	A03-7554	A3755412	A	FS	AD337302	A	MERCURY	0.6090	50.00			
08/08/03	13:00	JMB	A03-7554	A3755413	A	FS	AD337303	A	MERCURY	0.6337	50.00			
08/08/03	13:00	JMB	A03-7554	A3755414	A	FS	AD337304	A	MERCURY	0.6125	50.00			
08/08/03	13:00	JMB	A03-7554	A3755415	A	FS	AD337305	A	MERCURY	0.6231	50.00			
08/08/03	13:00	JMB	A03-7554	A3755416	A	FS	AD337306	A	MERCURY	0.6100	50.00			
08/08/03	13:00	JMB	A03-7554	A3755417	A	FS	AD337307	A	MERCURY	0.6209	50.00			
08/08/03	13:00	JMB		A3B0886401	A	LCS	AD337308	A	MERCURY	0.0447	50.00			
08/08/03	13:00	JMB		A3B0886402	A	MBLK	AD337309	A	MERCURY	0.6000	50.00			

Comments: Comments: EPPENDORF USED TO ADD SPIKES:
MA-03A-1404E 2ml
EPPENDORFS USED TO DISPENSE SET VOLUMES:
MA-03C-2304E .1ml
MA-03C-2303E .2ml
MA-03C-2302E .5ml
MA-03C-2301E 1ml

Color: Black Gray Red Yellow
Blue Green Violet Colorless
Brown Orange White

Clarity: Clear Cloudy Opaque

Texture: Fine (powdery)
Medium (sand)
Coarse (large crystals or rocks)

* Redigestion

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SOP No.	Revision No.	Date	Page
AME-MERCURY-50	7	September 26, 2006	28 of 28

TITLE: METHOD# 7470A, 7471A & 245.1 MERCURY PREPARATION AND ANALYSIS

SUPERCEDES: Revision 6

STL Buffalo
Date: 09/30/2003
Time: 09:21:36

METALS DIGESTION LOG
A3B08864 - 8/8/2003 NM-CLP SOIL Hg E1 (Closed)
SOLIDS

Page: 2
Rept: AN0764

Date	Time	Dig Emp	Jobno	Sample ID	Bot ID	Sample Type	Digest ID	Vl	Analysis Type	Initial Wgt (g)	Final (ml)	Color Before/After	Clarity Before/After	Textur
			MA-03C-2204E	1.5ml										
			MA-03C-2203E	2ml										
			MA-03C-2202F	5ml										
			MERCURY BATCH ADDITIONS											
			1.) HgLCS (W)	11-HGL-6										
			2.) Potassium Persulfate	180-0707-K2S2O8										
			3.) Potassium Permanganate	190-0721-KMnO4										
			4.) Stannous Chloride	191-0722-SnCl2										
			5.) Hydroxylamine Hydrochloride	180-0707-HyHy										
			6.) HgLCS (S)	10-HGL-09										
			CONC. NITRIC ACID - J.T. BAKER LOT# 7697-37-2											
			CONC. HCL ACID - J.T. BAKER LOT# X44A17											
			CONC. SULFURIC ACID - MALLINKRODT LOT#5557 X35A10											
			HOT BLOCK TEMP	- 95(+-)3 DEG C										
			PREPPED AND SPIKED BY:											
			ANALYZED BY:											

Color: Black Gray Red Yellow
Blue Green Violet Colorless
Brown Orange White

Clarity: Clear
Cloudy
Opaque

Texture: Fine (powdery)
Medium (sand)
Coarse (large crystals or rocks)

* Redigestion

Attachment V

Laboratory Standard Operating Procedures for Support Equipment Maintenance, Record Keeping, and Corrective Actions of Analytical Balances, Temperature Control Devices, and Reagent Water

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

Once printed, this is considered an uncontolled document

SOP No.	Revision No.	Effective Date	Page
AGP-SuppEquip-02	1	December 18, 2006	1 of 13

TITLE: SUPPORT EQUIPMENT: MAINTENANCE, RECORD KEEPING AND CORRECTIVE ACTIONS OF ANALYTICAL BALANCES, TEMPERATURE CONTROL DEVICES AND REAGENT WATER

Supercedes: Revision 0

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager		12/18/06
Christopher A. Spencer, Laboratory Director		12/19/06
John Schove, Operations Manager		12/18/06

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1.0 SCOPE

1.1. A critical factor in provision of accurate and precise analytical results is the ability to ascertain, within given tolerance, the exact weight, volume, purity or temperature of materials used in various steps of the analytical process.

2.0 SUMMARY

2.1. All piece of analytical support equipment is assessed for accuracy or verification of acceptability on each day of use.

2.1.1. Each analytical and top loading balance is checked each day of use for accuracy relative to ASTM Class "1" weight(s).

2.1.1.1. Reference to NIST Class S weights was superceded in 1978 with the terminology of ASTM Class 1.

2.1.2. Temperature control devices (ovens, incubators, coolers, etc.) require daily verification and recording of the temperature of said devices.

2.1.2.1. Temperature recording is based upon numbered, calibrated thermometers. Calibration of these thermometers is consistent with STL SOP AGP-ThermCal-33.

2.1.3. Deionized water sources throughout STL Buffalo are verified on each day of use.

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2.1.3.1. Conductivity of the water must fall within the specifications for ASTM Type II Reagent Water as listed in ASTM D 1193-77.

2.1.3.2. Water supply indicators of both in-line and off-line conductivity are monitored.

2.2. Generally, each department has one designated person and a back-up person to perform the accuracy and verification checks of the support equipment.

2.3. The checks are documented and maintained in bound logbooks.

3.0 DEFINITIONS

3.1. Analytical Balance: The upper range of weighing capacity is hundreds of grams and the sensitivity of the balance is generally sub-milligram (i.e., 0.001-0.0001 grams).

3.2. Top Loader Balance: Also known as a pan balance. The weighing capacities are significantly greater than the analytical balance but sensitivity is also less (i.e., 0.01 grams).

3.3. Class "1" Weights: High tolerance weights, less than 0.005-0.001% over the weight range of 1 mg to 100 grams. STL Buffalo's Class "1" weights are re-certified annually to further insure accuracy and precision.

3.4. Calibrated Thermometer: Temperature monitoring device verified annually to within specified tolerances of accuracy.

3.5. Reagent Water: Free of substances that interfere with analytical methods. Reagent water is used for dilution of reagents, preparation of inorganic standards and dilution of samples.

3.6. ASTM Type II Water: Type II is a class of reagent water, the specifications for which include requirements for Total Matter, Electrical Conductivity, Color Retention of KMnO4, and Soluble Silica. For purposes of analytical testing, STL Buffalo imposes the specification for Electrical Conductivity. The specifications for Total Matter and Soluble Silica are monitored through the use of method blanks in applicable analyses.

4.0 RESPONSIBILITIES

4.1. QA Department:

4.1.1. Maintain master list of all support equipment and due dates for annual re-calibration of thermometers, balances and weights

4.1.2. Coordinate the maintenance of the Class "1" weights.

4.1.2.1. Each set of Class "1" weights used to check the accuracy of a balance is NIST-certified by the vendor and is re-certified annually.

4.1.2.2. Certificates of certification or re-certification are maintained in laboratory files.

4.1.3. Ensure coordination of the annual service of the balances by the external service company.

4.1.4. Provide bound, paginated, document-controlled logbooks to laboratory departments for documentation of verification and accuracy checks.

4.2. Laboratory Supervisors:

4.2.1. Designate personnel to perform the required verifications and accuracy checks

4.2.1.1. Ensure that all temperature control devices are checked on the required frequency.

4.2.1.2. Ensure monitoring of the water polishing units

4.2.1.3. Ensure that all balances are checked at the required frequency

4.2.2. Review, initial and date the required logbooks on a periodic basis.

4.2.3. Notify QA Dept. if sample integrity may be affected by the equipment failure.

4.2.4. Arrange for service or replacement of malfunctioning equipment

4.3. Technical Analysts:

4.3.1. Ensure that daily accuracy checks have been performed prior to using equipment

4.3.2. Responsible for complete and accurate recordkeeping

4.3.3. Documentation of any non-conformances, corrective actions or routine adjustments to the support equipment.

4.3.4. Notify Supervisor if equipment appears to be malfunctioning

4.3.5. Tag support equipment that fail QAQC requirements as "out-of-service"

5.0 SAFETY

5.1. Eye protection, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled.

6.0 PROCEDURE

6.1. *BALANCES:*

6.2. After turning off any hoods or devices that might affect the balance due to drafts or vibrations, the weighing pans are checked for cleanliness and if necessary cleaned of any residue or debris.

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Supercedes: Revision 0

6.3. For the analytical balance, after the level of the balance is verified, a set of four (4) Class "1" weights are weighed on the balance and recorded in the bound balance logbook.

6.3.1. Weighing of the "1" weights proceeds by beginning with lowest weight and proceeding to the highest weight.

6.3.2. The four (4) Class "1" weights may vary slightly from one department/facility to another but are selected to span the range of the balance and the weighings being performed at that balance.

6.3.3. The four (4) Class "1" weights generally cover the range of milligram to approximately 100 grams.

6.4. For the top loader balances, again after the level of the balance has been verified, a 100 gram Class "1" weight is weighed and recorded.

6.4.1. Additionally, at least once per week a total of three (3) Class "1" weights are weighed and recorded.

6.5. Finally, each balance (analytical and top loader) is serviced and re-calibrated, if necessary, by a qualified outside contractor. This contractor tags each balance with the most recent date of service each time such service is rendered. Outside balance service and calibration, if necessary, is performed on each of STL Buffalo's balances once per year.

6.6. The criteria for balance specifications is 0.1% of true value for the 30, 50 and 100 g weights, and 1% for the 200mg and 20mg weights. If any of the following criteria are exceeded the balance is in need of corrective action:

Weight	Criteria
100g	±0.1g
50g	±0.05g
30g	±0.03g
20g	±0.02g
2g	±0.02g
200mg	±0.002g
20mg	±0.0002g

6.6.1. In the event that a Class "1" weight may be out-of-spec, the deviations seen are large and generally obvious problems with the weights are visible. STL has available a number of certified sets of Class "1" weights which can also be used to resolve out-of-specification events.

6.6.2. Additionally, a balance can be determined to be out-of-specification if the balance can not be leveled, zeroed or indicates uncorrectable drift in its recorded weights.

6.7. In the case of any out-of-specification balance conditions, the department supervisor or operations

manager is notified immediately.

6.7.1. A determination can usually be made as to the likely cause of the condition including the immediate restoration of the balance to useable condition.

6.7.2. The balance is not to be used in any out-of-specification range or condition until the condition has been remediated.

6.7.3. An "OUT-OF-SERVICE" card or label is placed on the balance to warn analysts not to use the balance until the condition has been corrected.

6.8. Corrective actions for these out-of-specification balance conditions include maintenance by STL staff such as cleaning or physical adjustments of the balance. In the event that these minor actions are unsuccessful, the balance will remain out of service until an outside service representative can re-certify the balance for use.

6.9. ***TEMPERATURE CONTROL DEVICES (refrigerators, ovens, incubators)***

6.10. Recordkeeping of temperatures for these devices includes:

6.10.1. Date
6.10.2. Time
6.10.3. Initials of Analyst
6.10.4. Thermometer number
6.10.5. Actual temperature for each control device per record period (daily or twice daily)
6.10.6. Acceptable temperature range for each device
6.10.7. Comment column

6.11. If the analyst finds that the actual (corrected, if necessary, based upon the calibrated thermometer) temperature exceeds the acceptance range (either positively or negatively) he/she is to comment or note the exceedance in the comment column.

6.11.1. A second reading should be attempted within 1-hour to ensure that the 1st reading was not an anomaly due to one of the following conditions.

6.11.1.1. Temperature readings of coolers, incubators or ovens could be inaccurate if the device is not kept closed for 15 minutes prior to taking the temperature.

6.11.1.2. Temperature readings could also be erroneous if the thermometer is cracked or the column is separated.

6.11.2. The analyst may adjust the temperature controls up or down to correct the temperature excursion.

6.11.2.1. Any adjustment of the controls must be documented in the logbook.

6.12. If two consecutive days (48 hours) indicate continued exceedances, the appropriate department supervisor must be immediately notified. The supervisor shall investigate the variance from acceptable conditions and determine that the variance is not a result of normal operating circumstances or activities.

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6.13. If the device is in fact believed to be in need of corrective action, the supervisor shall notify the Operations Manager, Technical Director or Quality Manager. Further appropriate action will then be taken in concert with the Quality Manager and Laboratory Directory to correct non-compliances with procedure/protocols.

6.14. ***Contingency for Catastrophic Sample Cooler Failure***

6.14.1. In the event that a sample (4°C) cooler has catastrophically failed, e.g., a mechanical breakdown not correctable by thermostat adjustment, the following contingency plan should be implemented.

6.14.2. If the cooler is a small unit, e.g., VOA storage, the contents should be immediately transferred to a larger (operational) unit until the defective cooler is repaired. Additionally, the department supervisor and QA department should be notified about the defective unit and subsequent (temporary) sample transfer.

6.14.3. If the cooler is a large walk-in style, STL Buffalo has arranged for 24/7 on-call service from the following contractor:

MJ Mechanical Services, Inc.
2040 Military Road
Tonawanda, NY 14150
pH: 716.874.9200

6.14.4. In the event of an "off-hours" failure of a large walk-in sample cooler, simply phone the above listed number and a service technician will respond (via phone) within approximately fifteen minutes.

6.14.5. Additionally, one of the following STL Buffalo personnel must be immediately notified(in order to coordinate with MJ Mechanical, Inc. for any emergency work):

Technical Director
Sample Control Supervisor
Operations Manager

6.14.6. (Home and cell phone numbers for the above individuals are posted throughout the facility).

6.15. *REAGENT WATER*

6.16. Maintenance of the water systems is performed at the following schedule:
6.16.1. Check conductivity – daily

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6.16.2. Check deionzer light – daily
6.16.3. Clean system – as required
6.16.4. Replace cartridge and large mixed bed resins – as required

6.17. The in-line conductivity is visually monitored at the point of use (discharge)

6.18. The off-line conductivity is collected separately for each water system, measured and recorded in the Deionized Water Monitoring Log.

6.18.1. This log is maintained by the Wet Chemistry Department, and the Wet Chemistry supervisor is responsible for monitoring conductivity compliance and notifying other departments in the event of any water quality excursions.

6.18.2. Acceptance criteria for Conductivity is ≤1.0uhm at 25°C

6.19. Water quality is also confirmed through the use of method blanks.
6.19.1. All analyses for each parameter performed at STL, consistent with our Laboratory Quality Manual, include the use of a method blank.

6.19.2. These analyses provide the means for monitoring laboratory grade water quality in addition to conductivity, pH, solids content, metals, common anions and other inorganic and organic water quality indicators.

6.19.3. Method blanks represent deionized water processed and analyzed as a sample.

6.19.4. Method blank analysis, consistent with the required analysis, method and protocol is provided to our clients and is available for review, not only in support of the quality of analytical results, but also for informational purposes regarding maintenance of the quality of our water supply.

6.19.5. Method blank results include the date of preparation and analysis and since they are treated as samples, the method blank data are traceable throughout the laboratory procedure in any of the analytical departments within the company.

6.19.6. The acceptance criteria for method blanks = All target analytes < Reporting Limit

6.19.6.1. For DoD projects, the acceptance criteria is <1/2 RL.

6.20. ***Documentation and Corrective Action Procedures***

6.21. Record keeping requires notation of the following items
6.21.1. Date
6.21.2. Time

6.21.3. Analyst Initials
6.21.4. Actual recording (weight, temperature, conductivity) measured for each control device
6.21.5. Acceptance Criteria

6.22. If an actual measurement falls outside the acceptance range, a comment or notation must be made in the logbook.
6.22.1. The following codes are appended in each logbook and should be used to document out-of-control situations and corrective action.

6.22.1.1. **1** = Manual Adjustment Done
6.22.1.2. **2** = Supervisor Notified / Service Requested
6.22.1.3. **3** = Service Complete

6.23. ***Out of Service Equipment***

6.24. Any piece of equipment that fails to meet the QA/QC requirement must not be used prior to an acceptable verification. The following steps are to be taken:

6.24.1. Determine if the item can be immediately repaired or restored to an acceptable performance level. If so, perform maintenance until an acceptable verification is obtained.

6.24.2. If an acceptable verification is unable to be obtained, or the item will remain in an unrepaired state for an extended period of time, perform the following:

6.24.2.1. Label the equipment as "OUT-OF-SERVICE".
6.24.2.2. The label should be clearly visible and properly secured to the equipment.
6.24.2.3. Include the date of removal
6.24.2.4. A description of the reason for removal from service may be included but is not required

6.24.2.5. Note in the appropriate maintenance logbook the "out of service" date.

6.24.2.6. Notify the supervisor who should arrange to have a service call placed with the appropriate manufacturer.

6.24.2.7. If determined to be the best option, discard and replace the unacceptable piece of equipment.

6.25. See SOP AGP-OutofService-65 for additional information related to tagging of support equipment.

7.0 REFERENCES

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7.1. ASTM D4753–87; "Evaluating, Selecting and Specifying Balances and Scales for Use in Soil and Rock Testing"
7.2. ASTM D1193-77 (1983) and D4519
7.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW846, Third Edition
7.4. STL Buffalo SOP AGP-OutofService-65, "Out of Service Equipment".

8.0 FLOWCHARTS, TABLES & DIAGRAMS

8.1. Exhibit 1: Example of Balance Verification Logbook
8.2. Exhibit 2: Example of Temperature Recording Logbook
8.3. Exhibit 3: Example of Water System Maintenance Logbook
8.4. Exhibit 4: DeIonized Water System Conductivity Logbook

9.0 CHANGES FROM LAST REVISION

9.1. Section 6.6: Changed reference from 40 grams to 30 grams.

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Exhibit 1: Example Balance Verification Logbook

STL Buffalo
VOLATILES BALANCE LOGBOOK
Logbook#

		METTLER AE 160 Serial # C26007				DENVER AL 500 Serial # 0063223				
Date	Analyst	20.0 mg acceptance range = +/- 0.0002 mg	2.0 g acceptance range = +/- 0.02 g	20.0 g acceptance range = +/- 0.02 g	100.0 g acceptance range = +/- 0.1 g	2.0 g acceptance range = +/- 0.02 g	20.0 g acceptance range = +/- 0.02 g	100.0 g acceptance range = +/- 0.1 g	Comment

Corrective Action Codes: 1 = Manual Adjustment Done
2 = Supervisor Notified / Service Requested
3 = Service Complete

Reviewed By_____ Date_____

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Supercedes: Revision 0

Exhibit 2: Example Temperature Record Logbook

STL Buffalo
ORGANIC PREP TEMPERATURE LOG
Logbook #

DATE	ANALYST	DRY WEIGHT OVEN #4 105°± 4°C Therm#:	SPIKE/SURR. INCUBATOR #2 4.0°± 2°C Therm#:	DIAZOMETHANE FREEZER #2 -5 to -15 °C Therm#:	EXTRA VOLUME INCUBATOR #3 4.0 °± 2°C Therm#:	EXTRA VOLUME FREEZER #3 -5 to -15 °C Therm#:	MAIN SAMPLE COOLER #1 2.0-4.0 °C Therm#:	STANDARD COMMENTS

STANDARD COMMENTS: 1. Manual Adjustment Done
2. Supervisor Notified/ Service Requested
3. Service Complete

Reviewed By_____ Date_____

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Supercedes: Revision 0

Exhibit 3: Example Water System Maintenance Logbook

STL Buffalo

ELGA WATER MAINTENANCE LOG
Logbook #

DATE: **TIME:** **OPERATOR:**

MAINTENANCE PERFORMED:

Replace Outside ELGA Filter: YES / NO
Part #. 36106

Lot # _____

Replace Multi-Media Cartridge Pack-Type 1: YES / NO
Part #. LC126

Batch #_____

CORRECTIVE MAINTENANCE: Required / Not Required

Problem: (State time of occurrence or time problem was recognized):

_____
_____
_____
_____

Action(s) Taken:

_____
_____
_____

Was the problem solved? YES / NO -- **Operation Continued @:** _____ **(enter time)**

If no, state next actions until resolution (use back of form for further explanation)

_____
_____
_____
_____
_____

Supervisor Review: _____ **Date:** _____

Corrective Action Codes: 1 = Manual Adjustment Done
2 = Supervisor Notified / Service Requested
3 = Service Complete

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Supercedes: Revision 0

Exhibit 4: De-Ionized Water Conductivity Logbook

STL BUFFALO
DEIONIZED WATER MONITORING LOG
(Units of Measure = µmhos/cm)
Logbook# A05-09-10

Date	Analyst	Time	KCL Concentration used	Temp (°C) ATC Probe KCl	Sp.Cond. @ 25°C KCL Acceptance Range +/- 10% actual	Temp (°C) ATC Probe Milli Q	Sp. Cond. @25°C Acceptable Range<1.0 Milli Q Water	Temp (°C) ATC Probe Di H2O	Sp. Cond. @25°C Acceptable Range<1.0 Di Water

STANDARD COMMENTS:
1. Manual Adjustment Done / Rechecked
2. Supervisor Notified / Service Requested
3. Service Complete

Reviewed By:_____ Date_____

Attachment W

Laboratory Control Limits for Organics and Inorganics

Job No: A07-8629
Project/Task: NY5A9406 6

Test No	Description	QAPP	Protcl	Method	Mtx	TCLP	Holding Type	Holding Tclp	Holding Extr	Holding Anal	Prep Type	Unit Measure	Detect Limit Type	Detect Limit Value	Spikes Code	Spikes Amount	Spikes Conc	Spikes QC Limits	Spikes RPD
CGAO5540	ENSR-SOIL-SW846-TOTAL TAL ME																		
CTA26315	ALUMINUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	10.00000	A00066	1.00 ML	1000.00 UG/ML	(80-120)	20.0
CTA26314	ANTIMONY - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	15.00000	A00066	1.00 ML	20.00 UG/ML	(75-125)	20.0
CTA26316	ARSENIC - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	2.00000	A00066	1.00 ML	20.00 UG/ML	(75-125)	20.0
CTA26317	BARIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.50000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26318	BERYLLIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.20000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26319	CADMIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.20000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26320	CALCIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	50.00000	A00066	1.00 ML	1000.00 UG/ML	(75-125)	20.0
CTA26321	CHROMIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.50000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26322	COBALT - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.50000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26323	COPPER - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	1.00000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26324	IRON - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	10.00000	A00066	1.00 ML	1000.00 UG/ML	(80-120)	20.0
CTA26325	LEAD - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	1.00000	A00066	1.00 ML	20.00 UG/ML	(75-125)	20.0
CTA26326	MAGNESIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	20.00000	A00066	1.00 ML	1000.00 UG/ML	(75-125)	20.0
CTA26327	MANGANESE - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.20000	A00066	1.00 ML	20.00 UG/ML	(75-125)	20.0
CTA26328	MERCURY - TOTAL - S		SW8463	7471	Soil	N	S	0	28	28	D	MG/KG	EQL	0.02000	A00066	2.00 ML	0.10 UG/ML	(80-120)	20.0
CTA26330	NICKEL - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.50000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26331	POTASSIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	30.00000	A00066	1.00 ML	1000.00 UG/ML	(75-125)	20.0
CTA26332	SELENIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	4.00000	A00066	1.00 ML	20.00 UG/ML	(80-120)	20.0
CTA26333	SILVER - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.50000	A00066	1.00 ML	5.00 UG/ML	(80-120)	20.0
CTA26334	SODIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	140.00000	A00066	1.00 ML	1000.00 UG/ML	(80-120)	20.0
CTA26337	THALLIUM - TOTAL - S (METHOD		SW8463	6020	Soil	N	S	0	180	180	D	MG/KG	EQL	0.01000	A00066	1.00 ML	20.00 UG/ML	(75-125)	20.0
CTA26335	VANADIUM - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	0.50000	A00066	1.00 ML	20.00 UG/ML	(75-125)	20.0
CTA26336	ZINC - TOTAL - S		SW8463	6010	Soil	N	S	0	180	180	D	MG/KG	EQL	2.00000	A00066	1.00 ML	20.00 UG/ML	(75-125)	20.0

Test No	Description	QAPP	Protcl	Method	Mtx	TCLP	Holding Type	Holding Tclp	Holding Extr	Holding Anal	Prep Type	Unit Measure	Detect Limit Type	Detect Limit Value	Spikes Code	Spikes Amount	Spikes Conc	Spikes QC Limits	Spikes RPD
CGAO5541	ENSR-SW8463-TOTAL TAL METALS																		
CTA26350	ENSR-ALUMINUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.20000	A00067	1.00 ML	500.00 UG/ML	(75-125)	20.0
CTA26352	ENSR-ANTIMONY - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.02000	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26354	ENSR-ARSENIC - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.01000	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA26355	ENSR-BARIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00200	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26356	ENSR-BERYLLIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00200	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26358	ENSR-CADMIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00100	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA26357	ENSR-CALCIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.50000	A00067	1.00 ML	500.00 UG/ML	(80-120)	20.0
CTA26359	ENSR-CHROMIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00400	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26360	ENSR-COBALT - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00400	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26361	ENSR-COPPER - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.01000	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26362	ENSR-IRON - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.05000	A00067	1.00 ML	500.00 UG/ML	(80-120)	20.0
CTA26363	ENSR-LEAD - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00500	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26364	ENSR-MAGNESIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.20000	A00067	1.00 ML	500.00 UG/ML	(75-125)	20.0
CTA26365	ENSR-MANGANESE - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00300	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA26366	ENSR-MERCURY - TOTAL - W		SW8463	7470	Water	N	S	0	28	28	D	MG/L	EQL	0.00020	A00067	2.00 ML	0.10 UG/ML	(80-120)	20.0
CTA26367	ENSR-NICKEL - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.01000	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA26368	ENSR-POTASSIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.50000	A00067	1.00 ML	500.00 UG/ML	(80-120)	20.0
CTA26369	ENSR-SELENIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.01500	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA26370	ENSR-SILVER - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00300	A00067	0.25 ML	10.00 UG/ML	(75-125)	20.0
CTA26371	ENSR-SODIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	1.00000	A00067	1.00 ML	500.00 UG/ML	(80-120)	20.0
CTA26372	ENSR-THALLIUM - TOTAL - W (M		SW8463	6020	Water	N	S	0	180	180	D	MG/L	EQL	0.00020	A00325	0.10 ML	10.00 UG/ML	(75-125)	20.0
CTA26373	ENSR-VANADIUM - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.00500	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA26374	ENSR-ZINC - TOTAL - W		SW8463	6010	Water	N	S	0	180	180	D	MG/L	EQL	0.01000	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0

Job No: A07-8629
Project/Task: NY5A9406 6

Test No	Description	QAPP	Protcl	Method	Mtx	TCLP	Holding Type	Holding Tclp	Holding Extr	Holding Anal	Prep Type	Unit Measure	Detect Limit Type	Detect Limit Value	Spikes Code	Spikes Amount	Spikes Conc	QC Limits	RPD
WC	WET CHEMISTRY																		
CTA36445	ENSR - SOIL - SW8463 9012A -		SW8463	9012A	Soil	N	R	0	0	12	N	MG/KG	EQL	1.00000	A00088	0.00 ML	50.00 UG/ML	(40-160)	15.0
CTA36446	ENSR - AQ - SW8463 9012A - C		SW8463	9012A	Water	N	S	0	0	14	N	MG/L	EQL	0.01000	A00088	0.00 ML	50.00 UG/ML	(90-110)	15.0

Job No: A07-8629 ENSR
Project/Task: NY5A9406 6 Dummy Site with no real samples (TEST)
Test No: CTA20056

Fraction:	GE	Holding Type:	S
Method:	8082	TCLP Extraction:	0.00 Days
Protocol:	SW8463	Pre-Conc:	0.00 Days
Test Matrix:	Water	Extraction:	7.00 Days
Level:	L	Analysis:	40.00 Days
GC Second Column:	N	Prep Type:	E
TCLP:	N	Extr:	SEPF
Spike Code:	A00222	GPC Cleanup?:	N
SUR/IS Code:	A00035 NONE	Units of Measure:	UG/L
TDL Calculation:	ADL/EQL		

				Spikes		QC Limits	
Seq	Parameter	Description	EQL (UG/L)	Amount	Conc	% REC	RPD
10	12674-11-2	Aroclor 1016	0.5000	1000.00 UL	5.00 NG/UL	61-123	50.0
20	11104-28-2	Aroclor 1221	0.5000				
30	11141-16-5	Aroclor 1232	0.5000				
40	53469-21-9	Aroclor 1242	0.5000				
50	12672-29-6	Aroclor 1248	0.5000				
60	11097-69-1	Aroclor 1254	0.5000				
70	11096-82-5	Aroclor 1260	0.5000	1000.00 UL	5.00 NG/UL	44-121	50.0
SUR	877-09-8	Tetrachloro-m-xylene		1000.00 UL	0.20 NG/UL	35-136	
SUR	REI-002	Decachlorobiphenyl		1000.00 UL	0.20 NG/UL	12-137	

Job No: A07-8629 ENSR
Project/Task: NY5A9406 6 Dummy Site with no real samples (TEST)
Test No: CTA36444

Fraction:	GE	Holding Type:	R
Method:	8082	TCLP Extraction:	0.00 Days
Protocol:	SW8463	Pre-Conc:	10.00 Days
Test Matrix:	Soil	Extraction:	10.00 Days
Level:	L	Analysis:	40.00 Days
GC Second Column:	N	Prep Type:	E
TCLP:	N	Extr:	SONC
Spike Code:	A00222	GPC Cleanup?:	N
SUR/IS Code:	A00035 NONE	Units of Measure:	UG/KG
TDL Calculation:	ADL/CDL		

			CDL	Spikes		QC Limits	
Seq	Parameter	Description	(UG/KG)	Amount	Conc	% REC	RPD
10	12674-11-2	Aroclor 1016	33.0000	1000.00 UL	5.00 NG/UL	59-134	50.0
20	11104-28-2	Aroclor 1221	67.0000				
30	11141-16-5	Aroclor 1232	33.0000				
40	53469-21-9	Aroclor 1242	33.0000				
50	12672-29-6	Aroclor 1248	33.0000				
60	11097-69-1	Aroclor 1254	33.0000				
70	11096-82-5	Aroclor 1260	33.0000	1000.00 UL	5.00 NG/UL	66-140	50.0
SUR	877-09-8	Tetrachloro-m-xylene		1000.00 UL	0.20 NG/UL	35-134	
SUR	REI-002	Decachlorobiphenyl		1000.00 UL	0.20 NG/UL	34-148	

Job No: A07-8629 ENSR
Project/Task: NY5A9406 6 Dummy Site with no real samples (TEST)
Test No: CTA19009

Fraction: MB
Method: 8270
Protocol: SW8463
Test Matrix: Water
Level: L
GC Second Column: N
TCLP: N
Spike Code: A00055
SUR/IS Code: A00001 A00010
TDL Calculation: ADL/EQL

Holding Type: S
TCLP Extraction: 0.00 Days
Pre-Conc: 0.00 Days
Extraction: 7.00 Days
Analysis: 40.00 Days
Prep Type: E
Extr: SEPF
GPC Cleanup?: N
Units of Measure: UG/L

			EQL	Spikes		QC Limits	
Seq	Parameter	Description	(UG/L)	Amount	Conc	% REC	RPD
10	83-32-9	Acenaphthene	5.0000	1000.00 UL	100.00 NG/UL	60-118	23.0
20	208-96-8	Acenaphthylene	5.0000				
25	98-86-2	Acetophenone	5.0000				
30	120-12-7	Anthracene	5.0000				
35	1912-24-9	Atrazine	5.0000				
36	100-52-7	Benzaldehyde	5.0000				
40	56-55-3	Benzo(a)anthracene	5.0000				
50	205-99-2	Benzo(b)fluoranthene	5.0000				
60	207-08-9	Benzo(k)fluoranthene	5.0000				
70	191-24-2	Benzo(ghi)perylene	5.0000				
80	50-32-8	Benzo(a)pyrene	5.0000				
100	100-51-6	Benzyl alcohol	20.0000				
105	92-52-4	Biphenyl	5.0000				
110	111-91-1	Bis(2-chloroethoxy) methan	5.0000				
120	111-44-4	Bis(2-chloroethyl) ether	5.0000				
130	108-60-1	2,2'-Oxybis(1-Chloropropan	5.0000				
140	117-81-7	Bis(2-ethylhexyl) phthalat	5.0000				
150	101-55-3	4-Bromophenyl phenyl ether	5.0000				
160	85-68-7	Butyl benzyl phthalate	5.0000				
165	105-60-2	Caprolactam	5.0000				
170	106-47-8	4-Chloroaniline	5.0000				
180	59-50-7	4-Chloro-3-methylphenol	5.0000	1000.00 UL	100.00 NG/UL	64-120	25.0
190	91-58-7	2-Chloronaphthalene	5.0000				
200	95-57-8	2-Chlorophenol	5.0000	1000.00 UL	100.00 NG/UL	47-120	33.0
210	7005-72-3	4-Chlorophenyl phenyl ethe	5.0000				
220	218-01-9	Chrysene	5.0000				
230	53-70-3	Dibenzo(a,h)anthracene	5.0000				
240	132-64-9	Dibenzofuran	5.0000				
245	84-74-2	Di-n-butyl phthalate	5.0000				
280	91-94-1	3,3'-Dichlorobenzidine	5.0000				
290	120-83-2	2,4-Dichlorophenol	5.0000				
300	84-66-2	Diethyl phthalate	5.0000				
310	105-67-9	2,4-Dimethylphenol	5.0000				
320	131-11-3	Dimethyl phthalate	5.0000				
330	534-52-1	4,6-Dinitro-2-methylphenol	10.0000				
340	51-28-5	2,4-Dinitrophenol	10.0000				
350	121-14-2	2,4-Dinitrotoluene	5.0000	1000.00 UL	100.00 NG/UL	58-125	20.0
360	606-20-2	2,6-Dinitrotoluene	5.0000				
380	117-84-0	Di-n-octyl phthalate	5.0000				
390	206-44-0	Fluoranthene	5.0000				
400	86-73-7	Fluorene	5.0000				
410	118-74-1	Hexachlorobenzene	5.0000				

Job No: A07-8629
Test No: CTA19009

Seq	Parameter	Description	EQL (UG/L)	Spikes Amount	Spikes Conc	QC Limits % REC	QC Limits RPD
420	87-68-3	Hexachlorobutadiene	5.0000				
430	77-47-4	Hexachlorocyclopentadiene	5.0000				
440	67-72-1	Hexachloroethane	5.0000				
450	193-39-5	Indeno(1,2,3-cd)pyrene	5.0000				
460	78-59-1	Isophorone	5.0000				
470	91-57-6	2-Methylnaphthalene	5.0000				
480	95-48-7	2-Methylphenol	5.0000				
490	106-44-5	4-Methylphenol	5.0000				
500	91-20-3	Naphthalene	5.0000				
510	88-74-4	2-Nitroaniline	10.0000				
511	99-09-2	3-Nitroaniline	10.0000				
512	100-01-6	4-Nitroaniline	10.0000				
513	98-95-3	Nitrobenzene	5.0000				
514	88-75-5	2-Nitrophenol	5.0000				
515	100-02-7	4-Nitrophenol	10.0000	1000.00 UL	100.00 NG/UL	16-120	30.0
516	86-30-6	N-nitrosodiphenylamine	5.0000				
521	621-64-7	N-Nitroso-Di-n-propylamine	5.0000	1000.00 UL	100.00 NG/UL	55-115	38.0
530	87-86-5	Pentachlorophenol	10.0000	1000.00 UL	100.00 NG/UL	39-136	27.0
540	85-01-8	Phenanthrene	5.0000				
550	108-95-2	Phenol	5.0000	1000.00 UL	100.00 NG/UL	17-120	39.0
560	129-00-0	Pyrene	5.0000	1000.00 UL	100.00 NG/UL	58-136	25.0
580	95-95-4	2,4,5-Trichlorophenol	5.0000				
590	88-06-2	2,4,6-Trichlorophenol	5.0000				
IS	1146-65-2	Naphthalene-D8		20.00 UL	2000.00 NG/UL	50-200	
IS	198-55-0	Perylene-D12		20.00 UL	2000.00 NG/UL	50-200	
IS	SU106-46-7	1,4-Dichlorobenzene-D4		20.00 UL	2000.00 NG/UL	50-200	
IS	SU218-01-9	Chrysene-D12		20.00 UL	2000.00 NG/UL	50-200	
IS	SU83-32-9	Acenaphthene-D10		20.00 UL	2000.00 NG/UL	50-200	
IS	SU85-01-8	Phenanthrene-D10		20.00 UL	2000.00 NG/UL	50-200	
SUR	118-79-6	2,4,6-Tribromophenol		1000.00 UL	150.00 NG/UL	52-132	
SUR	1718-51-0	p-Terphenyl-d14		1000.00 UL	100.00 NG/UL	24-136	
SUR	321-60-8	2-Fluorobiphenyl		1000.00 UL	100.00 NG/UL	48-116	
SUR	367-12-4	2-Fluorophenol		1000.00 UL	150.00 NG/UL	20-120	
SUR	4165-60-0	Nitrobenzene-D5		1000.00 UL	100.00 NG/UL	46-112	
SUR	SU108-95-2	Phenol-D5		1000.00 UL	150.00 NG/UL	16-120	

Job No: A07-8629 ENSR
Project/Task: NY5A9406 6 Dummy Site with no real samples (TEST)
Test No: CTA23216

Fraction:	MB	Holding Type:	S
Method:	8270	TCLP Extraction:	0.00 Days
Protocol:	SW8463	Pre-Conc:	0.00 Days
Test Matrix:	Soil	Extraction:	14.00 Days
Level:	L	Analysis:	40.00 Days
GC Second Column:	N	Prep Type:	E
TCLP:	N	Extr:	SONC
Spike Code:	A00055	GPC Cleanup?:	N
SUR/IS Code:	A00001 A00010	Units of Measure:	UG/KG
TDL Calculation:	ADL/EQL		

			EQL	Spikes		QC Limits	
Seq	Parameter	Description	(UG/KG)	Amount	Conc	% REC	RPD
20	83-32-9	Acenaphthene	170.0000	1000.00 UL	100.00 NG/UL	53-119	16.0
25	208-96-8	Acenaphthylene	170.0000				
26	98-86-2	Acetophenone	170.0000				
30	120-12-7	Anthracene	170.0000				
32	1912-24-9	Atrazine	170.0000				
35	100-52-7	Benzaldehyde	170.0000				
40	56-55-3	Benzo(a)anthracene	170.0000				
50	205-99-2	Benzo(b)fluoranthene	170.0000				
60	207-08-9	Benzo(k)fluoranthene	170.0000				
70	191-24-2	Benzo(ghi)perylene	170.0000				
80	50-32-8	Benzo(a)pyrene	170.0000				
90	65-85-0	Benzoic acid	4800.0000				
100	100-51-6	Benzyl alcohol	330.0000				
105	92-52-4	Biphenyl	170.0000				
110	111-91-1	Bis(2-chloroethoxy) methan	170.0000				
120	111-44-4	Bis(2-chloroethyl) ether	170.0000				
130	108-60-1	2,2'-Oxybis(1-Chloropropan	170.0000				
140	117-81-7	Bis(2-ethylhexyl) phthalat	170.0000				
150	101-55-3	4-Bromophenyl phenyl ether	170.0000				
160	85-68-7	Butyl benzyl phthalate	170.0000				
165	105-60-2	Caprolactam	170.0000				
170	106-47-8	4-Chloroaniline	170.0000				
180	59-50-7	4-Chloro-3-methylphenol	170.0000	1000.00 UL	100.00 NG/UL	49-125	20.0
190	91-58-7	2-Chloronaphthalene	170.0000				
200	95-57-8	2-Chlorophenol	170.0000	1000.00 UL	100.00 NG/UL	38-104	26.0
210	7005-72-3	4-Chlorophenyl phenyl ethe	170.0000				
220	218-01-9	Chrysene	170.0000				
230	53-70-3	Dibenzo(a,h)anthracene	170.0000				
240	132-64-9	Dibenzofuran	170.0000				
245	84-74-2	Di-n-butyl phthalate	170.0000				
280	91-94-1	3,3'-Dichlorobenzidine	170.0000				
290	120-83-2	2,4-Dichlorophenol	170.0000				
300	84-66-2	Diethyl phthalate	170.0000				
310	105-67-9	2,4-Dimethylphenol	170.0000				
320	131-11-3	Dimethyl phthalate	170.0000				
330	534-52-1	4,6-Dinitro-2-methylphenol	330.0000				
340	51-28-5	2,4-Dinitrophenol	330.0000				
350	121-14-2	2,4-Dinitrotoluene	170.0000	1000.00 UL	100.00 NG/UL	55-125	19.0
360	606-20-2	2,6-Dinitrotoluene	170.0000				
380	117-84-0	Di-n-octyl phthalate	170.0000				
390	206-44-0	Fluoranthene	170.0000				
400	86-73-7	Fluorene	170.0000				

Job No: A07-8629
Test No: CTA23216

Seq	Parameter	Description	EQL (UG/KG)	Spikes Amount	Spikes Conc	QC Limits % REC	QC Limits RPD
410	118-74-1	Hexachlorobenzene	170.0000				
420	87-68-3	Hexachlorobutadiene	170.0000				
430	77-47-4	Hexachlorocyclopentadiene	170.0000				
440	67-72-1	Hexachloroethane	170.0000				
450	193-39-5	Indeno(1,2,3-cd)pyrene	170.0000				
460	78-59-1	Isophorone	170.0000				
470	91-57-6	2-Methylnaphthalene	170.0000				
480	95-48-7	2-Methylphenol	170.0000				
490	106-44-5	4-Methylphenol	170.0000				
500	91-20-3	Naphthalene	170.0000				
510	88-74-4	2-Nitroaniline	330.0000				
511	99-09-2	3-Nitroaniline	330.0000				
512	100-01-6	4-Nitroaniline	330.0000				
513	98-95-3	Nitrobenzene	170.0000				
514	88-75-5	2-Nitrophenol	170.0000				
515	100-02-7	4-Nitrophenol	330.0000	1000.00 UL	100.00 NG/UL	44-137	25.0
520	86-30-6	N-nitrosodiphenylamine	170.0000				
525	621-64-7	N-Nitroso-Di-n-propylamine	170.0000	1000.00 UL	100.00 NG/UL	46-120	20.0
530	87-86-5	Pentachlorophenol	330.0000	1000.00 UL	100.00 NG/UL	33-136	27.0
540	85-01-8	Phenanthrene	170.0000				
550	108-95-2	Phenol	170.0000	1000.00 UL	100.00 NG/UL	36-110	25.0
560	129-00-0	Pyrene	170.0000	1000.00 UL	100.00 NG/UL	51-133	25.0
580	95-95-4	2,4,5-Trichlorophenol	170.0000				
590	88-06-2	2,4,6-Trichlorophenol	170.0000				
IS	1146-65-2	Naphthalene-D8		20.00 UL	2000.00 NG/UL	50-200	
IS	198-55-0	Perylene-D12		20.00 UL	2000.00 NG/UL	50-200	
IS	SU106-46-7	1,4-Dichlorobenzene-D4		20.00 UL	2000.00 NG/UL	50-200	
IS	SU218-01-9	Chrysene-D12		20.00 UL	2000.00 NG/UL	50-200	
IS	SU83-32-9	Acenaphthene-D10		20.00 UL	2000.00 NG/UL	50-200	
IS	SU85-01-8	Phenanthrene-D10		20.00 UL	2000.00 NG/UL	50-200	
SUR	118-79-6	2,4,6-Tribromophenol		1000.00 UL	150.00 NG/UL	46-129	
SUR	1718-51-0	p-Terphenyl-d14		1000.00 UL	100.00 NG/UL	51-125	
SUR	321-60-8	2-Fluorobiphenyl		1000.00 UL	100.00 NG/UL	43-119	
SUR	367-12-4	2-Fluorophenol		1000.00 UL	150.00 NG/UL	30-107	
SUR	4165-60-0	Nitrobenzene-D5		1000.00 UL	100.00 NG/UL	35-113	
SUR	SU108-95-2	Phenol-D5		1000.00 UL	150.00 NG/UL	36-116	

Job No: A07-8629 ENSR
Project/Task: NY5A9406 6 Dummy Site with no real samples (TEST)
Test No: CTA25699

Fraction:	MV	Holding Type:	S
Method:	8260	TCLP Extraction:	0.00 Days
Protocol:	SW8463	Pre-Conc:	0.00 Days
Test Matrix:	Water	Extraction:	0.00 Days
Level:	L	Analysis:	14.00 Days
GC Second Column:	N	Prep Type:	N
TCLP:	N	Extr:	
Spike Code:	A00039	GPC Cleanup?:	N
SUR/IS Code:	A00025 A00107	Units of Measure:	UG/L
TDL Calculation:	ADL/CDL		

			CDL	Spikes		QC Limits	
Seq	Parameter	Description	(UG/L)	Amount	Conc	% REC	RPD
10	67-64-1	Acetone	25.0000				
20	71-43-2	Benzene	5.0000	2.50 UL	50.00 NG/UL	79-121	13.0
30	75-27-4	Bromodichloromethane	5.0000				
40	75-25-2	Bromoform	5.0000				
50	74-83-9	Bromomethane	5.0000				
60	78-93-3	2-Butanone	25.0000				
70	75-15-0	Carbon Disulfide	5.0000				
80	56-23-5	Carbon Tetrachloride	5.0000				
90	108-90-7	Chlorobenzene	5.0000	2.50 UL	50.00 NG/UL	79-118	25.0
100	75-00-3	Chloroethane	5.0000				
120	67-66-3	Chloroform	5.0000				
130	74-87-3	Chloromethane	5.0000				
132	110-82-7	Cyclohexane	5.0000				
135	106-93-4	1,2-Dibromoethane	5.0000				
140	124-48-1	Dibromochloromethane	5.0000				
141	96-12-8	1,2-Dibromo-3-chloropropan	5.0000				
143	95-50-1	1,2-Dichlorobenzene	5.0000				
145	541-73-1	1,3-Dichlorobenzene	5.0000				
146	106-46-7	1,4-Dichlorobenzene	5.0000				
147	75-71-8	Dichlorodifluoromethane	5.0000				
150	75-34-3	1,1-Dichloroethane	5.0000				
160	107-06-2	1,2-Dichloroethane	5.0000				
170	75-35-4	1,1-Dichloroethene	5.0000	2.50 UL	50.00 NG/UL	71-147	16.0
180	156-59-2	cis-1,2-Dichloroethene	5.0000				
185	156-60-5	trans-1,2-Dichloroethene	5.0000				
190	78-87-5	1,2-Dichloropropane	5.0000				
200	10061-01-5	cis-1,3-Dichloropropene	5.0000				
210	10061-02-6	trans-1,3-Dichloropropene	5.0000				
220	100-41-4	Ethylbenzene	5.0000				
230	591-78-6	2-Hexanone	25.0000				
235	98-82-8	Isopropylbenzene	5.0000				
237	79-20-9	Methyl acetate	5.0000				
238	108-87-2	Methylcyclohexane	5.0000				
240	75-09-2	Methylene chloride	5.0000				
250	108-10-1	4-Methyl-2-pentanone	25.0000				
255	1634-04-4	Methyl-t-Butyl Ether (MTBE	5.0000				
260	100-42-5	Styrene	5.0000				
270	79-34-5	1,1,2,2-Tetrachloroethane	5.0000				
280	127-18-4	Tetrachloroethene	5.0000				
290	108-88-3	Toluene	5.0000	2.50 UL	50.00 NG/UL	69-120	18.0
295	120-82-1	1,2,4-Trichlorobenzene	5.0000				
300	71-55-6	1,1,1-Trichloroethane	5.0000				

Job No: A07-8629
Test No: CTA25699

Seq	Parameter	Description	CDL (UG/L)	Spikes Amount	Spikes Conc	QC Limits % REC	QC Limits RPD
310	79-00-5	1,1,2-Trichloroethane	5.0000				
314	76-13-1	1,1,2-Trichloro-1,2,2-trif	5.0000				
315	75-69-4	Trichlorofluoromethane	5.0000				
320	79-01-6	Trichloroethene	5.0000	2.50 UL	50.00 NG/UL	71-120	16.0
340	75-01-4	Vinyl chloride	5.0000				
350	1330-20-7	Total Xylenes	15.0000				
IS	3114-55-4	Chlorobenzene-D5		1.00 UL	125.00 NG/UL	50-200	
IS	540-36-3	1,4-Difluorobenzene		1.00 UL	125.00 NG/UL	50-200	
IS	SU106-46-7	1,4-Dichlorobenzene-D4		1.00 UL	125.00 NG/UL	50-200	
SUR	2037-26-5	Toluene-D8		1.00 UL	125.00 NG/UL	71-126	
SUR	460-00-4	p-Bromofluorobenzene		1.00 UL	125.00 NG/UL	73-120	
SUR	SU107-06-2	1,2-Dichloroethane-D4		1.00 UL	125.00 NG/UL	66-137	

Job No: A07-8629 ENSR
Project/Task: NY5A9406 6 Dummy Site with no real samples (TEST)
Test No: CTA25705

Fraction:	MV	Holding Type:	S
Method:	8260	TCLP Extraction:	0.00 Days
Protocol:	SW8463	Pre-Conc:	0.00 Days
Test Matrix:	Soil	Extraction:	0.00 Days
Level:	L	Analysis:	14.00 Days
GC Second Column:	N	Prep Type:	N
TCLP:	N	Extr:	
Spike Code:	A00039	GPC Cleanup?:	N
SUR/IS Code:	A00025 A00107	Units of Measure:	UG/KG
TDL Calculation:	ADL/EQL		

Seq	Parameter	Description	EQL (UG/KG)	Spikes Amount	Spikes Conc	QC Limits % REC	QC Limits RPD
10	67-64-1	Acetone	25.0000				
20	71-43-2	Benzene	5.0000	2.50 UL	50.00 NG/UL	78-122	25.0
30	75-27-4	Bromodichloromethane	5.0000				
40	75-25-2	Bromoform	5.0000				
50	74-83-9	Bromomethane	5.0000				
60	78-93-3	2-Butanone	25.0000				
70	75-15-0	Carbon Disulfide	5.0000				
80	56-23-5	Carbon Tetrachloride	5.0000				
90	108-90-7	Chlorobenzene	5.0000	2.50 UL	50.00 NG/UL	79-118	25.0
100	75-00-3	Chloroethane	5.0000				
120	67-66-3	Chloroform	5.0000				
130	74-87-3	Chloromethane	5.0000				
132	110-82-7	Cyclohexane	5.0000				
135	106-93-4	1,2-Dibromoethane	5.0000				
140	124-48-1	Dibromochloromethane	5.0000				
141	96-12-8	1,2-Dibromo-3-chloropropan	5.0000				
143	95-50-1	1,2-Dichlorobenzene	5.0000				
145	541-73-1	1,3-Dichlorobenzene	5.0000				
146	106-46-7	1,4-Dichlorobenzene	5.0000				
147	75-71-8	Dichlorodifluoromethane	5.0000				
150	75-34-3	1,1-Dichloroethane	5.0000				
160	107-06-2	1,2-Dichloroethane	5.0000				
170	75-35-4	1,1-Dichloroethene	5.0000	2.50 UL	50.00 NG/UL	70-142	22.0
180	156-59-2	cis-1,2-Dichloroethene	5.0000				
185	156-60-5	trans-1,2-Dichloroethene	5.0000				
190	78-87-5	1,2-Dichloropropane	5.0000				
200	10061-01-5	cis-1,3-Dichloropropene	5.0000				
210	10061-02-6	trans-1,3-Dichloropropene	5.0000				
220	100-41-4	Ethylbenzene	5.0000				
230	591-78-6	2-Hexanone	25.0000				
235	98-82-8	Isopropylbenzene	5.0000				
237	79-20-9	Methyl acetate	5.0000				
238	108-87-2	Methylcyclohexane	5.0000				
240	75-09-2	Methylene chloride	5.0000				
250	108-10-1	4-Methyl-2-pentanone	25.0000				
255	1634-04-4	Methyl-t-Butyl Ether (MTBE	5.0000				
260	100-42-5	Styrene	5.0000				
270	79-34-5	1,1,2,2-Tetrachloroethane	5.0000				
280	127-18-4	Tetrachloroethene	5.0000				
290	108-88-3	Toluene	5.0000	2.50 UL	50.00 NG/UL	74-123	25.0
295	120-82-1	1,2,4-Trichlorobenzene	5.0000				
300	71-55-6	1,1,1-Trichloroethane	5.0000				

Job No: A07-8629
Test No: CTA25705

Seq	Parameter	Description	EQL (UG/KG)	Spikes Amount	Spikes Conc	QC Limits % REC	QC Limits RPD
310	79-00-5	1,1,2-Trichloroethane	5.0000				
314	76-13-1	1,1,2-Trichloro-1,2,2-trif	5.0000				
315	75-69-4	Trichlorofluoromethane	5.0000				
320	79-01-6	Trichloroethene	5.0000	2.50 UL	50.00 NG/UL	79-121	24.0
340	75-01-4	Vinyl chloride	10.0000				
350	1330-20-7	Total Xylenes	15.0000				
IS	3114-55-4	Chlorobenzene-D5		1.00 UL	125.00 NG/UL	50-200	
IS	540-36-3	1,4-Difluorobenzene		1.00 UL	125.00 NG/UL	50-200	
IS	SU106-46-7	1,4-Dichlorobenzene-D4		1.00 UL	125.00 NG/UL	50-200	
SUR	2037-26-5	Toluene-D8		1.00 UL	125.00 NG/UL	71-125	
SUR	460-00-4	p-Bromofluorobenzene		1.00 UL	125.00 NG/UL	72-126	
SUR	SU107-06-2	1,2-Dichloroethane-D4		1.00 UL	125.00 NG/UL	64-126	

Attachment X

NYSDEC Analytical Service Protocol Exhibit B, Reporting and Deliverables Requirement

NEW YORK STATE

DEPARTMENT OF ENVIRONMENTAL CONSERVATION

ANALYTICAL SERVICE PROTOCOL

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

July 2005

EXHIBIT B

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PART I -- CONTRACT REPORTS/DELIVERABLES SCHEDULE AND DISTRIBUTION

1.0 Summary Table

The following table details the Protocol reporting and deliverable requirements, their schedule, and the distribution that is required for each. Detailed requirements for each lettered "Item" listed in the chart are given in Part II of this Exhibit.

ITEM	DESCRIPTION	# of COPIES[1]	DELIVERY SCHEDULE	DISTRIBUTION		
				1	2	3
A	Standard Operating Procedures (SOPs)	1	60 days after notification of contract award, and as required in Exhibit E.	X		
B	Quality Assurance Management Plan (QAMP)	1	60 days after notification of contract award, and as required in Exhibit E.	X		
C	Weekly Sample Receipt Summary	1	The Wednesday following the calender week samples are received.	X		
D[2]	Sample Data Summary Package	2	30 days after the VTSR[3] of the last sample in the Sample Delivery Group (SDG[4]).	As Directed		
E[2]	Sample Data Package (.PDF)	1	30 days after the VTSR[3] of the last sample in the SDG[4].	X		X
F[2]	Electronic Data Deliverables (EDD)	1	30 days after the VTSR[3] of the last sample in the SDG[4].	X		X
G	Electronic Instrument Data	1	Retain for 3 years after data submission, submit within 7 days of receipt of written request from BWAM.	As Directed		
H	Samples and Extracts[5]	N/A	Retain for 365 days after data submission, submit within 7 days of receipt of written request from BWAM.	As Directed		
I	Full Verification of Instrument Parameters	1	Retain for 3 years after data submission, submit within 7 days of receipt of written request from BWAM.	As Directed		
J	Preliminary Results[6,7]	2	When requested, within 72 hours after receipt of designated samples.		X	X
K	Results of PE sample(s)	1	30 days after receipt of such Performance Evaluation (PE) sample(s).	X		

Notes (for Summary Table)

[1] The number of copies specified is the number of copies required to be delivered to each recipient, for that item.

[2] Deliverables for Items D, E, and F are to be reported total and complete. Concurrent delivery is required. Delivery shall be made such that all designated recipients receive all the items they are scheduled to receive on the same calendar day. If a deliverable item due on the same date as other deliverable items is late, all items scheduled to be due on that day shall be considered late as well. If the deliverables are due on a Saturday, Sunday, or State holiday, then they shall be delivered on the next business day.

[3] Validated Time of Sample Receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report/Chain of Custody Record. Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less with the same laboratory turnaround and not exceeding 20 samples [excluding performance Evaluation (PE) Samples]. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.

[4] Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less and not exceeding 20 samples [excluding Performance Evaluation (PE) samples]. Note that preliminary results have no impact on defining the SDG. Data for all samples in the SDG are due concurrently, unless specified otherwise in a project work plan. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received.

[5] Actual unused samples and extracts are not considered a reportable item, and their return to NYSDEC, if requested, is not billable. Unused portions or samples and extracts are considered to be a deliverable only when their return is requested in writing by NYSDEC. As specified in the Protocol, and unless otherwise instructed by the BWAM, the Laboratory shall dispose of unused sample/extract volume and used sample bottles/containers no earlier than ninety (90) days following submission of analytical data in the form of the Sample Data Package. Until these ninety days have expired, NYSDEC samples and sample extracts are the exclusive property of NYSDEC and cannot be experimented upon, disposed of, or relinquished to third parties without written permission from NYSDEC.

[6] If requested at the time of sample scheduling the contractor shall provide preliminary results, consisting of Form I and Form I TIC analytical results, by fraction, for field and quality control (QC) sample analysis via telefacsimile (fax) or electronic mail, and Form X for Pesticides and Form X for Aroclors. The Contractor will be notified of the fax number or email address at the time of the sample scheduling. Chain of Custody (COC) Records and SDG Cover Sheets shall be submitted with the Preliminary Results. The contractor shall contact the Project Officer after confirming transmission. The Contractor shall document all communication in a telephone contact log.

[7] If a sample requiring Preliminary Results arrives before 5 p.m. (Contractor's local time), the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.

Distribution Addresses:

1. Quality Standards and Analytical Management Section
The Bureau of Watershed Assessment and Management
Division of Water
NYS Department of Environmental Conservation
625 Broadway, 4th Floor
Albany, New York 12233-3502

2. NYSDEC Sample Submitters

3. NYSDEC Project Officers

The BWAM acting on behalf of the Project Officer will provide the Laboratory with the list of addressees for the nine NYSDEC Regions. BWAM will provide the Laboratory with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

***NOTE**: Specific recipient names and addresses are subject to change during the term of the contract. The Bureau of Watershed Assessment and Management (BWAM) will notify the Laboratory in writing of such changes when they occur.*

PART II -- REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

1.0 Overview

The Laboratory shall provide reports and other deliverables as specified by the schedule in Part I of this Exhibit. The required content and assembly of each deliverable is described in Part II of this Exhibit.

Descriptions of the requirements for each deliverable "Item" listed in the chart in Part I, are specified in sections A-G of this Part. Items submitted concurrently MUST BE arranged in the order listed. Additionally, the components of each item MUST BE arranged in the order presented in this Section when the item is submitted.

Examples of specific data deliverables not included herein may be obtained by submitting a written request to The Bureau of Watershed Assessment and Management clearly stating the information requested and signed and dated by the Laboratory Manager.

1.1 All deliverables MUST BE as follows:

- Legible, as specified in Section V,
- Clearly labeled and completed in accordance with instructions in this Exhibit,
- Arranged in the order specified in this Exhibit, and
- Paginated sequentially according to instructions in this Exhibit, starting from the SDG Narrative.
- Information reported on the CLP Forms or CLP-type Forms listed in this exhibit must either be typewritten or computer-generated. Handwritten corrections to the information on the CLP Forms and CLP-type Forms are not permitted. Notes or handwritten corrections on the hardcopy instrument output files must be legible, signed, and dated. Raw data consisting of handwritten worksheets should be completed in a legible fashion.
- Extraneous information should be kept to a minimum. Raw data pages, which contain no information pertaining to NYSDEC samples or QC relating to NYSDEC samples, should be excluded from the sample data package.
- Do not include redundant copies of the same supporting data in the data package. For example, if different sets of raw data reference the same standard prep log pages, include only one copy of the pages and link to it from the appropriate sections.

1.2 The contractor shall use NYSDEC Case Numbers, SDG Numbers, and NYSDEC Sample Numbers to identify samples received under this

contract, both verbally and in reports and correspondence. The Contract number shall be specified in all correspondence.

1.3 Sections III and IV of this Exhibit contain instruction for the required data reporting forms in CLP-specified formats, along with examples and templates for certain NYSDEC specific forms. Section V of this Exhibit contains the specifications for the .PDF file created for the data package. The format for electronic data deliverables (EDD) or other database compatible files are contained in Exhibit H.

1.4 In subsequent Sections of this document the words "copy" and "copies" are used when describing elements used to construct the Sample Data Package and Sample Data Summary Package. The terms "copy" and "copies", when used in this context, refer to Adobe .PDF pages produced from the original documents and included in the main .PDF file for the Package.

1.5 In all instances where a method detection limit (MDL), practical quantitation limit (PQL), or other detection limit (DL) must be reported along with the sample result, the appropriate limit should be adjusted based on the individual sample amount (mass or volume), dilution, and any additional factors they influence the limit being reported. This is referred to as the "sample specific detection limit". A sample specific detection limit should be reported along with all NYSDEC sample results, for all NYSDEC requested analytes to which a MDL, PQL, or DL applies. The only instance where the Laboratory may omit reporting of the sample specific detection limit is when a positive result is being reported for a specific analyte and the CLP/ASP Form I being used does not allow space for reporting of both a positive result and the sample specific detection limit.

1.6 Where applicable, the Laboratory shall include examples of the calculations used to arrive at the reported results. These sample calculations shall use the raw numbers from an actual sample (non-U flagged) in the data package, and show how the final reported result was arrived at for a randomly selected analyte. One sample calculation shall be included for each method used for reporting data in the SDG.

2.0 Resubmission of Data

2.1 If submitted documentation does not conform to the above criteria Section 1.1-1.4), the Laboratory will be required to resubmit such documentation with the deficiencies corrected within 6 business days, at no additional cost to NYSDEC.

2.2 Whenever the Laboratory is required to submit or resubmit data as a result of an on-site laboratory evaluation or through a Bureau of Watershed Assessment and Management (BWAM) action, or through a Project Officer's request, the data must be clearly marked as "ADDITIONAL DATA" and distributed to the specified data recipients. A cover letter must be included which describes what data is being delivered, to which NYSDEC sample(s) it pertains, and who requested the data.

2.3 Whenever the Contractor is required to submit or resubmit data as a result of Contact Compliance Screening (CCS) review by BWAM, the data shall be sent to the two contractual data recipients (BWAM and Region) and to NYSDEC's designated recipient when a written request for Sample Data Package has been made. In all instances the Contractor shall include a color-coded cover sheet (Laboratory Response to Results of Contract Compliance Screening) provided by BWAM. Electronic deliverable should be submitted or resubmitted to BWAM and the Region.

A. – Standard Operating Procedures

See Exhibits E and F for requirements

B. – Quality Assurance Management Plan

See Exhibits E and F for requirements

C. – Weekly Sample Receipt Summary

1.0 Weekly Sample Receipt Summaries shall be submitted by the Wednesday following the calender week (Sunday through Saturday) for which samples are submitted. This information must be transmitted electronically (emailed) as a Microsoft Excel compatible file. NYSDEC will provide the Excel file structure and all appropriate fields in the Excel file should be completed prior to submission.

1.1 The Weekly Sample Receipt Summary shall contain the following items:

- Lab name
- Contract number
- NYSDEC Case #
- NYSDEC SDG #
- NYSDEC Sample ID #
- Lab ID #
- Name of NYSDEC Sample Submitter
- Code numbers for requested analyses from Contract Laboratory Sample Information Sheet
- Sample Analysis Price – full sample price from contract for each sample # reported.
- List of NYSDEC sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt.

***Note**: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the highest sample number (considering both alpha and numeric designations).*

1.2 The NYSDEC SDG# is found on the Contract Laboratory Sample Information Sheet. The SDG number is also reported on all data reporting forms.

D. – Sample Data Summary Package

As specified in the Delivery Schedule, one Sample Data Summary Package CD-ROM each shall be delivered to the project officer and the sample collector concurrently with delivery of other required sample data. The Sample Data Summary Package consists of Adobe .PDF copies of specified items from the Sample Data Package. These items are listed below and described in detail under part E, Sample Data Package.

The Sample Data Summary Package shall be ordered as follows and shall be submitted separately either as a separate .PDF file or clearly separated by a bookmark in the Sample Data Package .PDF directly preceding the Sample Data Package. Sample data forms shall be arranged by fraction, in increasing NYSDEC sample number order, considering both letters and numbers. E400 is a lower sample number than RH100, as E precedes R in the alphabet.

Specifications for the book marking of electronic (.PDF) data packages are given in Section V of this Exhibit. Sections that must be bookmarked are annotated with **"<B-*X*>**", where *X* is the numeric level of the bookmark required for the given Section or subsection. For further information on bookmarking requirements see Part V, Section 1.3.6.

The Sample Data Summary Package shall contain all data for all samples within one Sample Delivery Group of the Case as follows:

1. NYSDEC Data Package Summary Forms **<B-1>**

2. SDG Narrative **<B-1>**

3. By fraction (VOA, SV, PEST, ARO, IN, WC) and by sample within each fraction – tabulated target compound results (FORM I-*XXXX*) and tentatively identified compounds (FORM I-*XXXX*-TIC) (VOA and BNA only). (**<B-1>** for the "Sample Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the results for each separate fraction and/or analysis method)

   ***Note:** "XXXX" represents the code for the appropriate organic data reporting form.*

4. By fraction (VOA, SV, PEST, and ARO) – surrogate spike analysis results (FORM II-*XXXX*) by matrix (water and/or soil) and for soil, by concentration (low or medium). (**<B-1>** for the "Surrogate Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the surrogate results for each separate fraction and/or analysis method)

5. By fraction (VOA, SV, PEST, and ARO) – matrix spike/matrix spike duplicate/matrix spike blank results (FORM III-*XXXX*) – as required by method. (**<B-1>** for the "MS/MSD Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the MS/MSD results for each separate fraction and/or analysis method)

6. By fraction (VOA, SV, PEST, and ARO) – QC Check Sample/Standard Recovery Summary – If required by method. (**<B-1>** for the "Check Sample/Standard Recovery" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the check standard results for each separate fraction and/or analysis method)

7. By fraction (IN and WC only) – duplicate sample results (FORM VI-IN). (**<B-1>** for the "Duplicate Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the duplicate results for each separate fraction and/or analysis method)

8. By fraction (IN and WC only) – spike sample results (FORM V-IN). (**<B-1>** for the "Spike Sample Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the spike results for each separate fraction and/or analysis method)

9. By fraction (VOA, SV, PEST, ARO, IN, WC) – blank data (FORM IV-*XXXX* (for organics) and Form III-IN) and tabulated results (FORM I-*XXXX* (for organics) and FORM I-IN) including tentatively identified compounds (FORM I-*XXXX*-TIC)(VOA and BNA only). (**<B-1>** for the "Blank Results" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the blank results for each separate fraction and/or analysis method)

10. By fraction (VOA and SV only) – internal standard area data (FORM VIII-*XXXX*). (**<B-1>** for the "Internal Standard Recovery" section of the Sample Data Package Summary, **<B-2>** to separate and mark the beginning of the internal standard recovery for each separate fraction and/or analysis method)

E. – Sample Data Package

The Sample Data Package is divided into the eight major units described below. The last six units are each specific to an analytical fraction (volatiles, semivolatiles, pesticides/Aroclors, GC organics, inorganics, and conventional wet-chemistry). If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable.

The Sample Data Package shall include data for analyses of all samples in one Sample Delivery Group, including field samples, re-analyses, blanks, duplicates, control spikes, matrix spikes, matrix spike duplicates, and matrix spike blanks. In addition, the package will also include the results of Method Detection Limit studies and reports establishing interelement correction factors for ICP-AES.

All data produced in support of Superfund investigation/remediation as identified by checked boxes under the Contract Laboratory Section of the Contract Laboratory Sample Information Sheet (CLSIS) (See Exhibit A) shall be reported as specified for the Superfund Category/CLP (Section 1.0 below). All data generated in support of the

SPDES program as identified by a CASE # beginning with the letter "E" shall be reported using ASP Category B (Section 3.0 below). All other samples shall be reported using either ASP Category A or ASP Category B described in Section 2.0 and 3.0 below. The specific reporting level to be used shall be specified by the CLSIS, unless otherwise specified in a project work plan.

The Laboratory shall retain a CD-ROM/.PDF copy of the Sample Data Package for 3 years after final acceptance of data. See Section V for a detailed explanation of these requirements. After this time, the Laboratory may dispose of/delete the package.

Specifications for the book marking of electronic (.PDF) data packages are given in Section V of this Exhibit. Sections that must be bookmarked are annotated with **"<B-*X*>**", where *X* is the numeric level of the bookmark required for the given Section.

1.0 Superfund Category/CLP

1.1 Cover Documentation **<B-1>**

Cover Page for the Data Package shall include: laboratory name; laboratory code; contract number; Case number; SDG number; and NYSDEC sample numbers in alphanumeric order.

1.2 SDG Narrative **<B-1>**

1.2.1 This document shall be clearly labeled "SDG Narrative" and shall contain: Laboratory name; Case number; Sample Delivery Group number (SDG); NYSDEC sample numbers in the SDG, differentiating between initial analyses and re-analyses; Contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package. For soil samples collected and pre-weighed in the field the laboratory shall document all discrepancies between sample weights determined in the field and in the laboratory in the SDG Narrative. A statement on the use of background and interelement corrections performed for the samples should be included for inorganic analysis, if applicable.

1.2.2 The Laboratory shall document, in the SDG Narrative, the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. The Laboratory shall also provide, in the SDG Narrative, sufficient information, including equations or curves (at least on equation or curve per method), to allow the recalculation of sample results from raw instrument output. The Laboratory shall also include a discussion of any performance-based modifications performed on the Protocol requirements or on published methods. If modifications are reoccurring, the laboratory may provide separate documentation of the modifications and reference such modifications in the SDG Narrative. Additionally, the Laboratory shall also identify and explain any differences that exist between the Form Is and the supporting documentation provided in the

data package and those previously provided as preliminary results.

1.2.3 The Contractor shall also provide, in the SDG Narrative or as attachments referenced in the SDG narrative, sufficient information, including copies of equations and definitions of variables (at least one equation per method), to allow the recalculation of sample results from raw instrument output.

1.2.4 All Gas Chromatography (GC) columns used for analysis should be documented in the SDG Narrative, by fraction. List the GC column identification—brand name, the internal diameter (in millimeters), and the length (in meters), packing/coating material, and film thickness. The trap used for volatile analysis shall be described here. List trap name, when denoted by the manufacturer, its composition (packing material/brand name, amount of packing material, in length). The Laboratory shall include any technical and administrative problems encountered, the corrective action taken, the resolution, and an explanation for all flagged edits (e.g. manual edits) on quantitation lists. The Laboratory shall document in the SDG Narrative all instances of manual integration.

1.2.5 Whenever data from sample re-analysis are submitted, the Laboratory shall state in the SDG Narrative for each re-analysis, whether it considers the re-analysis to be billable, and if so, why.

1.2.6 The Laboratory shall list the pH determined for each water sample submitted for volatile analysis. This information may appear as a simple list or table in the SDG Narrative. The purpose of this pH determination is to ensure that all water volatiles samples were acidified in the field. No pH adjustment is to be performed by the Laboratory on water samples for volatiles analysis. The SDG Narrative shall conclude with the following statement, verbatim: *"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this Sample Data Package and in the electronic data deliverables has been authorized by the Laboratory Manager or his/her designee, as verified by the following signature."* This statement shall be directly followed by signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

1.3 Sample Log-In Sheet [FORM DC-1] **<B-1>**

NOTE: *Example copies of the DC-1 form can be found in CLP Exhibit B. Use the DC-1 Form in OLM04.2 for organic samples and the DC-1 Form in ILM05.3 for inorganics/conventional samples.*

In addition to the DC-1 Form, the contractor must include a listing showing NYSDEC sample numbers, in alphanumeric order, cross-referenced with laboratory Sample ID numbers.

1.4 Contract Lab Sample Information Sheets **<B-1>**

A copy of the Contract Lab Sample Information Sheets (CLSIS) for all of the samples in the SDG. The CLSIS shall be arranged in increasing NYSDEC sample number order, considering both letters and numbering in ordering samples.

1.5 Chain-of-Custody Forms **<B-1>**

Copies of both the external and internal chain-of-custody sheets for all samples within the SDG.

1.6 Superfund-CLP Volatiles Data **<B-1>**

1.6.1 QC Summary **<B-2>**

1.6.1.1 System Monitoring Compound or Deuterated Monitoring Compound Recovery Reports (FORM II VOA-1, VOA-2, VOA-3, VOA-4, VOA-SIM, VOA-SIM1, VOA-SIM2).

1.6.1.2 Matrix Spike/Matrix Spike Duplicate/Matrix Spike Blank Recovery Reports (FORM III VOA-1, VOA-2, VOA-SIM) – Provided when an MS/MSD analysis is requested by NYSDEC.

1.6.1.3 Method Blank Summary (FORM IV VOA, VOS-SIM) – If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank, by instrument.

1.6.1.4 GC/MS Instrument Performance Check (FORM V VOA) – If more than a single form is necessary, the forms must be arranged in chronological order, by instrument.

***Note**: This form is not required for the optical analysis when submitting data using the Selected Ion Monitoring (SIM) technique.*

1.6.1.5 Internal Standard Area and RT Summary (FORM VIII VOA, VOA-SIM) – If more than a single form is necessary, the forms must be arranged in chronological order, by instrument.

1.6.2 Volatiles Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with the Organic Analysis Data Sheet (FORM I VOA-1, VOA-2, including FORM I VOA-TIC), followed by the raw data for volatile samples. The sample data shall be placed in order of increasing NYSDEC sample number, considering both letters and numbers. Volatile sample data for SIM analysis must be arranged

together with the rest of the SIM Volatiles data at the end of the sub-Section.

1.6.2.1 Target Compound Results – Volatile Organics Analysis Data Sheet (FORM I VOA-1, VOA-2) – Tabulated results (identification and quantitation) of the specified Superfund-CLP target compounds (Exhibit C – Volatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.6.2.2 Target Compound Results – Volatile Organics Analysis Data Sheet (FORM I VOA-1, VOA-2) – Tabulated results (identification and quantitation) of the specified Superfund-CLP target compounds (Exhibit C – Volatiles) shall be included. The validation and release of the results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.6.2.3 Tentatively Identified Compounds (FORM I VOA-TIC) – FORM I VOA-TIC is the tabulated list of the highest probable match for up to 10 organic compounds not system monitoring compounds and are not target compounds, system monitoring compounds, internal standard compounds, or unsubstituted alkanes, or any other compound not listed in Exhibit C – Volatiles. It including the CAS (Chemical Abstracts Registry) number, tentative identification and estimated concentrations. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. This form must be included even if no compounds are found. If this occurs, enter a "0" in the field for "Number found" on the form.

Note: *The Laboratory must be consistent, i.e., use peak height for all comparisons or use total area count for all comparisons.*

1.6.2.4 Reconstructed Total Ion Chromatograms (RIC) (for each sample including dilutions and reanalyzes) – RICs must be normalized to the largest non-solvent component and contain the following header information:

- NYSDEC sample number;
- Date and time of analysis;
- GC/MS instrument ID;
- Lab file ID;
- Analyst ID.

Note: *Each Selected Ion Current Profile (SICP) for samples taken through the optional analysis using the SIM technique shall be labeled as in this Section.*

1.6.2.4.1 Internal standard and system monitoring compounds should be labeled with the names of compounds, either directly out from the peak, or are to be included on a printout of retention times when the retention times are directly located over the peak. Labeling of the compounds is not required and should not detract from the legibility of the required labels.

1.6.2.4.2 If automated system procedures are used for preliminary identification and/or quantification of the Superfund Target Compound List (Superfund-TCL) compounds, the complete data system report must be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories that do not use the automated data system procedures, a laboratory "raw data sheet", which contains the following information, must be included in the sample data package in addition to the chromatogram.

- NYSDEC sample number;
- Date and time of analysis;
- RT or scan number of identified target compounds;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- On column concentration/amount, including units;
- GC/MS instrument ID;

- Lab file ID;
- Analyst ID.

1.6.2.4.3 In all instances where the data system report has been edited, or where manual integration or manual quantitation has been performed, the GC/MS operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the Extracted Ion Current Profile (EICP) of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – Volatiles, internal standards, and system monitoring compounds.

1.6.2.5 Other required Information. For each sample, by each compound identified, the following shall be included in the data package:

1.6.2.5.1 Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C – Volatiles that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Spectra must be labeled with NYSDEC sample number, lab file ID, date, and time of analysis, and GC/MS instrument ID. Compound names must be clearly marked on all spectra.

1.6.2.5.2 Copies of mass spectra of organic compounds not listed in Exhibit C (Superfund-TCL) (Tentatively Identified Compounds), with associated best-match spectra (the three best matches), as labeled in 1.6.2.4 above.

1.6.3 Standards Data **<B-2>**

1.6.3.1 Initial Calibration Data (FORM VI VOA-1, VOA-2, VOA-3, VOA-SIM) – shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.6.3.1.1 Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, as labeled in 1.6.2.4 above. Spectra are not required.

1.6.3.1.2 All initial calibration data that pertain to samples in the data package must be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

1.6.3.1.3 Labels for standards shall be descriptive of the concentrations of the non-ketone (majority) analytes in µg/L.

1.6.3.1.4 EICPs displaying each manual integration.

1.6.3.2 Continuing Calibration (FORM VII VOA-1, VOA-2, VOA-3, VOA-SIM) – shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.6.3.2.1 Volatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibration verifications, as labeled in 1.6.2.4. Spectra are not required.

1.6.3.2.2 When more than one Continuing Calibration Verification is performed, forms must be in chronological order, by instrument.

1.6.3.2.3 EICPs displaying each manual integration.

1.6.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS Operator shall identify such edits or manual procedures by initializing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integration area with the letter "m" on the quantitation report. In addition a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – Volatiles, internal standards, and system monitoring compounds.

1.6.4 Volatiles Raw QC Data **<B-2>**

1.6.4.1 4-Bromofluorobenzene (BFB) shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized. **<B-3>**

1.6.4.1.1 Bar graph spectrum, as labeled in 1.6.2.4.

1.6.4.1.2 Mass listing, as labeled in 1.6.2.4.

1.6.4.1.3 Reconstructed total ion chromatogram (RIC), labeled as in 1.6.2.4.

1.6.4.2 Blank Data shall be arranged by type of blank (method, storage, instrument) and shall be in chronological order, by instrument. **<B-3>**

***Note**: This order is different from that used for sample data (Section 1.6.2).*

1.6.4.2.1 Tabulated results (FORM I VOA-1, VOA-2, VOA-SIM).

1.6.4.2.2 Tentatively Identified Compounds (FORM I-TIC) – even if none are found.

1.6.4.2.3 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled as in 1.6.2.4.

1.6.4.2.4 Target compound spectra with laboratory-generated standard, labeled as in 1.6.2.4. Data systems that are incapable of dual display shall provide spectra in the following order:

- Raw target compound spectra;
- Enhanced or background-subtracted spectra;
- Laboratory generated standard spectra.

1.6.4.2.5 GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in 1.6.2.4.

1.6.4.2.6 Quantitation/calculation of TIC concentrations.

1.6.4.3 Matrix Spike Blank Data **<B-3>**

1.6.4.3.1 Tabulated results (FORM I VOA-1, VOA-2, VOA-SIM) of all target compounds. Form I VOA-TIC is <u>not</u> required.

1.6.4.3.2 Reconstructed ion chromatogram(s) and quantitation report(s), as labeled in 1.6.2.4. Spectra are <u>not</u> required.

1.6.4.4 Matrix Spike Data **<B-3>**

1.6.4.4.1 Tabulated results (FORM I VOA-1, VOA-2) of all target compounds. FORM I VOA-TIC is <u>not</u> required.

1.6.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s), as labeled in 1.6.2.4. Spectra are not required.

1.6.4.5 Matrix Spike Duplicate Data **<B-3>**

1.6.4.5.1 Tabulated results (FORM I VOA) of all target compounds. FORM I VOA-TIC is not required.

1.6.4.5.2 Reconstructed ion chromatogram(s) and quantitation report(s), as labeled in 1.6.2.4. Spectra are not required.

1.6.5 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied, as well as, values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.6.6 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.7 Semivolatiles Data **<B-1>**

1.7.1 Semivolatiles QC Summary **<B-2>**

1.7.1.1 System Monitoring Compound Percent Recovery Summary (FORM II SV-1, SV-2, SV-3, SV-4, SV-SIM).

1.7.1.2 Matrix Spike/Matrix Spike Duplicate Summary (FORM III SV-1, SV-2, SV-SIM) - Provided when an MS/MSD analysis is requested by NYS DEC.

1.7.1.3 Method Blank Summary (FORM IV SV, SV-SIM) – If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

1.7.1.4 GC/MS Instrument Performance Check (FORM V SV) – If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

Note: *This form is not required when submitting data for the analysis of Polynuclear Aromatic Hydrocarbons (PAHs)/phenols using the SIM technique.*

1.7.1.5 Internal Standard Area and RT Summary (FORM VIII SV-1, SV-2) – If more than a single form is necessary, the forms shall be arranged in chronological order, by instrument.

1.7.1.6 Instrument Detection Limits.

1.7.2 Semivolatile Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with the Semivolatile Organics Analysis Data Sheet (FORM I SV-1, SV-2, including FORM I SV-TIC), followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing DEC sample number, considering both letters and numbers in ordering samples.

1.7.2.1 Target Compound Results, Semivolatiles Organics Analysis Data Sheet (FORM I SV-1, SV-2) – Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C – CLP Semivolatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.7.2.2 Semivolatile Tentatively Identified Compounds (FORM I SV-TIC) – Form I SV-TIC is the tabulated list of the highest probable match for up to 20 organic compounds that are not target compounds, system monitoring compound, internal standard compounds, and are not listed in Exhibit C – CLP Volatiles and Semivolatiles. It includes the CAS number (if applicable), tentative identification, and estimated concentration. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. This form must be included even if no compounds are found. If this occurs, enter a "0" in the field for "Number found" on the form.

Note: *This form is not required when submitting data for the optional analysis of PAHs/phenols using the SIM technique.*

Note*: The Laboratory must be consistent, i.e., use peak height for all comparisons or use total area count for all comparisons.*

1.7.2.3 PAHs/Phenols Analysis Data Sheet (FORM I SV-SIM) – This data form shall be submitted upon the NYS DEC's request for optional analysis of PAHs/phenols using the SIM technique. The specific target PAHs/phenols listed in Exhibit C – CLP Semivolatiles shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.7.2.4 Reconstructed Total Ion Chromatograms (RICs) (for each sample, including dilutions and reanalyzes). RICs must be normalized to the largest non-solvent component, and must contain the following header information:

- NYSDEC sample number;
- Date and time of analysis;
- GC/MS instrument ID;
- Lab file ID; and
- Analyst ID.

1.7.2.4.1 Internal standards and system monitoring compounds are to be labeled on RICs or SICPs with the names of compounds, either directly out from the peak, or are to be included on a printout of retention times if the retention times are printed directly over the peak.

1.7.2.4.2 If automated data system procedures are used for preliminary identification and/or quantification of the target compound, the complete data system report shall be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram or SICP for optional PAHs/phenols analysis. The complete data system report shall include all of the information listed below. For laboratories that do not use the automated data system procedures, a laboratory "raw data sheet," containing the following information, shall be included in the Sample Data Package, in addition to the chromatogram.

- NYSDEC sample number

- Date and time of analysis
- RT or scan number of identified Superfund-TCL compounds
- Ion used for quantitation with measured area
- Copy of area table from data system
- GC/MS instrument ID
- Lab file ID

1.7.2.4.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – CLP Semivolatiles, internal standards, and system monitoring compounds.

1.7.2.5 Other Required Information – For each sample, by each compound identified, the following shall be included in the data package:

1.7.2.5.1 Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C – CLP Semivolatiles that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. This includes PAH/phenol target compounds that are identified during the optional analysis using the SIM technique. Spectra shall be labeled with NYS DEC sample number, laboratory file ID, date, and time of analysis, and GC/MS instrument ID. Compound names must be clearly marked on all spectra.

1.7.2.5.2 Copies of mass spectra of non-system monitoring/non-internal standard organic compounds not listed in Exhibit C – CLP Semivolatiles with associated best-match spectra (maximum of three best matches). This

includes the mass spectra for tentatively identified alkanes. Spectra shall be labeled with NYS DEC Sample Number, laboratory file ID, date and time of analysis, and GC/MS instrument ID. Compound names shall be clearly marked on all spectra.

1.7.3 Semivolatiles Standards Data **<B-2>**

1.7.3.1 Initial Calibration Data (FORM VI SV-1, SV-2, SV-3) or FORM VI SV-SIM (when optional analysis of PAHs/phenols is performed) shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.7.3.1.1 Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five-point) calibration, labeled in 1.7.2.4. Spectra are not required.

1.7.3.1.2 When optional analysis of PAHs/phenols is requested, then SICPs and quantitation reports for the initial calibration standards (five-point), labeled as in Section 1.7.2.4, shall be submitted. Spectra are not required.

1.7.3.1.3 All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which SDG. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

1.7.3.1.4 Labels for standards shall reflect the concentrations of the majority of the analytes in µg/L.

1.7.3.1.5 EICPs displaying each manual integration.

1.7.3.2 Continuing Calibration Verification Data (FORM VII SV-1, SV-2, SV-3) or FORM VII SV-SIM (when optional analysis of PAHs/phenols is performed) shall be included in order by instrument, if more than one instrument used. **<B-3>**

1.7.3.2.1 Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for all opening, closing, and continuing calibrations verifications, as labeled in Section 1.7.2.4. Spectra are not required.

1.7.3.2.2 When optional analysis of PAHs/phenols is requested, then SICPs and quantitation reports

for all opening, closing, and CCVs, labeled as in Section 1.7.2.4. Spectra are not required.

1.7.3.2.3 When more than one continuing calibration is performed, forms must be in chronological order, by instrument.

1.7.3.2.4 EICPs displaying each manual integration.

1.7.3.3 In all instances where the data system report has been edited, or where the manual integration or quantitation has been performed, the GC/MS Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integration area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C – CLP Semivolatiles, internal standards, and system monitoring compounds.

1.7.4 Semivolatiles Raw Quality Control (QC) Data **<B-2>**

1.7.4.1 Decafluorotriphenylphosphine (DFTPP) data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized. **<B-3>**

1.7.4.1.1 Bar graph spectrum, as labeled in 1.7.2.4.

1.7.4.1.2 Mass listing, as labeled in 1.7.2.4.

1.7.4.1.3 Reconstructed total ion chromatogram (RIC), labeled as in 1.7.2.4.

1.7.4.2 Blank Data shall be in chronological order by extraction date. **<B-3>**

Note: *This order is different from that used for samples.*

1.7.4.2.1 Tabulated results (FORM I SV-1, SV-2, SV-SIM).

1.7.4.2.2 Tentatively Identified Compounds (FORM I SV-TIC) – even if none found.

1.7.4.2.3 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled in 1.7.2.4.

1.7.4.2.4 Target compound spectra with laboratory-generated standard, as labeled in 1.7.2.4. Data

systems that are incapable of dual display shall provide spectra in the following order:

- Raw target compound spectra;
- Enhanced or background-subtracted spectra;
- Laboratory-generated standard spectra.

1.7.4.2.5 GC/MS library search spectra for Tentatively Identified Compounds (TICs), as labeled in 1.7.2.4.

1.7.4.2.6 Quantitation/Calculation of TIC concentrations.

1.7.4.3 Semivolatiles Matrix Spike Blank Data **<B-3>**

1.7.4.3.1 Tabulated results (FORM I SV) of all target compounds. Form I SV-TIC not required.

1.7.4.3.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled in 1.7.2.4. Spectra are required.

1.7.4.4 Semivolatiles Matrix Spike Duplicate Data **<B-3>**

1.7.4.4.1 Tabulated results (FORM I SV-1, SV-2) of all target compounds. FORM I SV-TIC is not required.

1.7.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), as labeled in 1.7.2.4. Spectra are not required.

1.7.4.5 Semivolatile Gel Permeation Chromatography (GPC) Data – The two most recent Ultra Violet (UV) traces of the (GPC) calibration solution, and the reconstructed ion chromatogram and data system reports for the GPC blank shall be arranged in chronological order by GPC for the GPC calibration. **<B-3>**

1.7.4.5.1 Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak.

1.7.4.5.2 Reconstructed ion chromatogram and data system report(s) labeled as specified in Section 1.7.2.4 for the GPC blank analysis.

1.7.4.5.3 Reconstructed ion chromatogram and data system report(s) for all standards used to quantify compounds in the GPC blank, labeled, as specified in section 1.7.2.4.

1.7.5 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.7.6 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.8 Pesticide Data **<B-1>**

1.8.1 Pesticide QC Summary **<B-2>**

1.8.1.1 Surrogate Recovery (FORM II PEST-1, PEST-2)

1.8.1.2 Matrix Spike/Matrix Spike Duplicate/Matrix Spike Blank Recovery (FORM III PEST-1, PEST-2): MS/MSD is required for the Pesticide fraction of an SDG, unless otherwise specified by the NYS DEC.

1.8.1.3 Laboratory Control Sample Recovery (FORM III PEST-1, PEST-2).

1.8.1.4 Method Blank Summary (FORM IV PEST): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

1.8.2 Pesticide Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with the Pesticide Organic Analysis Data Sheet (FORM I PEST), followed by the raw data for pesticide samples. These sample packets should then be

placed in increasing NYSDEC sample number order, considering both letters and numbers in ordering samples.

1.8.2.1 Target Compound Results, Pesticide Organics Analysis Data Sheet (FORM I PEST). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C – CLP Pesticides) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 1.2). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.8.2.2 Copies of Pesticide Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of RTs on the data system printout if RTs are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following information:

- NYSDEC sample number;
- Volume injected (µL);
- Date and time of injection;
- On column concentration/ amount including units;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

1.8.2.3 Copies of pesticide chromatograms from second GC column shall be included and labeled as in Section 1.8.2.2.

1.8.2.4 Data System Printout. A printout of RT, corresponding peak height or peak area, and on the column amount shall accompany each chromatogram. The printout shall be labeled with the NYS DEC sample number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the Gas Chromatograph/Electron Capture Detector (GC/ECD) Operator shall identify all such edits or manual procedures by initialing and dating the

changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integration area with the letter "m" on the quantitation report.

1.8.2.5 All manual worksheets shall be included in the Sample Data Package.

1.8.2.6 Other Required Information. If pesticides are confirmed by GC/MS, the Laboratory shall submit copies of reconstructed ion chromatograms, raw spectra, and background-subtracted mass spectra of target compounds listed in Exhibit C – CLP Pesticides that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Compound names shall be clearly marked on all spectra. For Toxaphene confirmed by GC/MS, the Laboratory shall submit mass spectra of 3 major peaks from samples and standards.

1.8.3 Pesticides Standards Data **<B-2>**

1.8.3.1 Initial Calibration of Single Component Analytes (FORM VI PEST-1, PEST-2): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.2 Initial Calibration of Multicomponent Analytes (Toxaphene, etc.) (FORM VI PEST-3, PEST-4): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.3 Analyte Resolution Check Summary (FORM VI PEST-5): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.4 Performance Evaluation Mixture (PEM) (FORM VI PEST-6): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.5 Individual Standard Mixture A (FORM VI PEST-7): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.6 Individual Standard Mixture B (FORM VI PEST-8): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.7 Individual Standard Mixture C (FORM VI PEST-9, PEST-10): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.8 Calibration Verification Summary (FORM VII PEST-1): For all mid-point concentrations of Individual Standard Mixtures A and B or C and instrument blanks used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instruments. **<B-3>**

1.8.3.9 Calibration Verification Summary (FORM VII Pest-2, Pest-3): For all mid-point concentrations of Individual Standard Mixtures A and B or C and instrument blanks used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.10 Analytical Sequence (FORM VIII PEST): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.8.3.11 Florisil Cartridge Check (FORM IX PEST-1): For all lots of cartridges used to process samples in the SDG, using Individual Standard Mixtures A or C. **<B-3>**

1.8.3.12 GPC Calibration Verification (FORM IX PEST-2): For all GPC columns, in chronological order by calibration verification date. **<B-3>**

1.8.3.13 Identification Summary for Single Component Analytes (FORM X PEST): For all samples with positively identified single component analytes, in order by increasing NYSDEC Sample Number. **<B-3>**

1.8.3.14 Chromatograms and data system printouts are required for all standards including the following: **<B-3>**

- Resolution Check Mixture.
- Performance Evaluation (PE) mixtures, all.
- Individual Standard Mixture A and B, both at five concentrations, for each initial calibration and Individual Standard Mixture B, at five concentrations, for each initial calibration.

Or

- Individual Standard Mixture C, at five concentrations, each initial calibration.
- Toxaphene, at five concentrations, each initial calibration.

- All mid-point concentrations of Individual Standard Mixtures A and B or C used for calibration verification.
- All toxaphene standards analyzed for confirmation.
- All lots of Florisil cartridge check solution
- Pesticide GPC Calibration Check Solution, all calibrations relating to samples in the SDG.
- All multicomponent analyte standards analyzed for confirmation.

1.8.3.15 A printout of RT and corresponding peak height or peak areas shall accompany each chromatogram. The printout shall be labeled with the NYSDEC Sample Number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following: **<B-3>**

- NYSDEC Sample Number for the standard (e.g., INDA10K, INDA20K, etc., See Forms Instructions for details);
- Label all standard peaks for all individual compounds either directly out from the peak or on the printout of retention times if retention times are labeled over the peak;
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram;
- Date and time of injection;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

Note: *In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator shall identify such edits or manual procedures by initialing and changes made to the report, shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report.*

1.8.4 Pesticides Raw Quality Control (QC) Data **<B-2>**

1.8.4.1 Blank Data shall be arranged by type of blank (method, instrument, sulfur cleanup) and shall be in chronological order by instrument. **<B-3>**

1.8.4.1.1 Tabulated results (FORM I PEST).

1.8.4.1.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.2 Pesticide LCS Data **<B-3>**

1.8.4.2.1 Tabulated results (FORM I PEST) of target compounds for both GC columns.

1.8.4.2.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.3 Pesticides Matrix Spike Data **<B-3>**

1.8.4.3.1 Tabulated results (FORM I PEST) of target compounds for both GC columns.

1.8.4.3.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.4 Pesticides Matrix Spike Duplicate Data **<B-3>**

1.8.4.4.1 Tabulated results (FORM I PEST) of target compounds for both GC columns.

1.8.4.4.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.4.5 Matrix Spike Blank Data **<B-3>**

1.8.4.5.1 Tabulated results (FORM I-CLP-PEST) of all Superfund-TCL compounds.

1.8.4.5.1.1 Chromatogram(s) and data system printout(s) (GC), as labeled in 1.8.2.2 and 1.8.2.4 above.

1.8.5 Raw Gel Permeation Chromatograph (GPC) Data **<B-2>**

1.8.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.

1.8.5.1.1 UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of the RTs when the RTs are printed directly over the peak.

1.8.5.1.2 Chromatograms and data system report(s) labeled as specified in Sections 1.8.2.2 and 1.8.2.4 above.

1.8.5.1.3 Chromatograms and data system report(s) for all standards used to identify compounds in the GPC blank labeled as specified in Section 1.8.3.14 and 1.8.3.15 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, Individual Standard Mixture C, and the Toxaphene standards).

1.8.5.2 GPC Calibration Verification. The Chromatogram and the data system report(s) shall be arranged in chronological order for the GPC calibration check.

1.8.5.2.1 Chromatograms and data system printouts labeled as specified in Sections 1.8.2.2 and 1.8.2.4 for the GPC calibration verification solution analyses.

1.8.5.2.2 Chromatogram and the data system report(s) for the standards used to quantify compounds in the GPC calibration verification solution labeled as specified in Section 1.8.3.14 and 1.8.3.15 (i.e., Individual Standard Mixtures A and B or C from the initial calibration sequence).

1.8.6 Raw Florisil Data **<B-2>**

1.8.6.1 The chromatogram and the data system report(s) shall be arranged in chronological order by Florisil cartridge performance check analysis.

1.8.6.1.1 Chromatograms and data system reports, labeled as specified in Sections 1.8.2.2 and 1.8.2.4 for the Florisil cartridge performance check analysis.

1.8.6.1.2 Chromatograms and data system reports for standard analyses used to quantify compounds in the Florisil cartridge performance check analysis, labeled as specified in Section 1.8.3.14 and 1.8.3.15 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, Individual Standard Mixture C, and the 2,4,5-Trichlorophenol solution).

1.8.7 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.8.8 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.9 Aroclor Data **<B-1>**

1.9.1 Aroclor QC Summary **<B-2>**

1.9.1.1 Surrogate Recovery (FORM II ARO-1, ARO-2).

1.9.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (FORM III ARO-1, ARO-2): MS/MSD is required for the Aroclor fraction, unless otherwise specified by NYSDEC. One MS/MSD set is required per SDG.

1.9.1.3 LCS Recovery (FORM III ARO-3, ARO-4).

1.9.1.4 Method Blank Summary (FORM IV ARO): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

1.9.2 Aroclor Sample Data (**<B-2>** to mark Section heading, **<B-3>** to mark the beginning of each data "packet")

Sample data shall be arranged in packets with Aroclors Organics Analysis Data Sheet (FORM 1 ARO), followed by the raw data for Aroclor samples. These sample packets should then be placed in

order of increasing NYSDEC Sample Number, considering both letters and numbers.

Note: *For a Sample analysis in which "S" flags are reported a FORM I ARO is required for the original analysis (NYSDEC Sample Number = XXXXX) in which the "S" flags are reported, and a FORM I ARO is required for the billable reanalysis (NYSDEC Sample Number = XXXXXRE) of the sample performed after a valid 5-point calibration of the detected Aroclor. An additional FORM I ARO is required for any necessary dilutions (NYSDEC Sample Number = XXXXXDL).*

1.9.2.1 Target Compound Results, Aroclors Organics Analysis Data Sheet (FORM I ARO). Tabulated results (identification and quantification) of the specified target compounds (Exhibit C – Aroclors) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (Section 1.2). In the event that the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

1.9.2.2 Copies of Aroclor Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of the RTs on the data system printout if the RTs are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following information:

- EPA Sample Number;
- Volume injected (µL);
- Date and time of injections;
- On column concentration/amount including units;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

1.9.2.3 Copies of Aroclor chromatograms for the second GC column shall be included and labeled as in Section 1.9.2.2.

1.9.2.4 Data System Printout

A printout of RT, corresponding peak height or peak area, and the on column amount shall accompany each

chromatogram. The printout shall be labeled with the EPA Sample Number and standard concentration level. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" in the quantitation report.

1.9.2.5 All manual worksheets shall be included in the Sample Data Package.

1.9.2.6 Other Required Information. If Aroclors are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms. Raw spectra and background-subtracted mass spectra must be submitted for at least three major peaks of Aroclor target compounds (see Exhibit C – Aroclors) that are identified in the sample and corresponding standard mass spectra. Compound names shall be clearly marked on all spectra.

1.9.3 Aroclor Standard Data **<B-2>**

1.9.3.1 Initial Calibration of Aroclors (FORM VI ARO-1, ARO-2, and ARO-3): For all GC columns, all instruments, in chronological order by GC column and instrument. **<B-3>**

1.9.3.2 Calibration Verification Summary (FORM VII ARO): For all calibration verification standards on all GC columns and instruments, in chronological order by GC column and instruments. **<B-3>**

1.9.3.3 Analytical Sequence (FORM VIII ARO): For all GC columns and instruments, in chronological order by GC column and instrument. **<B-3>**

1.9.3.4 Identification Summary for Multicomponent Analytes (FORM X ARO): For all samples with positively identified Aroclors, in order by increasing EPA Sample Number. **<B-3>**

1.9.3.5 Chromatograms and data system printouts shall be included for all standards, including the following:

- All Aroclor standards used for initial calibration on each column and instrument.
- All Aroclor standards used for calibration verification on each GC column and instrument.

- All Aroclor standards analyzed for confirmation.

1.9.3.6 A printout of RT and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA Sample Number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D, and shall be labeled with the following:

- NYSDEC Sample Number for the standard (e.g., AR10161OK, AR12601OK).
- Label all standard peaks with the compound name, either directly out from the peak on the chromatogram, or on the printout of RTs on the data system printout, if RTs are printed over the peak on the chromatogram.
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram.
- Date and time of injection.
- GC column identifier (by stationary phase and internal diameter).
- GC instrument identifier.
- Scaling factor (label the x and y axes using a numerical scale).

Note: *In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report.*

1.9.4 Aroclor Raw Quality Control (QC) Data **<B-2>**

1.9.4.1 Blank data shall be arranged in chronological order by extraction data. **<B-3>**

Note: *This order is different from that used for samples.*

- Tabulated results (FORM I ARO).
- Chromatogram(s) and data system printout(s) for each GC column and instrument used for analysis, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.4.2 Aroclor Laboratory Control Sample (LCS) Data **<B-3>**

- Tabulated results (FORM I ARO) of target compounds for both GC columns.

- Chromatograms and data system printouts for both GC columns, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.4.3 Aroclors Matrix Spike Data **<B-3>**

- Tabulated results (FORM I ARO) of target compounds for both GC columns.

- Chromatograms and data system printouts for both GC columns, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.4.4 Aroclors Matrix Spike Duplicate Data **<B-3>**

- Tabulated results (FORM I ARO) of target compounds for both GC columns.

- Chromatograms and data system printouts for both GC columns, labeled as in Sections 1.9.2.2 and 1.9.2.4.

1.9.5 Raw Gel Permeation Chromatography (GPC) Data **<B-2>**

1.9.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of RTs when the RTs are printed directly over the peak.

- Chromatograms and data system report(s) labeled as specified in Sections 1.9.2.2 and 1.9.2.4 for the GPC blank analyses.

- Chromatogram and data system report(s) for all standards used to assess the Aroclor pattern, labeled as specified in Section 1.9.2.2 and 1.9.2.4 (i.e., AR10161OK, AR12601OK from the initial calibration).

1.9.5.2 GPC Calibration Verification. The chromatogram and the data system reports(s) shall be arranged in chronological order for the GPC calibration check.

- Chromatograms and data system report(s) for standards used to assess the Aroclor pattern, labeled as specified in Sections 1.9.2.2 and 1.9.2.4 (i.e., Aroclor Standard Mixture 1016/1260 from the initial calibration sequence).

1.9.6 Copy of Calculations **<B-2>**

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

1.9.7 Copy of Extraction Logs **<B-2>**

These logs must be legible and include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. matrix spike, matrix spike duplicate, matrix spike blank) correspond to each batch extracted, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) final volumes and vial identification numbers.

1.10 Inorganic Data **<B-1>**

Sample data shall be submitted with the Inorganic Analysis Data Reporting Forms for all samples in the SDG, arranged in increasing alphanumeric NYSDEC sample number order, followed by the QC analyses data, quarterly and annual verification of method and instrument parameter forms, raw data, and copies of the digestion and distillation logs.

1.10.1 Results – Inorganic Analysis Data Sheet [FORM IA-IN and FORM IB-IN] – Tabulated analytical results (identification and quantitation) of the requested analytes (Exhibit C) must be accompanied by a signed statement in the SDG narrative. This signature validates and allows for the release the results. If the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page. (**<B-2>** marking the beginning of results from each new fraction and/or analysis method)

1.10.1.1 Appropriate concentration units must be specified and entered on FORM IA-IN and FORM IB-IN. The quantitative values shall be reported in units of micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. Other units are acceptable only for trace level analyses. Results for solid sample must be reported

on a dry weight basis. Analytical results must be reported to two significant figures if the result value is less than 10 and to three significant figures if the value is greater than or equal to 10. Results for percent solids must be reported to one decimal place. The preceding discussion concerning significant numbers applies to FORM IA-IN, IB-IN, and IX-IN only. For the other forms, follow the Reporting Requirements and Order of Data Deliverables (Con't) instructions specific to those forms as discussed in this exhibit.

1.10.2 Quality Control (QC) Data **<B-2>**

1.10.2.1 The QC Summary for inorganic analysis shall contain the forms listed below.

Note: *If more than one form is necessary, duplicate forms must be arranged in chronological order.*

1.10.2.1.1 Initial and Continuing Calibration Verification [FORM IIA-IN] **<B-3>**

1.10.2.1.2 CRQL Check Standard [FORM IIB-IN]

1.10.2.1.3 Blanks [Form III-IN] **<B-3>**

1.10.2.1.4 ICP-AES Interference Check Sample [FORM IVA-IN] **<B-3>**

1.10.2.1.5 ICP-MS Interference Check Sample [FORM IVB-IN] **<B-3>**

1.10.2.1.6 Matrix Spike Sample Recovery [FORM VA-IN] **<B-3>**

1.10.2.1.7 Post-Digestion Spike Sample Recovery [FORM VB-IN] **<B-3>**

1.10.2.1.8 Duplicates [FORM VI-IN] **<B-3>**

1.10.2.1.9 Laboratory Control Sample [FORM VII-IN] **<B-3>**

1.10.2.1.10 ICP-AES and ICP-MS Serial Dilutions [FORM VIII-IN] **<B-3>**

1.10.2.1.11 Method Detection Limits (Annually) [FORM IX-IN] **<B-3>**

1.10.2.1.12 ICP-AES Interelement Correction Factors (Quarterly) [FORM XA-IN] **<B-3>**

1.10.2.1.13 ICP-AES Interelement Correction Factors (Quarterly) [FORM XB-IN] **<B-3>**

1.10.2.1.14 ICP-AES and ICP-MS Linear Ranges (Quarterly) [FORM XI-IN] **<B-3>**

1.10.2.1.15 Preparation Log [FORM XII-IN] **<B-3>**

1.10.2.1.16 Analysis Run Log [FORM XIII-IN] **<B-3>**

1.10.2.1.17 ICP-MS Tune [FORM XIV-IN] **<B-3>**

1.10.2.1.18 ICP-MS Internal Standards Relative Intensity Summary [FORM XV-IN] **<B-3>**

***Note**: Copies of Verification of Instrument Parameters forms for the current quarter must be submitted with each data package*.

1.10.3 Raw Data **<B-2>**

For each reported value, the Laboratory shall include in the Sample Data Package all raw data from the instrument used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample results. This statement does not apply to the quarterly and annual Verifications of Instrument Parameters submitted as part of each Sample Data Package. When analysis of the ICP-AES or ICP-MS target analytes listed in Exhibit C (or any subset or additional analytes) is requested, the raw data shall include, for all samples, not only the results for the requested analytes(s), but also those for all the interferents. The raw data shall also contain the results of any other analyte(s), which have been determined to interfere with the requested analyte(s).

1.10.3.1 Raw data must contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Batch Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the Method Detection Limit (MDL). Raw data shall not be corrected for dilutions or volume adjustments. All Atomic Absorption (AA), Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES), and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) instruments shall provide a legible hardcopy of the direct real-time instrument readout (i.e., strip charts, printer tapes, etc.) or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included. A hardcopy of the instrument's direct sequential readout shall be included for cyanide if the instrument has the capability.

1.10.3.2 The order of raw data in the Sample Data Package for inorganic analyses shall be: ICP-AES, Graphite Furnace Atomic Adsorption (GFAA), ICP-MS, Mercury, and Cyanide. All raw data shall include concentration units for ICP and absorbance or concentration units for AA, Mercury, and Cyanide. (**<B-3>** marking the beginning of raw data for each separate method)

1.10.3.3 The ICP-MS raw data shall also contain the turbidity measurement results [in Nephelometric Turbidity Units (NTU)] for the field samples.

1.10.3.4 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

1.10.3.5 Raw data shall be labeled with NYSDEC sample number and appropriate codes, as shown in Exhibit B, "Table 2 – Codes for Labeling Data", to unequivocally identify:

- Calibration standards, including source and preparation date. Standard preparation logbooks can be submitted if they contain this information;
- Initial and Continuing Calibration Blanks (ICBs/CCBs) and Preparation Blanks (PBs).
- Initial and Continuing Calibration Verification (ICV/CCV) standards, Interference Check Samples, serial dilution samples, Contract Required Quantitation Limit (CRQL), Check Standard (CRI), Laboratory Control Sample (LCS), and Post Digestion Spike;
- Diluted and undiluted samples (by NYSDEC sample number) and all weights, dilutions, and volumes used to obtain the reported values (if the volumes, weights and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient);
- Duplicates;
- Spikes (indicating standard solutions used, final spike concentrations, and volumes involved). If spike information (source, concentration, volume) is consistent for a given SDG, a general statement outlining these parameters is sufficient;

- Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation; and

- Time and date of each analysis. Instrument run logs can also be submitted if they contain time and date of analysis. If the instrument does not automatically provide times of analysis, these shall be manually entered on all raw data (e.g., ICV/CCV, blanks, and the CRQL check standard.

- All information for furnace analysis clearly and sequentially identified on the raw data, including DEC sample number, sample and analytical spike data, percent recovery, coefficient of variation, full MSA data, MSA correlation coefficient, slope and intercepts of linear fit, final sample concentration (standard addition concentration), and type of background correction used (BS for Smith-Heiftje, BD for deuterium Arc, or BZ for Zeeman).

- Integration times for AA analyses.

1.10.3.6 Digestion and Distillation Logs. The following logs shall be submitted as appropriate for each preparation procedure: digestion logs for ICP-AES, ICP-MS, mercury preparations, and cyanide. These logs shall include: (1) date; (2) sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; (3) sufficient information to unequivocally identify which QC samples (i.e., LCS, PB) correspond to each batch digested; (4) comments describing any sufficient sample changes or reactions which occur during preparation shall be entered in the log and noted in the SDG Narrative; (5) indication of pH less than 2 or greater than 12, as applicable; and (6) identification of the sample preparer(s) [signature(s)]. **<B-3>**

1.10.4 Copy of Calculations – The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the instrument output report. If manipulations are performed by a software package, a copy of the formula used must be supplied, as well as, values for all terms in the formula. **<B-2>**

Note: *All correction factors and equations utilized must be indicated on the work sheet.*

2.0 ASP Category A

2.1 Cover Documentation **<B-1>** - See Requirements listed in Section 1.1 above.

2.2 SDG Narrative **<B-1>** - See Requirements listed in Section 1.2 above.

2.2.1 In addition to the requirements listed in Section 1.2, the Laboratory shall also document any out of range QC parameters associated with the data. Indicate what QC parameters were out of control, the limit that was exceeded, the result of the QC in exceedance, what samples are associated with that QC item, and how the results of those samples may be affected by the out of range QC.

2.3 Contract Lab Sample Information Sheets **<B-2>** - See Requirements listed in Section 1.4 above.

2.4 Chain-of-Custody Forms **<B-1>** - See Requirements listed in Section 1.5 above.

2.5 NYSDEC Data Package Summary Forms **<B-1>** - Requirements and Instructions for these forms are listed in Section IV of this Exhibit.

2.6 GC/MS Volatiles Data **<B-1>**

2.6.1 Sample Data

Sample data shall be arranged in packets consisting of the respective "Organic Analysis Data Sheet" (FORM I VOA-1, VOA-2) followed by the FORM I VOA-TIC for that sample. These packets shall be arranged in order of increasing NYSDEC sample number, considering both numbers and letters. For a detailed explanation of the Volatile FORM I requirements, see Sections 1.6.2.1 and 1.6.2.2 above.

2.7 GC/MS Semivolatiles Data **<B-1>**

2.7.1 Sample Data

Sample data shall be arranged in packets consisting of the respective "Organic Analysis Data Sheet" (FORM I SV-1, SV-2, SV-SIM) followed by the FORM I SV-TIC for that sample. These packets shall be arranged in order of increasing NYSDEC sample number, considering both numbers and letters. For a detailed explanation of the Semivolatile FORM I requirements, see Sections 1.7.2.1, 1.7.2.2, and 1.7.2.3 above.

2.8 Pesticide Data **<B-1>**

2.8.1 Sample Data

Sample data shall be reported on individual "Organic Analysis Data Sheet(s)" (FORM I PEST). These forms shall be arranged in order of increasing NYSDEC sample number, considering both

numbers and letters. For a detailed explanation of the Pesticide FORM I requirements, see Sections 1.8.2.1, above.

2.9 Aroclor Data **<B-1>**

2.9.1 Sample Data

Sample data shall be reported on individual "Organic Analysis Data Sheet(s)" (FORM I ARO). These forms shall be arranged in order of increasing NYSDEC sample number, considering both numbers and letters. For a detailed explanation of the Aroclor FORM I requirements, see Sections 1.9.2.1, above.

2.10 GC Organic Data (Includes all Organic data generated using a GC or GC-type instrument that does not fit into any of the categories listed in Sections 2.6-2.9.) **<B-1>**

2.10.1 Sample Data

Sample data should be reported using modified versions of the "FORM I" used in the above organic categories. Questions regarding the modification of the FORM I's for this data should be directed to the NYSDEC Quality Standards and Analytical Management Section. See also Section 3.10 for further explanation.

2.11 Inorganic Data **<B-1>**

2.11.1 Sample Data

Sample data shall be submitted with the "Inorganic Analysis Data Reporting Forms" (FORM IA-IN and FORM IB-IN) for all samples in the SDG, arranged in increasing alphanumeric NYSDEC sample number order. For a detailed explanation of the Inorganic FORM I requirements, see Sections 1.10.2, above.

2.12 Toxicity Characteristic Leaching Procedure (TCLP) Data **<B-1>**

2.12.1 Sample Data (**<B-2>** the beginning of data for each unique analysis fraction)

Sample data shall be submitted on modified reporting forms based on the reporting forms used in Sections 2.6-2.11. The analysis specific FORM I's should be modified to include the following TCLP specific information, either in the footer or the header of the form:

- Matrix of Original Sample
- % Solid content of the sample, if the sample was a filterable liquid please fill this field with "<0.5%".
- Start date/time of TCLP extraction

- End date/time of TCLP extraction
- Start Temperature of TCLP extraction room
- End Temperature of TCLP extraction room.
- TCLP Fluid used (#1 or #2)
- Sample pH
- Ending extract pH

3.0 ASP Category B

3.1 Cover Documentation **<B-1>** - See Requirements listed in Section 1.1 above.

3.2 SDG Narrative **<B-1>** - See Requirements listed in Section 1.2 above.

3.3 Contract Lab Sample Information Sheets **<B-1>** - See Requirements listed in Section 1.4 above.

3.4 Chain-of-Custody Forms **<B-1>** - See Requirements listed in Section 1.5 above.

3.5 NYSDEC Data Package Summary Forms **<B-1>** - Requirements and Instructions for these forms are listed in Section IV of this Exhibit.

3.6 GC/MS Volatiles Data **<B-1>**

3.6.1 Volatiles QC Summary **<B-2>**

3.6.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.6.1.1.

3.6.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.6.1.2.

3.6.1.3 QC Check Sample/Standard – (If Applicable) Reported on a modified version of FORM I VOA-1, VOA-2. The form should be modified in such a way that the header clearly states that the results being reported are from a "QC Check Sample/Standard".

3.6.1.4 Method Blank Summary – See requirements listed in Section 1.6.1.3.

3.6.1.5 GC/MS Instrument Performance Check – See requirements listed in Section 1.6.1.4.

3.6.1.6 Internal Standard Area and RT Summary – See requirements listed in Section 1.6.1.5.

3.6.1.7 Instrument Detection Limits – Reported on a modified version of FORM I VOA-1, VOA-2. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the FORM I's should not be used.

3.6.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.6.2.

3.6.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.6.3.

3.6.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.6.4. In addition to the requirements listed in Section 1.6.4, the raw data for "QC Check Sample/Standard" should be reported following the raw data for "Matrix Spike Duplicate Data" as follows:

3.6.4.1 QC Check Sample/Standard **<B-3>**

3.6.4.1.1 Tabulated results (FORM I-VOA) of <u>all</u> target compounds. FORM I-VOA-TIC is <u>not</u> required.

3.6.4.1.2 Reconstructed ion chromatograms(s) and quantitation reports(s) or legible (GC/MS), labeled as in Section 1.6.2.4. Spectra are <u>not</u> required.

3.6.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.6.5.

3.6.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.6.6.

3.7 GC/MS Semivolatiles Data **<B-1>**

3.7.1 QC Summary **<B-2>**

3.7.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.7.1.1.

3.7.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.7.1.2.

3.7.1.3 QC Check Sample/Standard – (If Applicable) Reported on a modified version of FORM I SV-1, SV-2. The form should be modified in such a way that the header clearly states that the results being reported are from a QC Check Sample/Standard.

3.7.1.4 Method Blank Summary – See requirements listed in Section 1.7.1.3.

3.7.1.5 GC/MS Instrument Performance Check – See requirements listed in Section 1.7.1.4.

3.7.1.6 Internal Standard Area and RT Summary – See requirements listed in Section 1.7.1.5.

3.7.1.7 Instrument Detection Limits – Reported on a modified version of FORM I SV-1, SV-2. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the Form Is should not be used.

3.7.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.7.2. In addition to all the requirements listed under Section 1.7.2, any GPC Chromatograms produced during the analysis of the samples should be included at the end of Section 3.7.2.

3.7.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.7.3. In addition to all the requirements listed under Section 1.7.3, data for "Semivolatile GPC Calibration Data" should be listed as follows:

3.7.3.1 Semivolatile GPC Calibration Data – UV detector traces showing peaks that correspond to the compounds in the semivolatile GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak. Do not include FORM IX Pest-2, as the compounds used on that form

are not appropriate for semivolatile sample extracts. **<B-3>**

3.7.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.7.4. In addition to the requirements listed in Section 1.7.4, the following should be added directly after the raw data for "Matrix Spike Duplicate Data" but before the GPC Raw QC data:

3.7.4.1 QC Check Sample/Standard **<B-3>**

3.7.4.1.1 Tabulated results (FORM I-SV) of all target compounds. FORM I-SV-TIC is not required.

3.7.4.1.2 Reconstructed ion chromatograms(s) and quantitation reports(s) or legible (GC/MS), labeled as in Section 1.7.2.4. Spectra are not required.

3.7.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.7.5.

3.7.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.7.6.

3.8 GC/ECD and GC/MS Pesticide Data **<B-1>**

3.8.1 QC Summary **<B-2>**

3.8.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.8.1.1.

3.8.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.8.1.2.

3.8.1.3 Laboratory Control Sample Recovery – See requirements listed in Section 1.8.1.3.

3.8.1.4 QC Check Sample/Standard – (If Applicable) Reported on a modified version of FORM I PEST-1. The form should be modified in such a way that the header clearly states that the results being reported are from a QC Check Sample/Standard.

3.8.1.5 Method Blank Summary – See requirements listed in Section 1.8.1.4.

3.8.1.6 GC/MS Instrument Performance Check – (if Applicable) No Form exists for this requirement. A Narrative statement should be included for GC/MS pesticide data. The narrative should document the following.

- Frequency at which instrument performance checks were performed. Include the date and time the check was run and the sample runs (file IDs) associated with the check.
- The results of the Instrument Performance Check (Pass or Fail).
- The criteria used to evaluate the acceptance of the check.

3.8.1.7 Instrument Detection Limits – Reported on a modified version of FORM I PEST-1. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the Form Is should not be used.

3.8.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.8.2, up to and including Section 1.8.2.5 (omit 1.8.2.6). In addition to all the requirements listed under Section 1.8.2, please include the following:

3.8.2.1 UV traces from GPC (if GPC performed).

3.8.2.2 If pesticides are confirmed by GC/MS or run solely via GC/MS, the Laboratory shall submit copies of reconstructed ion chromatograms, raw spectra and copies of background-subtracted mass spectra of Pesticide target compounds listed in Exhibit C that are identified in the sample and corresponding background-subtracted Superfund-TCL standard mass spectra. Compound names must be clearly marked on all spectra. For multi-component pesticides/Aroclors confirmed by GC/MS, the Laboratory shall submit mass spectra of 3 major peaks of multi-component compounds from samples and standards.

3.8.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.8.3. For the purposes of NYSDEC ASP Category B reporting the requirements of Section 1.8.3.4-7 may be omitted. In addition to the requirements of Section 1.8.3, please include the following:

3.8.3.1 Pesticide GPC Calibration Data – UV detector traces showing peaks that correspond to the compounds in the pesticide GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak. **<B-3>**

3.8.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.8.4. In addition to the requirements listed in Section 1.8.4, the following should be added directly after the raw data for "Matrix Spike Duplicate Data":

3.8.4.1 QC Check Sample/Standard **<B-3>**

3.8.4.1.1 Tabulated results (FORM I-PEST) of all target compounds.

3.8.4.1.2 Chromatogram(s) and data system printout(s) (GC), as labeled in Section 1.8.2.2.

3.8.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.8.5.

3.8.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.8.6.

3.9 GC/ECD and GC/MS Aroclor Data **<B-1>**

3.9.1 QC Summary **<B-2>**

3.9.1.1 System Monitoring Compound Summary – See requirements listed in Section 1.9.1.1.

3.9.1.2 Matrix Spike/Matrix Spike Duplicate Summary – See requirements listed in Section 1.9.1.2.

3.9.1.3 Laboratory Control Sample Recovery – See requirements listed in Section 1.9.1.3.

3.9.1.4 QC Check Sample/Standard – (If applicable) Reported on a modified version of FORM I ARO. The form should be modified in such a way that the header clearly states that the results being reported are from a QC Check Sample/Standard.

3.9.1.5 Method Blank Summary – See requirements listed in Section 1.9.1.4.

3.9.1.6 GC/MS Instrument Performance Check – (if Applicable) No Form exists for this requirement. A Narrative statement should be included for GC/MS Aroclor data. The narrative should document the following.

- Frequency at which instrument performance checks were performed. Include the date and time the check was run and the sample runs (file IDs) associated with the check.
- The results of the Instrument Performance Check (Pass or Fail)
- The criteria used to evaluate the acceptance of the check.

3.9.1.7 Instrument Detection Limits – Reported on a modified version of FORM I ARO. The form should be modified in such a way that the header clearly states that the results being reported are the statistically determined detection limits for a given instrument using a given method. Detection limits should be determined annually. The "Q" column on the Form Is should not be used.

3.9.2 Sample Data **<B-2>**

Sample Data should be reported in the same format and order as detailed in Section 1.9.2. In addition to all the requirements listed under Section 1.9.2, please include the following:

3.9.2.1 UV traces from GPC (if GPC performed).

3.9.2.2 If pesticides are confirmed by GC/MS or run solely via GC/MS, the Laboratory shall submit copies of reconstructed ion chromatograms, raw spectra and copies of background-subtracted mass spectra of Pesticide target compounds listed in Exhibit C that are identified in the sample and corresponding background-subtracted Superfund-TCL standard mass spectra. Compound names must be clearly marked on all spectra. For multi-component pesticides/Aroclors confirmed by GC/MS, the Laboratory shall submit mass spectra of 3 major peaks of multi-component compounds from samples and standards.

3.9.3 Standards Data **<B-2>**

Standard Data should be reported in the same format and order as detailed in Section 1.9.3. In addition to the requirements of Section 1.9.3, please include the following:

3.9.3.1 Pesticide GPC Calibration Data – UV detector traces showing peaks that correspond to the compounds in the pesticide GPC calibration mixture. Traces must be labeled with GPC column identifier, date of calibration, and with compound names labeled either directly out from the peak, or on a printout of retention times, if retention times are printed over the peak. **<B-3>**

3.9.4 Raw QC Data **<B-2>**

Raw QC Data should be reported in the same format and order as detailed in Section 1.9.4. In addition to the requirements listed in Section 1.9.4, the following should be added directly after the raw data for "Matrix Spike Duplicate Data":

3.9.4.1 QC Check Sample/Standard **<B-3>**

3.9.4.1.1 Tabulated results (FORM I ARO) of all target compounds.

3.9.4.1.2 Chromatogram(s) and data system printout(s) (GC), as labeled in Section 1.9.2.2.

3.9.5 Copy of Calculations **<B-2>**

Please provide copies of calculations as specified in Section 1.9.5.

3.9.6 Copy of Extraction Logs **<B-2>**

Please provide copies of extraction logs as specified in Section 1.9.6.

3.10 GC Organic Data **<B-1>**

On occasion NYSDEC may require samples to be analyzed by various GC methods for organic analytes. The reporting of these analytes represents a challenge because no EPA CLP forms exist to report this data. Since most environmental reporting software packages are very rigid in their output formats, it is prohibitive for NYSDEC to develop specialized reporting forms for GC organic data. NYSDEC recognizes that some software venders have created "CLP-like" reporting for GC organic data, and when feasible NYSDEC recommends the use of such software for this data. If such software is not available or unobtainable to the laboratory, the laboratory should modify and use the reporting formats and reports specified in Sections 1.6, 1.7, 1.8, and 1.9. The order of the reporting elements should be unaltered from the original Section being modified. If the reporting software package allows, the identifier for the Forms should be changed to "GC" (i.e. FORM I GC, FORM II GC, etc.). The basic structure of this reporting section should be as follows:

3.10.1 QC Summary **<B-2>**

3.10.1.1 Surrogate/System Monitoring Compounds Recovery Reports (FORM II GC)

3.10.1.2 Matrix Spike/Matrix Spike Duplicate Summary (FORM III GC)

3.10.1.3 QC Check Sample/Standard (FORM I GC + Raw Data)

3.10.1.4 Method Blank Summary (FORM IV GC)

3.10.1.5 Instrument Detection Limits (Performed annually)

3.10.2 Sample Data **<B-2>**

3.10.2.1 Results and raw data for each individual sample should be assembled in packets as follows, and placed in order according to NYSDEC Sample ID, from lowest to highest:

3.10.2.1.1 Target Compound Results (FORM I GC)

3.10.2.1.2 Manual calculation worksheets, if applicable,

3.10.2.1.3 Appropriate raw instrument data**,**

3.10.2.1.4 GPC chromatograms or other qualitative sample specific clean-up data, if applicable.

3.10.3 Standards Data **<B-2>**

3.10.3.1 Initial Calibration Data

3.10.3.2 Continuing Calibration Data

3.10.3.3 Standard chromatograms and data system printouts for all standards.

3.10.4 Copy of Calculations **<B-2>**

3.10.5 Copy of Extraction Logs **<B-2>**

3.11 Inorganic Data **<B-1>**

Sample data shall be submitted with the Inorganic Analysis Data Reporting Forms for all samples in the SDG, arranged in increasing alphanumeric DEC sample number order, followed by the QC analysis data, Quarterly Verification of Instrument Parameter forms, raw data, and copies of the digestion and distillation logs.

3.11.1 Results – Should be reported on FORM IA-IN and FORM IB-IN, and reported according to the specifications in Section 1.10.1. **<B-2>**

3.11.2 Quality Control Data – Should be reported and ordered per the specifications listed above in Section 1.10.2. Verification of Instrument Parameters should also be reported in this Section. Frequency of verifications is unmodified from the CLP requirements. **<B-2>**

3.11.3 Raw Data – Should be reported and ordered per the specifications listed above in Section 1.10.3. **<B-2>**

3.11.4 Digestion and Prep Logs – Should be reported and ordered per the specifications listed above in Section 1.10.4. **<B-2>**

3.12 Wet Chemistry Data **<B-1>**

On occasion NYSDEC may require samples to be analyzed by wet chemistry methods for "conventional" analytes. The reporting of these analytes represents a challenge because no EPA CLP forms exist to report such data. Since most environmental reporting software packages are very rigid in their output formats, it is prohibitive for NYSDEC to develop specialized reporting forms for wet chemistry analysis data. NYSDEC recognizes that some software venders have created "CLP-like" reporting for wet chemistry parameters, and when feasible NYSDEC recommends the use of such software for this data. If such software is not available or unobtainable to the laboratory, the laboratory should modify and use the reporting formats and reports specified in Sections 1.10 (Inorganics). The order of the reporting elements should be unaltered from the original Section being modified. If the reporting software package allows, the identifier for the Forms should be changed to "WC" (i.e. FORM I-WC, FORM II-WC, etc.). The basic structure of this reporting section should be as follows:

3.12.1 Results – Modified Inorganic Analysis Data Sheet **<B-2>**

Tabulated analytical results (identification and quantitation) of the specified analytes (Exhibit C) must be accompanied by a specific, signed statement in the SDG Narrative, which authorizes the validation and release of analytical results (Section 1.2). If the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

Appropriate concentration units must be specified and entered on FORM I-WC. The quantitative values shall be reported in units of micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. Units may be adjusted in order to make excessively large or small concentration numbers more manageable. Results for solid samples must be reported on a dry weight basis. Analytical results must be reported to two significant figures if the result value is less than 10; to three significant figures if the value is greater than or equal to 10. Results for percent solids must be

reported to one decimal place. Data qualifiers should be added according to Table 2.

3.12.2 Quality Control Data – include each only when applicable to the parameter being analyzed. **<B-2>**

3.12.2.1 Initial and Continuing Calibration Verification

3.12.2.2 CRQL Standard for Wet-Chemistry Analysis

3.12.2.3 Blanks

3.12.2.4 Spike Sample Recovery

3.12.2.5 Post Digest Spike Sample Recovery

3.12.2.6 Duplicates

3.12.2.7 Laboratory Control Sample

3.12.2.8 Holding Times

3.12.3 Raw Data **<B-2>**

For each reported value, the Laboratory shall include in the data package all raw data from the instrument used to obtain that value and the QA/QC values reported (except for raw data for Verifications of Instrument Parameters). Raw data must contain all instrument readouts used for the sample results, including those readouts that may fall below the IDG. ALL instruments must provide a legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct sequential instrument readout must be included. A hardcopy of the direct instrument readout for cyanide must be included if the instrumentation has the capability. All raw data shall include absorbance values with concentration units (unless instrument direct readout is in concentration units). A photocopy of manual worksheets used must be included for all non-instrumental parameters. Raw data must be labeled with NYSDEC sample number or be associated to a group of NYSDEC sample numbers for the following:

3.12.3.1 Calibration standards, including source and prep date.

3.12.3.2 Initial and continuing calibration blanks and preparation blanks.

3.12.3.3 Initial and continuing calibration verification standards.

3.12.3.4 Diluted and undiluted samples (by NYSDEC sample number) and all weights, dilutions and volumes used to obtain the reported values. (If the volumes, weights, and dilutions are consistent for all samples in a given

SDG, a general statement outlining these parameters is sufficient).

3.12.3.5 Duplicates.

3.12.3.6 Spikes (indicating standard solutions used, final spike concentrations, volumes involved). If spike information (source, concentration, volume) is consistent for a given SDG, a general statement outlining these parameters is sufficient.

3.12.3.7 Instrument used, any instrument adjustments, data corrections, or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation.

3.12.3.8 Time and date of each analysis. Instrument run logs can be submitted if they contain this information. If the instrument does not automatically provide times of analysis, these must be manually entered on all raw data for initial and continuing calibration verification and blanks, as well as, interference check samples and linear range analysis.

3.12.4 Digestion and Distillation Logs **<B-2>**

These logs must include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e., laboratory control sample, preparation blank) correspond to each batch digested, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) indication of pH <2 or >12, as applicable.

3.13 Toxicity Characteristic Leaching Procedure (TCLP) Data **<B-1>**

Sample data shall be submitted with the Toxicity Characteristic Leaching Procedure Analysis Data Reporting Forms for all samples in the SDG, arranged in packets by analysis fraction. The packets shall consist of the sample results in increasing alphanumeric DEC sample number order, followed by the QC analyses data, Verification of Instrument Parameters forms, raw data, and copies of the digestion and distillation logs pertaining to that analysis fraction. The logbook page or pages dedicated to the TCLP extraction procedure should be included at the end of all the packets for the applicable analysis fractions.

Neither NYSDEC nor EPA CLP have created specific forms for reporting the results of TCLP extracted analytes. Due to the lack of any standardized forms for this data, it is unlikely that any commercial software would be or will be available to report TCLP analysis data. NYSDEC requests that the laboratory report TCLP analysis results on the analogous FORM *X* reports for each analysis and/or QC procedure

performed on the TCLP extraction fluid. The only modification to the traditional CLP-type Forms specified for use in the NYSDEC ASP is that these forms clearly be marked either in the header or in the footer comments that the results being reported on the form are from the analysis of a TCLP extract. If feasible the codes for the forms should be modified and a final suffix of "-TCLP" should be added. For example a "FORM 1 VOA-1" reported for the analysis of a TCLP extract would be "FORM 1 VOA-1-TCLP".

Note: *Data for every separate analysis performed on a TCLP extract should be separated and marked with a second level bookmark* ***(<B-2>)****.*

3.13.1 Results – Toxicity Characteristic Leaching Procedure (TCLP) Analysis Data Sheet (TCLP Modified FORM Is) **<B-3>**

Tabulated analytical results (identification and quantitation) of the specified analytes (Exhibit C) must be accompanied by a specific, signed statement in the SDG Narrative, which authorizes the validation and release of analytical results (Section 3.1). If the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

Appropriate concentration units must be specified and entered on TCLP Modified FORM Is. The quantitative values shall be reported in units of milligrams per liter (mg/L). No other units are acceptable. Analytical results must be reported to two significant figures if the result value is less than 10; to three significant figures if the value is greater than or equal to 10. Results for percent solids must be reported to one decimal place. Qualifiers are to be added according to Table 1 and Table 2.

3.13.1.1 Organic Data Results – Should be reported in order by NYSDEC Sample ID, with the raw data and TIC's (if applicable) directly following the modified FORM I from the sample. See specifications in Sections 1.6.2, 1.7.2, 1.8.2, and 1.9.2 for instructions of reporting sample result for TCLP Organics

3.13.1.2 Inorganic Data Results – Should be reported according to the specifications listed in Section 1.10.1. Raw data will not be assembled directly after the sample data, but included later in Section 3.14.4.

3.13.2 TCLP Quality Control Data – quality control reporting should be accomplished in a manner similar to that used to report sample data on the modified FORM I's above. The key features of all CLP or CLP-like reporting forms should be retained, while notation should be added to denote that the results being reported are from the analysis of a TCLP extract. **<B-3>**

3.13.2.1 Organic Analysis of TCLP Extracts

3.13.2.1.1 Report all QC data according to the specifications listed in Sections 1.6.1, 1.7.1, 1.8.1, and 1.9.1.

3.13.2.2 Inorganic analysis of TCLP Extracts

3.13.2.2.1 Report all QC data according to the specifications listed in Section 1.10.2.

3.13.3 Verification of Instrument Parameters **<B-3>**

3.13.3.1 Organic Analysis of TCLP Extracts – Not required to be included in data package.

3.13.3.2 Inorganic analysis of TCLP Extracts – Data pertaining to the verification of inorganic instrument parameters relative to TCLP extract analysis should be reported according to the specifications in Section 1.10.3.

Note: *Copies of Verification of Instrument Parameters forms for the current quarter must be submitted with each data package.*

3.13.4 Raw Data **<B-3>**

3.13.4.1 Organic Raw Data – Raw data supporting sample results should be included in Section 3.13.1.

3.13.4.1.1 Standards Data – This section should include the raw data for calibration and calibration verifications run to support the analysis of the TCLP extract. See Sections 1.6.3, 1.7.3, 1.8.3, and 1.9.3 for instructions and specifications.

3.13.4.1.2 Raw QC Data – This section should include the raw data need to support the QC results reported in Section 3.13.2.1. The data should be presented and arranged according to the specifications in Sections 1.6.5, 1.7.5, 1.8.5, and 1.9.5.

3.13.4.2 Inorganic Raw Data – Raw data supporting the results reported in Section 3.13.1 and Section 3.13.2 should be included in this section. The raw data should follow the order and format specified in section 1.10.3.

3.13.5 Prep/Digestion Logs (Analysis Specific) – Directly following the Forms and raw data for a fraction packet, all applicable preparation and digestion logs should be included that are relevant to that analysis fraction. **<B-3>**

3.13.6 Prep Logs (TCLP Specific) – A report or copy of the logbook for the TCLP extraction process is required. If multiple TCLP extraction batches were performed within the SDG, a report or logbook page per TCLP batch is required. This report should include the following information: **<B-2>**

- NYSDEC Sample IDs
- Laboratory Sample IDs
- Sample Matrix
- % Total Solids for Sample
- Extract Filterable or Non-filterable
- Average Particle Size in Sample
    - Was Sample Particle Size Reduced?
- Data on Extraction Fluid Determination
    - Initial pH of Sample
    - pH of Sample after Addition of Acid
    - Extraction Fluid Used (Type 1 or Type 2)
- Data on the Extraction Fluid
    - Extraction Fluid Type
    - Extraction Fluid Batch ID
    - Initial pH of Fluid
- Amount (grams) of Sample Extracted
- TCLP Extraction Start Date and Time
- Temperature of TCLP Extraction Room at Start Time
- TCLP Extraction End Date and Time
- Temperature of TCLP Extraction Room at End Time
- pH of TCLP Extract at End Time

F. – Data In Computer Readable Form

Exhibit H details the requirements for electronic data deliverables (EDDs) and any other sample data submissions required to comply with NYSDEC database requirements.

For the purposes of this Protocol, and specifically Exhibit H, Sample Data Packages and Sample Summary Data Packages in the form of .PDF files are not considered “Data In

Computer Readable Form". Requirements for .PDF files are given in this Exhibit, under Section V.

G. – Electronic Instrument Data

The Laboratory must archive all raw and processed instrument data on portable electronic storage media, in the format specified by the instrument manufacturer. Portable electronic storage media can be any of the following: magnetic tapes, CD-ROM, DVD-ROM, DAT, ZIP Disks, or any other portable storage media meeting the following requirements: must be "locked, read only" after the initial "write" to the media, stable over time, easily stored on site. Data may be archived to a non-portable media such as an auxiliary hard drive, but the capability must exist to extract data upon request from NYSDEC. Data archived to an auxiliary hard drive must meet the following criteria: (a) the capability must exist to migrate the files back into the instruments data system in order to generate/regenerate appropriate analysis data and (b) the capability must exist to transfer archived files to portable storage media in order to ship the raw data to NYSDEC. This storage media must contain all instrument files used directly or indirectly to construct the NYSDEC Sample Data Packages. NYSDEC related instrument files do not need to be archived separately if the lab uses an all-inclusive archive technique for instrument data. Output files subject to this archive requirement include, but are not limited to, samples, blanks, spikes, matrix spikes, matrix spike duplicates, calibration standards, continuing calibrations, instrument tunes, as well as all laboratory-generated spectral libraries and quantitation reports required to generate the data package. The Laboratory shall maintain a written reference logbook of stored files to NYSDEC sample number, calibration data, standards, blanks, matrix spikes, and matrix spike duplicates. The logbook should include NYSDEC sample numbers and standard and blank ID's, identified by Case and Sample Delivery Group.

The Laboratory is required to retain the stored files for 3 years after data submission. During that time, the Laboratory shall submit copies of archived files and associated logbook pages within seven days after receipt of a written request from the Bureau of Watershed Assessment and Management.

H. – Samples and Extracts

1.0 Unused and Excess Sample Amounts

After the required sample aliquot has been successfully analyzed and reported, the Laboratory shall preserve any unused and excess sample amounts at the required storage temperature and conditions as specified in Exhibit I. Samples should be stored in their original containers, clearly lableled with their NYSDEC Sample Numbers and associated Case and SDG numbers. The Laboratory is required to retain samples for 365 days following data submission. During that time, the Laboratory shall submit samples and associated custody documents within seven days following receipt of a written request from the Bureau Watershed Assessment and Management or the Project Officer.

2.0 Sample Extracts (Organincs only)

The Laboratory shall preserve sample extracts at a temperature less than 4°C in bottles/vials with Teflon-lined septa. Extract bottles/vials shall be labeled with

NYSDEC sample number, Case number, and Sample Delivery Group (SDG) number. The Contractor shall maintain a logbook of stored extracts, listing NYSDEC Sample Numbers and associated Case and SDG numbers. The Laboratory is required to retain extracts for 365 days following data submission. During that time, the Laboratory shall submit extracts and associated logbook pages within seven days following receipt of a written request from the Bureau Watershed Assessment and Management or the Project Officer.

I. – Verification of Instrument Parameters

1.0 Organic Verifications

The contractor shall perform and report annual verification of MDLs by the technique specified in 40 CFR Part 136 using the analytical methods specified in Exhibit D (by type, matrix, and model for each instrument used on the contract) to the Bureau of Watershed Assessment and Management. All the MDLs shall meet the CRQLs specified in Exhibit C.

2.0 Inorganic Verifications

The Laboratory shall perform verification of instrument detection limits, method detection limits, correction factors, and linear ranges for those instrument-types specified in Exhibit E. The methods and frequency for such verifications are detailed in Exhibit E. For the ICP instrumentation and methods, the Laboratory shall also report annually interelement correction factors (including method of determination), wavelengths used, and integration times. Verification of Instrument Parameters forms for the current period shall be submitted in each Sample Delivery Group data package, using Forms X, XI, and XII. Submission of Full Verification of Instrument Parameters shall include the raw data used to determine those values reported.

3.0 All Analyses

Method Detection Limit (MDL) Study is to be performed at minimum annually, or for each new instrument brought into service, whichever is more frequent. Some analyses and methods may require more frequent running of the MDL study. If a method requires more frequent running of the MDL study, that requirement supercedes the annual requirement set herein. The information on current and past MDL studies should be maintained on file at the laboratory. The Laboratory shall maintain records for any and all instrument performance verifications performed for a period of 3 years. During that time, the Laboratory shall submit copies of such records within seven days following receipt of a written request from the Bureau Watershed Assessment and Management or the Project Officer.

J. – Preliminary Results

1.0 Organic Preliminary Results

The FORM I data results shall be submitted for all samples in one SDG of a Case. This includes tabulated target compound results (FORM I *XXXX-X*) for the volatile, semivolatile, pesticide, and Aroclor fractions, and Tentatively Identified

Compounds (FORM I *XXXX*-TIC) for the volatile and semivolatile fractions. The contractor shall clearly identify the Preliminary Results by labeling each FORM I and FORM I TIC as "Preliminary Results" under each form title (e.g., under "Volatile Organics Analysis Data Sheet", "Volatile Organics Analysis Data Sheet Tentatively Identified Compounds").

2.0 Inorganic Preliminary Results

The FORM I IN data results (including all appropriate qualifiers and flags) shall be submitted for all samples in one SDG of a Case. Sample analysis shall follow all requirements stipulated in the Method, Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each FORM I as "Preliminary Results" under the form title (e.g., under "Inorganic Analysis Data Sheet"). The Contractor shall also include a disclaimer in the "Comments" field on all Form Is stating that the "Data results contained on the Form I are for scanning purposes only, and may not have been validated for CLP/ASP criteria." Copies of Sample Traffic Reports/Chain of Custody Records shall be submitted with the Preliminary Results.

3.0 All Preliminary Results (Organic and Inorganic)

Copies of Sample Traffic Reports/Chain of Custody Records shall be submitted with the Preliminary Results. The Contractor shall also submit a Cover Page following the specifications in Exhibit B, Part E, Section 1.1. In addition, the Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The Cover Page shall contain the following statement, (usually included in the SDG Narrative) verbatim: **"I certify that these Preliminary Results are in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as a verified by the following signature."** This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

K. – Results of PE Samples

Results of Performance Evaluation (PE) Samples should be reported similar to a standard environmental sample with deliverables as specified in Items E and F (Sample Data Package (.PDF) and Electronic Data Deliverables (EDD)).

Table 1

List of Organic Method Qualifiers

Qualifier (Q)	Description
B	Entered if the analyte is found in the associated blank as well as the sample.
C	Applied to pesticide results when the identification has been confirmed by GC/MS.
D	Included when the all identified compounds in the analysis are at the secondary dilution factor.
E	Identified compounds whose concentrations exceed the calibration range of the instrument for that specific analysis.
J	Indicates an estimated value, may indicate one of the following, depending on the situation: (1) The reported value is estimated and below the MDL. (2) Used when estimating a concentration for TIC where a 1:1 response is assumed or when the result indicates the presence of a compound that meets the identification criteria, but the results is less than the quantitation limit, but greater than zero. (3) QC associated with this analyte is within warning limits.
N	Included for TIC that indicate presumptive evidence of a compound.
U	Entered if the analyte was analyzed for, but not detected.
P	Used for a pesticide/Aroclor target analyte when the concentration difference between 2 GC columns is greater than 25%; the lower value is flagged with a "P".
EMPC	"Estimated Maximum Possible Concentration" – The amount of analyte cannot be accurately quantified, so a maximum concentration has been estimated for the compound.
"XYZ"	"Wildcard" or Laboratory defined qualifier.

Note: *Form I allows only one character in each qualifier column. If multiple qualifiers are applicable, please assess qualifier priority in the following order: U, E, J, B, D, C, P, N. Reporting done in the EDD may include multiple qualifiers when applicable, separated by a single space.*

Table 2

List of Inorganic Method Qualifiers

Qualifier	Column[1]	Description
Concentration qualifiers		
B	C	Entered if the reported value was less than the CRDL, but greater than the IDL.
U	C	Entered if the analyte was analyzed for, but not detected.
J	C	Entered if the reported value is estimated and below the MDL.
*	C	Duplicate precision exceeds RPD limit.
M	C	Replicate precision exceeds RPD limit.
"XYZ"	C	"Wildcard" or Laboratory defined qualifier.
Qualifier specific entries		
E	Q	Entered if the reported value is estimated because of the presence of interferences.
Method qualifiers		
A	M	Flame atomic absorption
AS	M	Semi-automated spectrophotometric
AV	M	Automated cold vapor atomic absorption
C	M	Manual spectrophotometric
F	M	Furnace atomic absorption
MS	M	Mass spectrometry (ICP-MS)
NR	M	Analyte is not required to be analyzed
P	M	Inductively coupled plasma (ICP)
" "	M	No data have been entered

[1] The term "Column" is used to indicate under which column heading in the reporting forms that the qualifier will be found under.

Note: *Form I allows only one character in each qualifier column. If multiple qualifiers are applicable to column C, please assess qualifier priority in the following order: U, J, B. Reporting done in the EDD may include multiple qualifiers when applicable, separated by a single space.*

PART III -- CLP REPORTING FORMS AND INSTRUCTION GUIDE

1.0 NYSDEC has not created any specific reporting forms for the purpose of ASP reporting. Since most data is now reported using software formatted to produce data in the EPA CLP or EPA CLP-Like Forms, the ASP relies on the forms and instructions specified by the EPA in the CLP. Copies of the CLP SOWs, containing the required Organic and Inorganic Reporting forms and their instructions, can be found in ASP Exhibit D, in the CLP folder.

2.0 The Exhibit B forms and instructions contained in the CLP SOWs can be followed verbatim in most cases. Please note that the following exceptions and modifications to the CLP Forms and Form Instructions should be made.

2.1 Substitutions, General

- All references to "USEPA" or "EPA" should be substituted with "NYSDEC".
- All references to "EPA Sample Number" should be substituted with "NYSDEC Sample Number".
- All references to the "CLP SOW" or "SOW" should be substituted with "NYSDEC ASP" or "ASP", respectively.
- All references to "USEPA Regional Contract Laboratory Program Project Officer (CLP PO)", "USEPA OERR Analytical Operations/Data Quality Center (AOC)" and "Inorganic Program Manager (AOC PM)" should be substituted with "NYSDEC Bureau of Watershed Assessment and Management".
- The "Laboratory Code" to be used on all reporting documents should be the NYSDOH ELAP code assigned to the laboratory.

2.2 All references to the following can be disregarded:

- Non-Routine Analytical Services (NRAS)
- Sample Traffic Reports

2.3 The Forms and Instructions for Organic Data Reporting should follow CLP, Draft SOM01.X, Exhibit B with the following exceptions:

- References to "Modification Reference Number" or "Mod. Ref. Num." can be omitted or ignored in ASP reporting.

2.4 The Forms and Instructions for Inorganic Data Reporting should follow CLP, ILM05.3, Exhibit B, Section 3 with the following exceptions (All Section Numbers refer to directly to the CLP documents):

- The items under Section 3.3.5 may be disregarded.

- The requirement listed in Section 3.4.1.2.1 requiring the entry of the Statement of Work as “ILM05.3” should be modified and the label “ASP2004” should be inserted in the field for the SOW.

- Section 3.6 (CSF Instructions) may be disregarded.

PART IV -- NYSDEC DATA PACKAGE SUMMARY FORMS

The completion of Data Package Summary Forms is no longer a standard requirement for NYSDEC sample data or sample data packages. However for a small portion of NYSDEC Projects, completion of summary forms will be requested and required. These requests will be dependent upon the needs of the data users at NYSDEC. NYSDEC may also request changes in the style and content of the summary forms from those given herein.

The Data Package Summary Forms provided in this Exhibit are similar to the summary forms requested by NYSDEC in the past. If summary forms are requested and no specific template or blank forms have been provided to the laboratory, the following forms should be considered the default format. If custom forms are requested, the laboratory must report the summary data in the format requested. When summary data is requested in a non-standard format, the Laboratory should anticipate that the amount of information required in the summary forms would be similar to the amount of data required to complete the standard summary forms.

Instructions for NYSDEC Data Package Summary Forms

I. Sample Identification and Analytical Requirement Summary (Form S-I)

A. NYSDEC Sample ID/Code

Sample code number or ID assigned to the sample by NYSDEC personnel.

B. Laboratory Sample ID/Code

Code number given to respective sample by the laboratory and used for identification throughout analysis.

C. Analytical Requirements

This column is broken down into 6 sub-columns. The heading of each sub-column is an analytical parameter group. If the sample listed in a row is being analyzed for the parameter group listed at the top of the sub-column, complete the box below with the method number being used to analyze that sample for that parameter group. If no analysis is being performed in that parameter group, the space should be left blank.

II. Sample Preparation and Analysis Summary - Semivolatile (BNA), Volatile (VOA), and Pesticides/PCB's (Form S-IIa/b/c)

A. Laboratory Sample ID

The sample code number that the laboratory will use throughout the analysis for a specific sample.

B. Matrix

Label the sample with matrix indicated as water, soil, oil, grease, or drum solvent, etc.

C. Date Collected

Record the date that sample was collected on site.

D. Date Received at Laboratory

Record the date the Laboratory received the sample. (Validated Time of Sample Receipt - VTSR)

E. Date Extracted

Record the date the sample was extracted. This field should be left blank for aqueous VOA samples.

F. Date Analyzed

Record the date the sample was analyzed.

III. Sample Preparation and Analysis Summary – Miscellaneous Organics (Form S-III)

A. Laboratory Sample ID

The sample code number that the laboratory will use throughout analysis for a specific sample.

B. Matrix

Label the sample with matrix indicated as water, soil, oil, grease, or drum solvent, etc.

C. Analytical Protocol

Record the number of the method used to analyze the sample.

D. Extraction Method

Write the method used for sample extraction.

E. Auxiliary Clean-Up

If cleanup was done on sample, record the method or methods used.

F. Dil/Con Factor

If sample was diluted, record the final (just prior to analysis) dilution factor, or if concentrated, record also.

IV. Sample Preparation and Analysis Summary - Inorganics Analysis

A. Laboratory Sample ID

The sample code number that the laboratory will use throughout analysis for a specific sample.

B. Matrix

Label the sample with matrix indicated as water, soil, oil, grease, or drum solvent, etc.

C. Metals Requested

List metals that are to be analyzed. If for NYSDEC ASP, write full TCL in column, or more individual metals required.

C. Date Received at Laboratory

Record the date the Laboratory received the sample. (Validated Time of Sample Receipt - VTSR).

D. Date Digested

Date the sample was digested or otherwise prepared for analysis.

E. Date Analyzed

Date sample was analyzed on instrument.

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-I

SAMPLE IDENTIFICATION AND ANALYTICAL REQUIREMENT SUMMARY

NYSDEC Sample ID/Code	Laboratory Sample ID/Code	Analytical Requirements					
		VOA GC/MS (Method #)	BNA GC/MS (Method #)	VOA GC (Method #)	Pest PCBs (Method #)	Metals (Method #)	Other (Method #)

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IIa

SAMPLE PREPARATION AND ANALYSIS SUMMARY
SEMIVOLATILE (BNA)
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd at Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IIb

SAMPLE PREPARATION AND ANALYSIS SUMMARY
VOLATILE (VOA)
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd at Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IIc

SAMPLE PREPARATION AND ANALYSIS SUMMARY
PESTICIDE/PCB
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd at Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-III

SAMPLE PREPARATION AND ANALYSIS SUMMARY
MISCELLANEOUS ORGANIC
ANALYSES

Laboratory Sample ID	Matrix	Analytical Protocol	Extraction Method	Auxiliary Cleanup	Dil/Conc Factor

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

FORM S-IV

SAMPLE PREPARATION AND ANALYSIS SUMMARY
INORGANIC ANALYSES

Laboratory Sample ID	Matrix	Metals Requested	Date Rec'd at Lab	Date Digested	Date Analyzed

PART V – NYSDEC ACROBAT DOCUMNENT REQUIREMENTS

1.0 Sample Data Package .PDF File

In order to comply with the Paperless Office requirements being implemented by various New York State government organization, the Department of Environmental Conservation requires that all data packages be submited as Adobe Acrobat .PDF files on a CD-ROM. The following steps must be followed for the submission of Sample Data Packages and other related documents in .PDF format to insure that all data received by NYS DEC can be easily read, understood, and used for Department decision making.

1.1 CD-ROM Requirements

1.1.1 The CD-ROM containing the sample data package must be of the CD-R media type. Use of CD-RW media type is strictly prohibited for the submittal of NYSDEC Sample Data Packages.

1.1.2 The Laboratory is required to produce an additional copy of the Data Package CD-ROM submitted to NYSDEC and retain it for their records, stored for a minimum period of 3 years. This archive copy of the Sample Data Package and accociated SDG submitted files should be stored on CD-R type media. Use of CD-RW media is not permitted.

1.2 Sample Data Package Hardcopy Requirements

1.2.1 Generation of a hardcopy original Sample Data Package for storage at the Laboratory facility is no longer required.

1.2.1.1 Two (2) hardcopies of the SDG Cover Page and SDG Narrative from the Sample Data Package must be generated and signed by the appropriate Laboratory representative. One set of copies must be submitted to NYSDEC with the Sample Data Package CD-ROM. The second set of copies must be kept on file at the laboratory for a minimum period of 3 years from the date of sample receipt.

1.2.2 At the request of NYSDEC the lab should be prepared to generate a hardcopy of the full Sample Data Package, certifiy the newly generated hardcopy with the appropriate signatures, and submit the entire certified Sample Data Package to NYSDEC within 7 business days.

1.2.2.1 The associated computer files required to produce a hardcopy data package should be archived and stored at the laboratory for a minimum of 3 years from the date of sample receipt.

1.3 .PDF File Requirements

Sample Data Packages submitted to NYS DEC in .PDF file format should be of the "Formatted Text and Graphics" .PDF-type. Sample Data Package .PDFs should not be "Image Based" documents. This format allows .PDF documents to be searched for specific text strings within the data package. It also prevents poor integrity of original documents and poor scan qaulity from affecting the overall legibility of the data package.

1.3.1 File to .PDF Conversion – Whenever possible data packages should be constructed from instrument output files and report generator output files converted to .PDF format by processing the files through Adobe Acrobat Writer. When output files are converted into .PDF, the .PDF files created are searchable and the characters/fonts tend to be more legible. Care must be taken to insure that the fonts contained in output files are recognized by Acrobat and are properly converted. Converted files should also be checked to insure formatting (spacing, margins, etc.) and graphics are preserved from the original.

1.3.2 Hardcopy to .PDF Conversion - In some cases output files cannot be used and hard copy data must be scanned to create an image file (non-.PDF) and then converted into .PDF format. In these cases the integrity of the scanned document and the quality of the scan must be closely monitored to insure to overall legibility of the data package. The following requirements should be adhered to when creating .PDF files from hardcopy data.

1.3.2.1 The document should be scanned at 300 dpi or greater.

1.3.2.2 The document should be scanned at a speed slow enough not to distort the fonts or images in the rusultant image file.

1.3.2.3 NYS DEC requires that all scanned image files be processed through the Adobe Acrobat Capture Utility to convert the image file into a Formatted Text and Graphics .PDF. Whenever possible, original hardcopy documents should have no smaller than an 8 pt. font.

Note: *All text of 8 pt. size and greater, orientated along the horizontal axis of the page, should be recognizeable and convertable when processed through ScanSoft OmniPage or a similar Optical Charact Recognition (OCR) software engine. The OCR conversion should produce a .rtf document with an accuracy of 99% or greater when compared to the .PDF original. Text smaller than 8 pt. size or text not oriented along the horizontal axis of the document is not subjuect to the 99% accuracy requirement.*

1.3.3 Cropping of Pages - The pages in the .PDF file should be completely viewable to the reader, with a minimum margin width, on the left, right, top, and bottom of the document, of 0.5 inches when printed on a standard 8.5 by 11 inch piece of paper,. No part of an original image "page" shall be cropped in order to fit the document into a single .PDF "page". If necessary an original document may be proportionally reduced in size by 78%. If a document requires reduction greater than 78% in order to fit on a

single page, the document should be cafefully divided into equally sized parts and a .PDF page created for each part. An 8.5 by 14 inch legal sized document reduced by 78% will fit into a standard page by this requirement.

1.3.4 Page Orientation – Every effort should be made to have pages in the .PDF pages oriented in a consistant manner. NYS DEC prefers all pages to be in the portrait orientation when feasible. If the data system allows for the format of instrument output to be programmed between portrait and landscape, the output should be set to the portrait mode. If landscape is the only output mode possible, or in the case of the NYS Sample Summarry Forms, .PDF pages with landscape orientation should be inserted into the .PDF rotated counter-clockwise 90°. Landscape pages setup with this orientation would be displayed normally after a 90° clockwise rotation by the reader. If, due to the unprogrammable format of instrument data systems or report generation software, the majority of the pages are converted into .PDF in landscape orientation, they may remain in landscape orientation. If landscape is the majority orientation of the pages, portrait pages should be rotated counter-clockwise 90°, so that a clockwise rotation of 90° by the reader will orientate the image properly.

1.3.5 Linked Table of Contents – NYS DEC requuires that all Sample Data Packages include a Table of Content. The Table of Contents in the .PDF file should provide clickable links to the various sections and sub-sections listed in the Table.

1.3.6 Bookmarks – The Sample Dat Package shall contain bookmarks within the Adobe Acrobat file, arranged in the following manner:

1.3.6.1 The Sample Data Package .PDF should contain bookmarks to separate individual sections and the subsections within. All sections and subsections requiring bookmarks are marked in this Exhibit with a "**<B-*X*>**".

1.3.6.1.1 Sections marked with "**<B-1>**" should be bookmarked with a level one bookmark. Level one is the hightest level of bookmarking in the data package.

1.3.6.1.2 Sections marked with "**<B-2>**" should be bookmarked with a level two bookmark. Level two bookmarks are sub-bookmarkes to the parent level one bookmarks.

1.3.6.1.3 Sections marked with "**<B-3>**" should be bookmarked with a level three bookmark. Level three bookmarks are sub-bookmarkes to the parent level two bookmarks.

1.3.6.2 All items listed in the table of contents should be bookmarked within the .PDF and accessable from the bookmark navigation panel in Acrobat Reader.

1.3.6.3 Sample Data Packages should be further bookmarked when either one of the following conditions are met.

1.3.6.3.1 In cases when sample data exceeds more than 5 pages per sample data "packet", in either a "Sample Results" Section or a "Raw Data" Section, the beginning of each data "packet" must be bookmarked with the appropriate level bookmark **<B-(*X*+1)>**. Where *X* is the level of the parent bookmark for the Section in which the data is being placed in.

1.3.6.3.2 In cases when the total amount of data in any of the Sample Data Package sections designated for either a "Sample Results" or "Raw Data" exceeds 40 pages, the beginning of each data "packet" must be bookmarked with the appropriate level bookmark **<B-(*X*+1)>**. Where *X* is the level of the parent bookmark for the Section in which the data is being placed in.