U.S. NAVY ENGINEERING FIELD ACTIVITY - NORTHEAST
REMEDIAL ACTION CONTRACT (RAC)
CONTRACT NO. N62472-99-D-0032
CONTRACT TASK ORDER NO. 069

SAMPLING PLAN FOR THE CHARACTERIZATION OF PCB CONTAMINATED SOILS AND CONCRETE GOULD ISLAND NAVAL STATION NEWPORT MIDDLETOWN, RHODE ISLAND

Issued:
November 21, 2001

Prepared for:
ENGINEERING FIELD ACTIVITY - NORTHEAST
NAVAL FACILITIES ENGINEERING COMMAND
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1	9/14/01	S. Leach, P.E.	R. Woodworth	All
2	11/21/01	S. Leach, P.E.	R. Woodworth	All

Prepare in quintuplicate (original and 4 copies)

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CONTRACT NO.	CONTRACT TASK ORDER NO.	ACTIVITY LOCATION
N62472-99-D-0032	**0069**	**Gould Island – Newport, RI**

PROJECT TITLE:
PCB Characterization and Removal – Gould Island

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Foster Wheeler Environmental Corp. - Program QCM: Thomas Kelly	November 21, 2001
TO:	DATE
C. Davis (E-Copy)	November 21, 2001

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ITEM NO.	SUBMITTAL DESCRIPTION	PREPARED/ SUBMITTED BY	APPROVED	DISAPPROVED	REMARKS
1	SD-08, Statements; Draft Sampling Plan for the Characterization of PCB Contaminated Soils and Concrete – Gould Island (R2)	Thomas Kelly			

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ACRONYMS

ASTM	American Society for Testing and Materials
CFR	Code of Federal Regulations
COC	Chain of Custody
COCs	Constituents of Concern
CRC	Contamination Reduction Corridor
CRZ	Contamination Reduction Zone
CSO	Caretaker Site Office
DOT	Department of Transportation
DQO	Data Quality Objective
EFANE	Engineering Field Activity Northeast
EPA	U.S. Environmental Protection Agency
EZ	Exclusion Zone
HEPA	High Efficiency Particulate Air
LDR	Land Disposal Restriction
MSDS	Material Safety Data Sheet
MS/MSD	Matrix Spike/Matrix Spike Duplicate
OSHA	Occupational Safety and Health Administration
PAL	Project Action Levels
PCBs	Polychlorinated Biphenyls
PPE	Personal Protective Equipment
ppm	parts per million
PQCM	Program Quality Control Manager
PQL	Practical Quantitation Limits
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RAC	Remedial Action Contract
RCRA	Resource Conservation Recovery Act
RIDEM	Rhode Island Department of Environmental Management
ROICC	Resident Officer In Charge of Construction
SHSO	Site Health and Safety Officer
SHSP	Site Health and Safety Plan
SOP	Standard Operating Procedure
SPCC	Spill Prevention Control and Countermeasure
SQCM	Site Quality Control Manager
SVOC	Semivolatile Organic Compound
SZ	Support Zone
TAL	Target Analyte List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TSCA	Toxic Substances Control Act
VOC	Volatile Organic Compound

1.0 INTRODUCTION

Foster Wheeler Environmental Corporation (Foster Wheeler) was contracted by the U.S. Navy Engineering Field Activity Northeast (EFANE) to provide Demolition of Selected Buildings on Gould Island located in Newport, Rhode Island. As part of that contract, Foster Wheeler previously prepared a work plan for the project entitled, "Work Plan for Gould Island Demolition of Selected Buildings - Phases I, II, & III," (the "Work Plan"). In response to the discovery of PBC contamination during building demolition, EFANE issued Contract Task Order No. 0069 under the U.S. Navy Engineering Field Activity Northeast (EFANE) Remedial Action Contract (RAC) N62472-99-D-0032. Under CTO 0069, this sampling plan has been prepared to focus on the characterization of PCB contaminated soil associated with the Transformer Vaults and concrete roadways on the site. The transformer vaults are identified as Buildings 53, 54, 56, 60, 61, and the switch house/transformer vault, Building 59.

The primary objectives of this Sampling Plan are to delineate the areas of PCB contamination within and around the six transformer vaults on-site, to perform in-situ characterization of potential PCB contaminated concrete roadways, and if evidence of PCB contamination of concrete roadways is found, to delineate the extent of PCB contaminated soil below the concrete. Following completion of this sampling effort, the data will be used to develop a work plan for an interim removal action. It is anticipated that implementation of the interim removal will occur in 2002. A proposed schedule for this scope and the interim removal action is provided in Appendix D. Following completion of interim removal actions, a sitewide closure standard will be developed based on a risk evaluation/assessment.

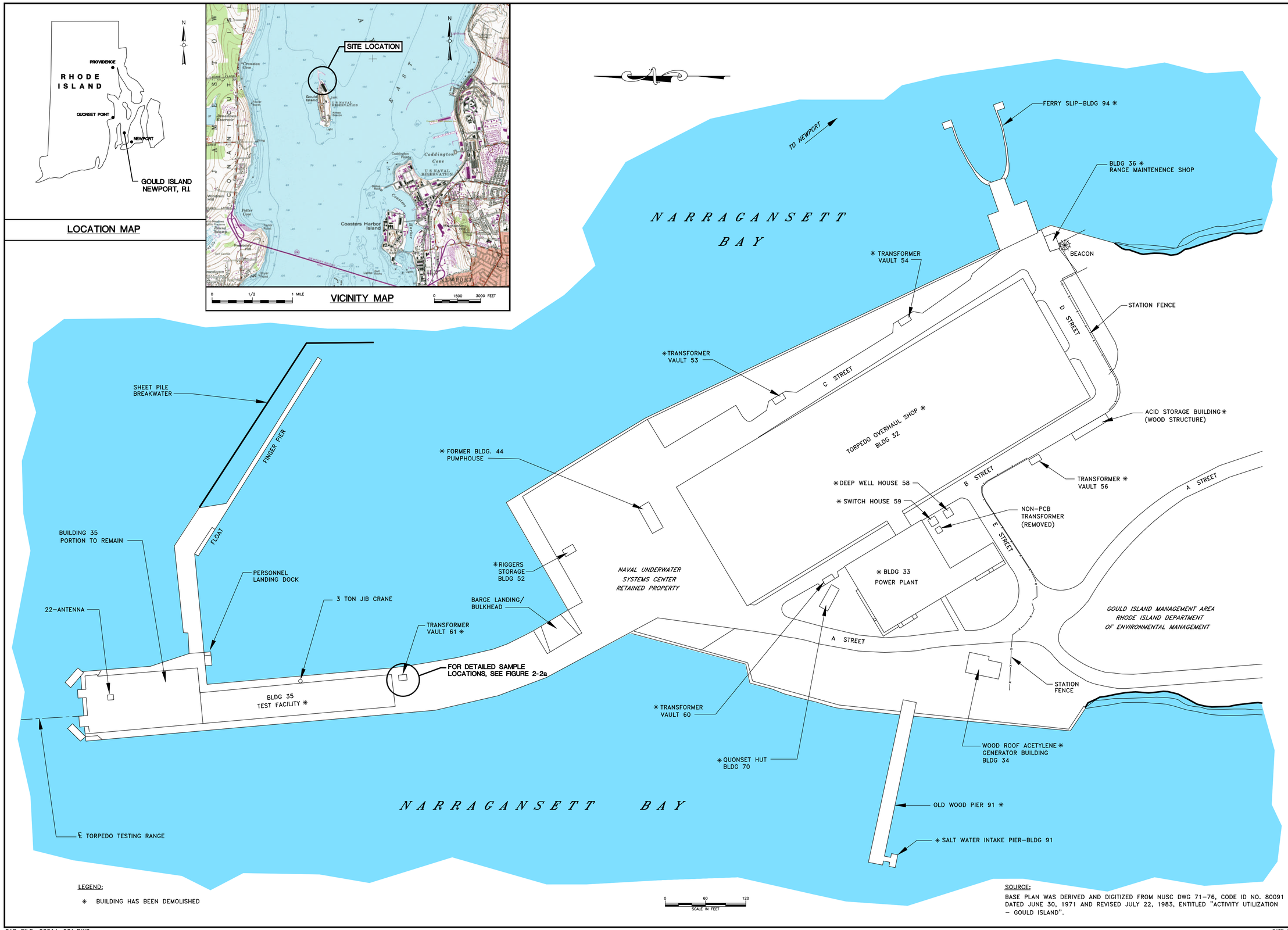
This Sampling Plan presents the proposed implementation of the project. Section 2.0 outlines the project history. Section 3.0 outlines the management structure. Section 4.0 presents the Construction Quality Control Plan. Section 5.0 details the field sampling and laboratory testing programs. Environmental protection and regulatory compliance for this project is presented in Section 6.0. Sections 7.0 and 8.0 discuss the sequencing of the work in the field relating to the sampling activities and transportation and off-site disposal. Reporting documentation is discussed in Section 9.0. All backup documents cited herein have been noted in Section 10.0.

2.0 PROJECT HISTORY

2.1 Project Overview

Gould Island is located in Narragansett Bay approximately 1.5 miles from Naval Station, Newport. As described in the original Work Plan, Foster Wheeler performed asbestos abatement (Section 4.3 of the Work Plan), hazardous waste removal activities (Section 4.4 of the Work Plan), demolition of designated buildings (Section 4.5 of the Work Plan), and removal of designated building slabs and foundations (Section 5.0 of the Work Plan and Work Plan Addendum No. 3) on Gould Island. Foster Wheeler performed this work in three phases. Refer to Figure 2-1 for the Gould Island Site Plan.

Phases I and II included asbestos abatement, hazardous waste removals, and demolition of the buildings to the slab elevation only. Phase I and II were completed in May 2001. In support of the building demolition, Foster Wheeler performed concrete chip sampling of the interior floor and wall surfaces of the transformer vaults and the switch house. Phase III activities included the removal and crushing of selected building slabs and foundations. The results of that sampling indicated the presence of contamination in some of the floor locations. This Sampling Plan is being prepared to assist in



LEGEND:

✳ BUILDING HAS BEEN DEMOLISHED

SOURCE:

BASE PLAN WAS DERIVED AND DIGITIZED FROM NUSC DWG 71-76, CODE ID NO. 80091 DATED JUNE 30, 1971 AND REVISED JULY 22, 1983, ENTITLED "ACTIVITY UTILIZATION - GOULD ISLAND".

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DEPARTMENT OF THE NAVY
NAVAL FACILITIES ENGINEERING COMMAND
LESTER PENNSYLVANIA

ENGINEERING FIELD ACTIVITY - NORTHEAST

GOULD ISLAND, NEWPORT, RHODE ISLAND

SITE PLAN

EFANE FOR COMMANDER, NAVFAC

APPROVED DATE

SEAL AREA

DATE AUGUST 2001
CODE I.D. NO. 80091
SCALE : AS SHOWN
SPEC. NO. 04-
CONSTRN. CONTR. NO. N62472-99-D-0032
NAVFAC DRAWING NO.
SHEET 1 OF 4
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FIGURE 2-1

delineating the extent of contamination in support of the Phase III activities. Note that the data collected from previous activities is only being utilized to focus on the additional, more detailed investigation proposed in this Sampling Plan. Previously collected data will not be used to confirm that clean-up goals were achieved, or to eliminate areas from further investigation.

2.2 Pre-demolition Sampling Results/Building Demolition

Concrete chip samples were collected from the floor and walls of each Transformer Vault and the Switch House (Transformer side) prior to any demolition activity. Refer to Figure 2-2 for the sampling locations and analytical results. The wall samples indicated that the walls of each vault and the switch house (Transformer side) had PCB levels below the RIDEM Residential Standard of 10 ppm. The highest wall sampling result was 2.9 ppm (Transformer side of Building 59). The samples collected from the floor of the structures indicated levels ranging from 73 ppm (Transformer side of Building 59) to 10,000 ppm (Building 60). Based on these results the vault floors were covered with 10-mil poly sheeting followed by ¾-inch CDX plywood. The poly sheeting and the CDX plywood provided a barrier against contact between the PCB contaminated floor surface and the upper structure during demolition. The roof and walls of each structure have been demolished. Since the concrete debris from the roof and walls contained less than 10 ppm PCBs, it was not regulated as a hazardous material under RIDEM regulations, and was used as fill in the former Building 44 UST excavation.

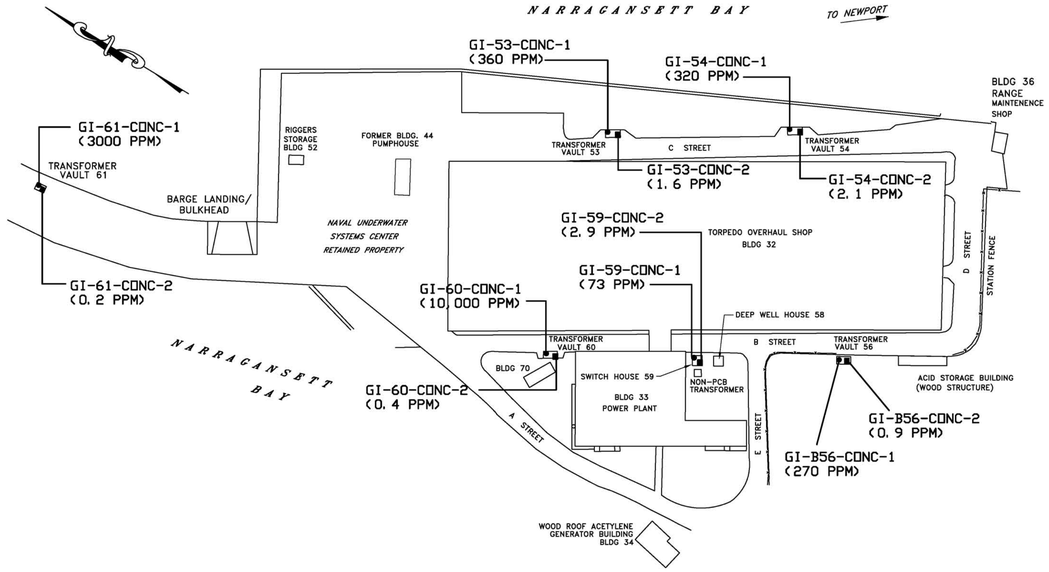
2.3 Transformer Vaults and Switch House Floor Slab/Foundation Demolition Sampling Results

Demolition of the transformer vaults and the switch house floor slab/foundations began on March 27, 2001 with the slab removal of Building 54. During the demolition process it was discovered that each vault has a basement area under the floor slab that consists of four (4) concrete foundation walls and a dirt floor. The basement area of each vault does not have a concrete floor. Buildings 53, 54, 56, 60 & 61 are all constructed in a similar fashion while Building 59 contains two separate basement areas. Buildings 53, 54, 56, 59 (Switch House side) & 60 contained varying amounts of water and, in the case of Building 54, a small quantity of oil as floating product. The floor slabs of Buildings 53, 54, 56, 59 (Transformer side), 60 and 61 have been removed. Building 59 (Transformer side) and 61 basements were dry upon slab removal. Samples were collected from the water in the basement areas of Buildings 53, 56, 59 (Switch House side) & 60, the oil in the Building 54 basement area and the absorbent pads that were used to collect the oil. A summary of the demolition activities and results at each location is provided in the following sections.

On April 12 & 16, 2001 Foster Wheeler collected a series of soil samples, using a 1.5-meter sampling grid, from the perimeter of each Transformer Vault and the Switch House. Where a concrete slab was present, a hole was punched through the slab using the ram hoe to access the soil underneath. Concrete debris was then removed from the hole prior to obtaining a soil sample using a sterile disposable scoop. In areas where no concrete was present, samples were collected from six to twelve inches below the surface. Refer to Figure 2-2 for perimeter sampling locations and analytical results.

2.3.1 Building 53 (Transformer Vault)

Building 53 (Transformer Vault) is located along the eastside of the site just south of the Foster Wheeler trailer. In May 2000, prior to any demolition, a composite sample of concrete dust was collected from 0 to 1 inch below the floor surface. This sample was sent to an off-site laboratory and exhibited levels of

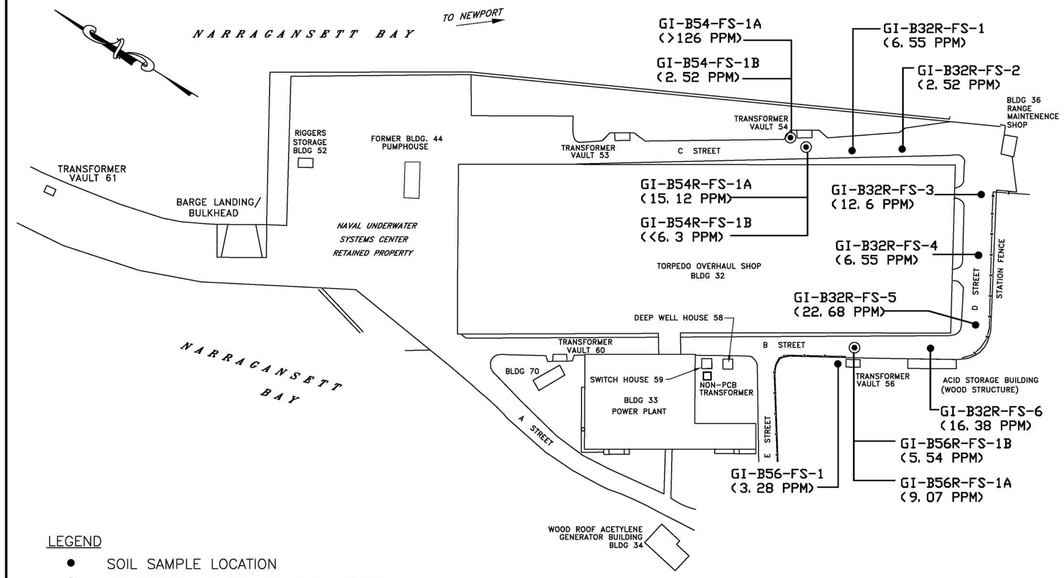


LEGEND

- ● GRAB SAMPLE (FLOOR) (SAMPLING PERFORMED 5/00)
- ■ 4-POINT COMPOSITE WALL SAMPLE (SAMPLING PERFORMED 6/14/00)

PCB SAMPLING LOCATIONS AND ANALYTICAL RESULTS

0 60 120
SCALE IN FEET



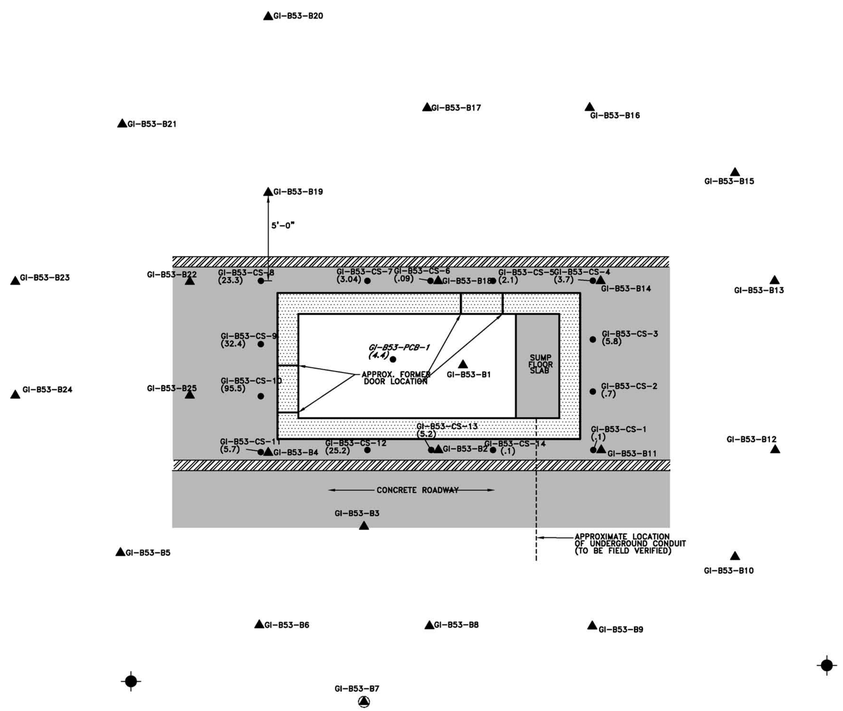
LEGEND

- ● SOIL SAMPLE LOCATION
- ⊙ CONCRETE AND SOIL SAMPLE LOCATION
- A DESIGNATES CONCRETE SAMPLE TAKEN AT SURFACE
- B DESIGNATES SOIL SAMPLE TAKEN BELOW CONCRETE
- R INDICATES ROADWAY LOCATION
- FS INDICATES PCB FIELD SCREEN

PCB FIELD SCREENING LOCATIONS AND RESULTS

SAMPLING PERFORMED ON 5/9/01

0 60 120
SCALE IN FEET

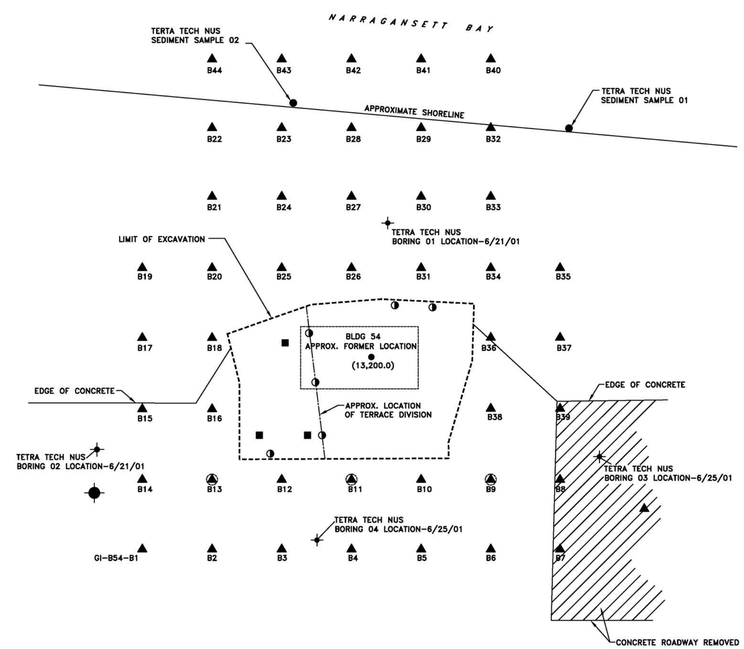


TRANSFORMER VAULT BUILDING 53 SAMPLING GRID

APPROXIMATE INTERIOR SLAB SIZE = 6' x 15'
PERIMETER SAMPLED ON 4/12/01
SCALE: 1"=4'-0"

LEGEND

- CONCRETE SLAB
- CONCRETE FOUNDATION
- 8"HIGH CONCRETE CURB
- CONCRETE RETAINING WALL
- ● EXISTING SURFACE SAMPLING LOCATION
- PROPOSED CONCRETE SAMPLING LOCATION
- ▲ PROPOSED SOIL BORING LOCATION
- PROPOSED CONCRETE AND SOIL SAMPLE LOCATION
- ●▲ PROPOSED BORING TO BE PLACED AT SAME LOCATION AS EXISTING SURFACE SAMPLE LOCATION
- ■ BOTTOM SAMPLE WITH 5/21/01 FIELD SCREEN RESULT >126 ppm
- SIDEWALL SAMPLE WITH 5/21/01 FIELD SCREEN RESULT >126 ppm
- (23.3) SOIL SAMPLE ANALYTICAL LAB PCB RESULTS IN ppm
- (95.5) WATER SAMPLE ANALYTICAL LAB PCB RESULTS IN ppm



TRANSFORMER VAULT BUILDING 54 EXCAVATION PROPOSED POTENTIAL BORING LOCATIONS

SCALE: 1"=10'

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GOULD ISLAND, NEWPORT, RHODE ISLAND
SAMPLING RESULTS AND PROPOSED BORING LOCATIONS

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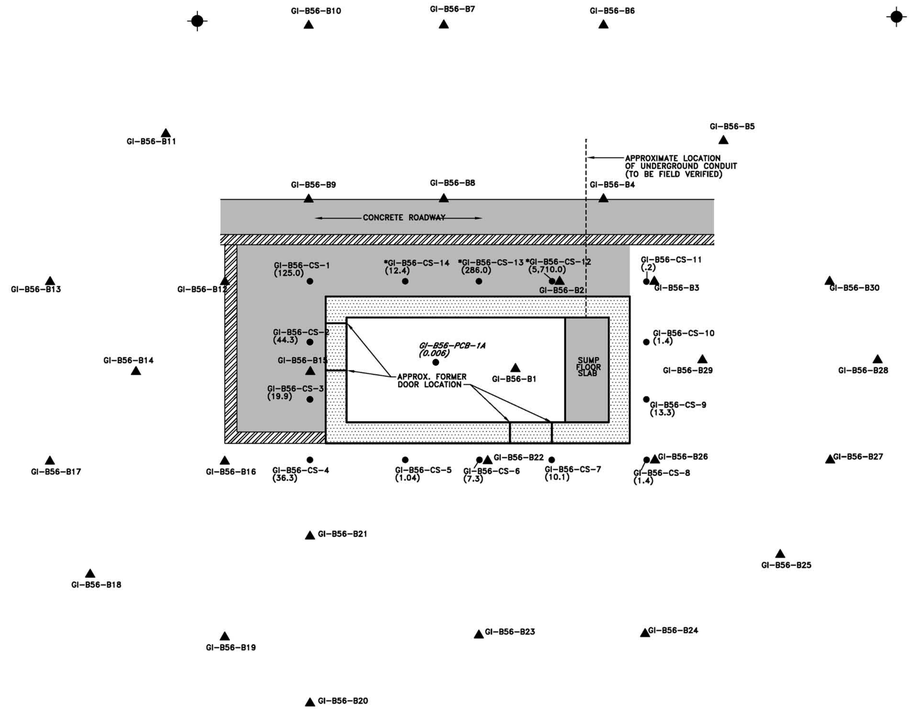
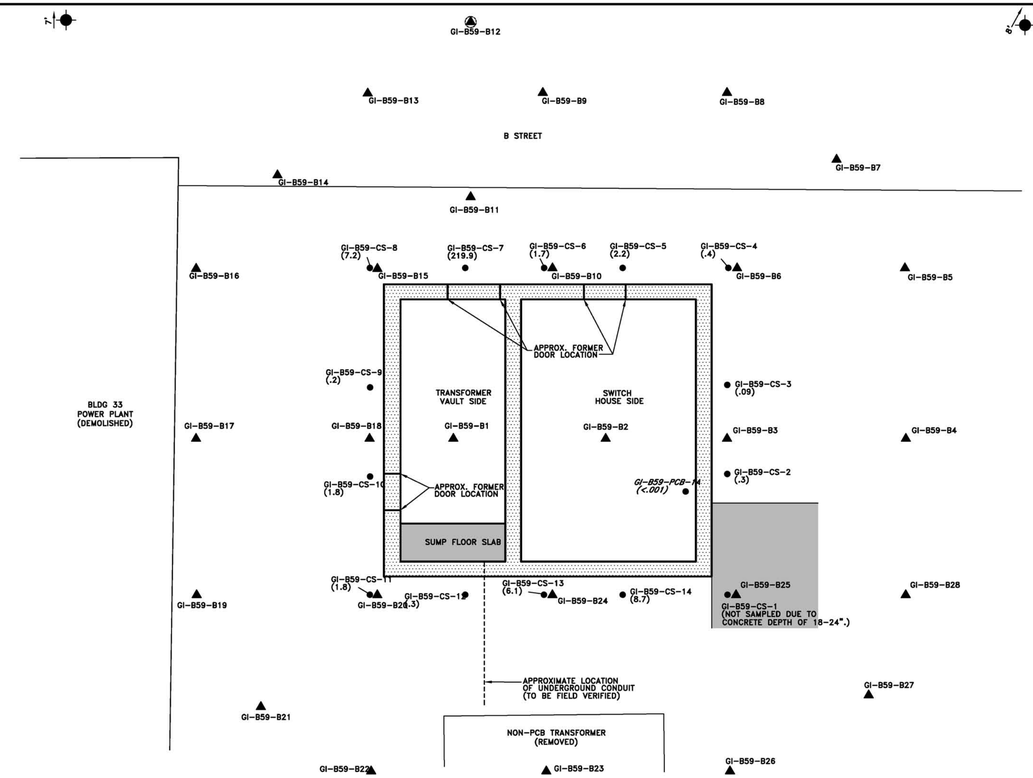


TRANSFORMER VAULT - BUILDING 56 SAMPLING GRID

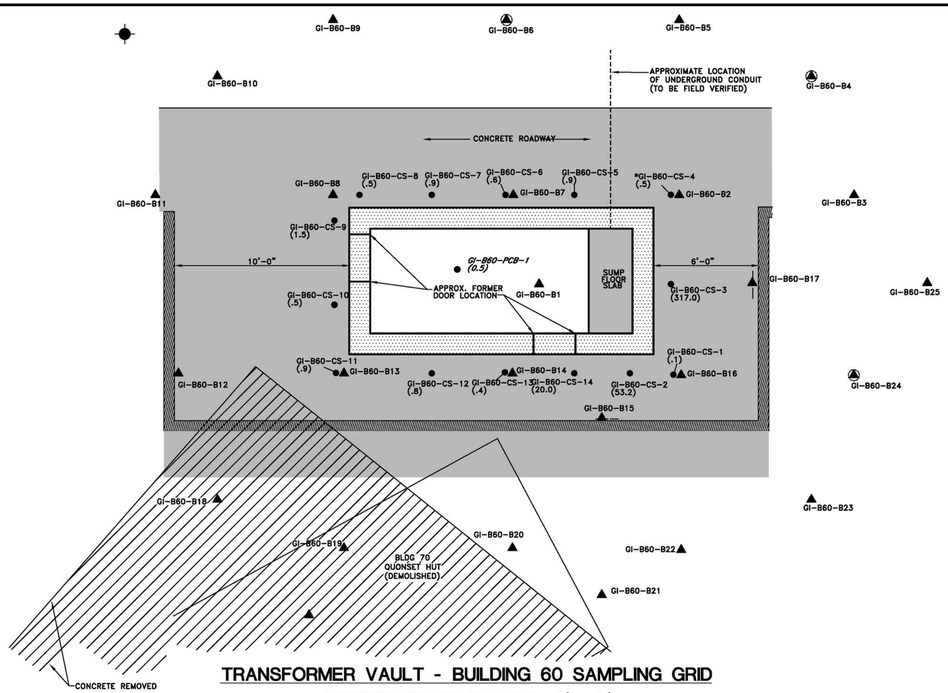
APPROXIMATE INTERIOR SLAB SIZE = 6' x 15'
PERIMETER SAMPLED ON 4/12/01
SCALE: 1"=4'-0"

* SAMPLED ON 4/16/01



SWITCH HOUSE - BUILDING 59 SAMPLING GRID

APPROXIMATE INTERIOR SLAB SIZE = 15' x 16'
PERIMETER SAMPLED ON 4/12/01
SCALE: 1"=4'-0"

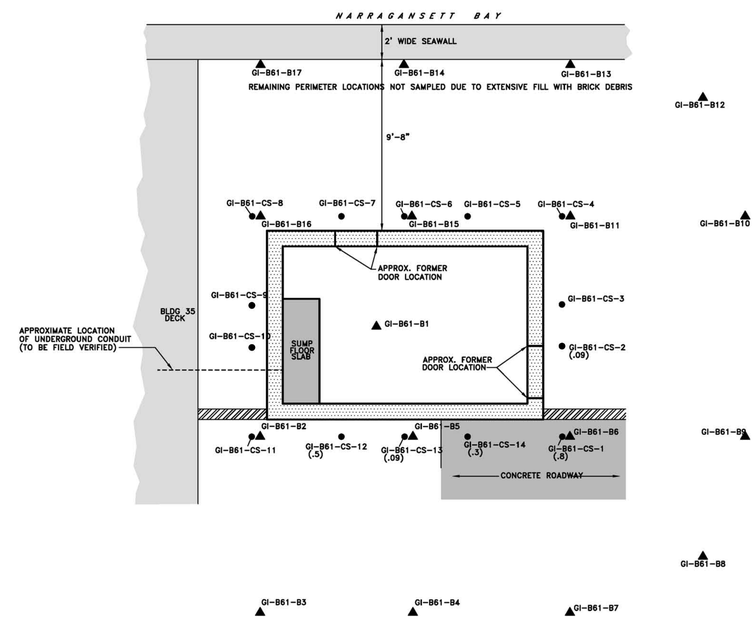


TRANSFORMER VAULT - BUILDING 60 SAMPLING GRID

APPROXIMATE INTERIOR SLAB SIZE = 6' x 15'
PERIMETER SAMPLED ON 4/12/01
SCALE: 1"=4'-0"

LEGEND

- CONCRETE SLAB
- CONCRETE FOUNDATION
- 8"HIGH CONCRETE CURB
- CONCRETE RETAINING WALL
- EXISTING SURFACE SAMPLING LOCATION
- PROPOSED CONCRETE SAMPLING LOCATION
- PROPOSED SOIL BORING LOCATION
- PROPOSED CONCRETE AND SOIL SAMPLE LOCATION
- PROPOSED BORING TO BE PLACED AT SAME LOCATION AS EXISTING SURFACE SAMPLE LOCATION
- (23.3) SOIL SAMPLE ANALYTICAL LAB PCB RESULTS IN ppm
- (95.5) WATER SAMPLE ANALYTICAL LAB PCB RESULTS IN ppm



TRANSFORMER VAULT- BUILDING 61 SAMPLING GRID

APPROXIMATE INTERIOR SLAB SIZE = 9' x 14'
PERIMETER SAMPLED ON 4/16/01
SCALE: 1"=4'-0"

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GOULD ISLAND, NEWPORT, RHODE ISLAND
SAMPLING RESULTS AND PROPOSED BORING LOCATIONS

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LATEST CHANGE
CHANGED BY:

PCBs in excess of the 10 ppm standard established by RIDEM. The concentration of PCB Arochlor 1260, detected in the concrete floor sample was 360 ppm. A composite concrete dust sample was collected from the four walls of the vault in June 2000. The concentration of PCB Arochlor 1260 detected in the wall sample was 1.6 ppm. Based on these results, the roof and walls of the building were demolished in June 2000 and the concrete debris was used on-site as fill in the Building 44 UST excavation. During demolition of the roof and walls, the floor slab was covered with poly. The concrete floor of the vault was then demolished in March 2001. The concrete floor debris was sent off site as TSCA waste to Chemical Waste Management (CWM) in Model City, NY. After floor slab demolition, a water sample was collected from beneath the slab area and analyzed in an off-site laboratory for PCBs. The water contained 4.4 ppm PCBs. In April 2001, further characterization adjacent to the vault was performed by collecting 14 perimeter soil samples using a 1.5-meter grid. These samples were sent to an off-site laboratory for analyses. Concentrations of PCB Arochlor 1260 detected in the perimeter soil ranged from 0.09 to 95.5 ppm. Data from each of these sampling events is summarized on Figure 2-2.

2.3.2 Building 54 (Transformer Vault)

Building 54 (Transformer Vault) is located along the eastside and near the south end of the Building 32 floor slab. In May 2000, a composite sample of concrete dust collected from 0 to 1 inch below the floor surface exhibited levels of PCBs in excess of the 10 ppm standard established by RIDEM. The concentration of PCB Arochlor 1260, detected in the concrete floor sample was 320 ppm. A composite concrete dust sample was collected from the four walls of the vault in June 2000. The concentration of PCB Arochlor 1260 detected in the wall sample was 2.1 ppm. The building and roof were then demolished in June 2000 and the concrete debris was used on-site as fill in the Building 44 UST excavation. On March 27, 2001 demolition of Building 54 continued with removal of the slab. The concrete demolition debris from the floor slab and soil were disposed of as TSCA waste at CWM in Model City, NY.

The basement area below the slab contained water and a small quantity of oil as floating product. The oil and water found in the Building 54 basement area were sampled and absorbent pads were used to collect the oil. The absorbent pads were then removed and placed in a roll-off container with the contaminated concrete slab debris. Initial Transformer Vault demolition activities were halted on March 29, 2001. On May 15, 2001 Foster Wheeler resumed demolition activities of Building 54. The foundation and associated soils were removed to the approximate depth of the foundation. Soil samples were collected from seventeen locations within the excavation area and analyzed using the SDI Rapid Assay Field Test Kit for PCB's. Based on these results, additional soils were removed on May 21, 2001. Soil samples were collected from twenty locations within the excavation and analyzed using the SDI Rapid Assay Field Test Kit for PCB's. Based on these results, a soil sample was collected, for laboratory analysis, from the bottom of the excavation prior to covering the excavation with poly and partially backfilling with clean fill. This sample contained 13,200 ppm PCBs.

2.3.3 Building 56 (Transformer Vault)

Building 56 (Transformer Vault) is located along the westside and near the south end of the Building 32 floor slab. In May 2000, a composite sample of concrete dust collected from 0 to 1 inch below the floor surface exhibited levels of PCBs in excess of the 10-ppm standard established by RIDEM. The concentration of PCB Arochlor 1260, detected in the concrete floor sample was 270 ppm. A composite concrete dust sample was collected from the four walls of the vault in June 2000. The concentration of PCB Arochlor 1260 detected in the wall sample was 0.9 ppm. The building walls and roof were then demolished in June 2000 and the concrete debris was used on-site as fill in the Building 44 UST excavation. During demolition a hydraulic ram hoe was used to create a hole in the floor of the slab from which a sub-slab water sample was collected. The water sample contained 0.006 ppm PCBs. The slab was demolished during March 2001, as discussed previously. The concrete debris and soil was sent off site as TSCA waste to CWM in Model City, NY facility. In April 2001, 14 perimeter soil samples were collected using a 1.5-meter grid to further characterize contamination near the vault. Concentrations of PCB Arochlor 1260 detected in the perimeter soil ranged from 0.21 to 5,710 ppm based on off-site analytical laboratory data. On May 9, 2001 one additional soil sample was collected from the north side of Building 56. This sample was analyzed using the SDI Rapid Assay Field Test Kit for PCB's. This sample contained 4 ppm PCBs.

2.3.4 Building 59 (Switch House/Transformer Vault)

Building 59 (Switch House/Transformer Vault) is located at the southeast corner of the Building 33 floor slab. Building 59 contains two separate basement areas, one beneath each the transformer side and the switchgear side of the building. In May 2000, a composite sample of concrete dust, collected from 0 to 1 inch below the floor surface of the transformer vault side, exhibited levels of PCBs in excess of the 10 ppm standard established by RIDEM. The concentration of PCB Arochlor 1260, detected in the concrete floor sample was 73 ppm. A composite concrete dust sample was collected from the four walls of the transformer vault side in June 2000. The concentration of PCB Arochlor 1260 detected in the wall sample was 2.9 ppm. In early June 2000, the vault walls and roof were demolished from both sides of the building and the concrete debris was used on-site as fill in the Building 44 UST excavation. The floor slab of Building 59 was removed from both sides of the building in March 2001. The concrete debris was sent off site as TSCA waste to CWM in Model City, NY facility. The Transformer Side of the basement was dry. The Switchgear Side of the building contained water below the slab and a sample was collected. Off-site analysis indicated the sample contained less than 0.001 ppm PCBs. In April 2001, 13 perimeter soil samples were collected using a 1.5-meter grid. The samples were analyzed at an off-site laboratory. Concentrations of PCB Arochlor 1260 detected in the perimeter soil ranged from 0.096 to 219 ppm.

2.3.5 Building 60 (Transformer Vault)

Building 60 (Transformer Vault) is located at the northeast corner of the Building 33 floor slab. In May 2000, a composite sample of concrete dust collected from 0 to 1 inch below the floor surface exhibited levels of PCBs in excess of the 10 ppm standard established by RIDEM. The concentration of PCB Arochlor 1260 detected in the concrete floor sample was 10,000 ppm. A composite concrete dust sample was collected from the four walls of the vault in June 2000. The concentration of PCB Arochlor 1260 detected in the wall sample was 0.4 ppm. Based on these results, the building roof and walls were demolished in June 2000 and the concrete debris was used on-site as fill in the Building 44 UST excavation. In December 2000, 14 concrete dust samples were collected from the surrounding roadway. The concentrations of PCB Arochlor 1260 detected in the concrete ranged from <0.1 to 0.8 ppm. Foster Wheeler utilized the hydraulic ram hoe to punch a hole in the Building 60 slab in order to collect a water

sample prior to demolition. The water sample was analyzed off-site and contained 0.536 ppm PCBs. The floor slab of Building 60 was removed in March 2001 and disposed of as TSCA waste at CWM in Model City, NY. In April 2001, 14 perimeter soil samples were collected using a 1.5-meter grid. Concentrations of PCB Arochlor 1260 detected in the perimeter soil ranged from 0.11 to 317 ppm.

2.3.6 Building 61 (Transformer Vault)

Building 61 (Transformer Vault) is located at the south end of the Building 35 floor slab. In May 2000, a composite sample of concrete dust collected from 0 to 1 inch below the floor surface exhibited levels of PCBs in excess of the 10 ppm standard established by RIDEM. The concentration of PCB Arochlor 1260, detected in the concrete floor sample was 3000 ppm. A composite concrete dust sample was collected from the four walls of the vault in June 2000. The concentration of PCB Arochlor 1260 detected in the wall sample was 0.2 ppm. The transformer vault walls and roof were demolished in June 2000 and the concrete debris was used on-site as fill in the Building 44 UST excavation. In March 2001 the floor slab was removed. The concrete debris was sent off site as TSCA waste to CWM in Model City, NY. The Building 61 basement was dry upon slab removal, therefore no water sample was collected. In April 2001, 5 perimeter soil samples were collected using a 1.5-meter grid. The samples were analyzed off-site. Concentrations of PCB Arochlor 1260 detected in the perimeter soil ranged from 0.09 to 0.847 ppm.

2.3.7 Concrete Roadway

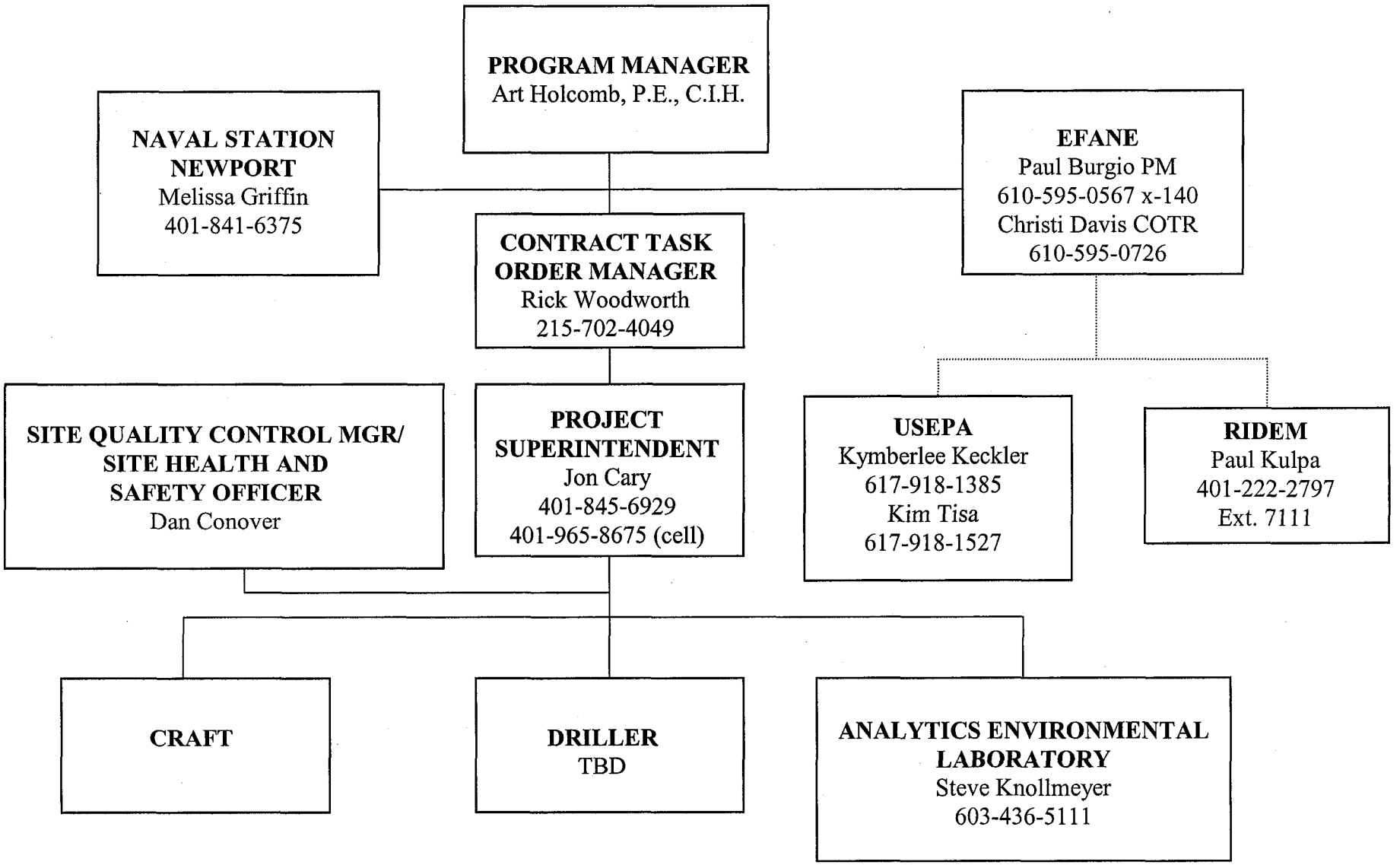
Concrete roadways are located throughout the site. In April 2001, the concrete roadway located at the southern end of the Building 32 floor slab and at the southwest corner of the Building 33 floor slab was removed. Since the concentrations in the concrete, per waste characterization sampling, were non-detect for PCBs, the material was shipped to JAM Materials in Newport, RI for recycling. On May 9, 2001 Foster Wheeler collected six soil samples from the roadway areas around the southern end of Building 32 where concrete has been removed. One concrete and one soil sample was collected from the roadway adjacent to Building 54 and 56, one concrete and one soil sample was collected from the north side of Building 54, and one soil sample was collected from the north side of Building 56. These samples were analyzed using the SDI Rapid Assay Field Test Kit for PCB's. The concentration of PCB Arochlor 1260 detected in the soil samples ranged from less than 6 to 23 ppm. Refer to Figure 2-2 for sampling locations and field analytical results.

3.0 PROJECT MANAGEMENT

The Foster Wheeler organizational chart for this Sampling Plan is depicted as Figure 3-1. As noted on the chart, and discussed during negotiations, certain key Foster Wheeler personnel will perform multiple roles at the site for project implementation. The responsibilities for each of the roles are discussed in the following sections.

The Project Management Team is configured such that one person, Rick Woodworth, has overall administrative and technical responsibilities for this Contract Task Order. As the Contract Task Order Manager, Mr. Woodworth reports directly to Art Holcomb, the RAC Program Manager. Mr. Woodworth is responsible for planning and scheduling the removal actions, ensuring project deliverables are submitted on a timely basis, tracking and managing budgets and schedules, and maintaining a safe work environment and a record of site activities. Mr. Woodworth will also attend bi-weekly quality control

FIGURE 3-1
ORGANIZATIONAL CHART
CHARACTERIZATION OF PCB CONTAMINATED SOILS AND CONCRETE – GOULD ISLAND



(QC) meetings to discuss schedule and progress updates with the Navy. All coordination with the regulatory agencies will be the responsibility of the Navy.

Jon Cary will act as the Project Superintendent and will be responsible for managing and directing all on-site activities. These activities will include supervision of all field staff and subcontractors, site procurement of materials, interfacing with the Navy Resident Officer In Charge of Construction (ROICC), and ensuring compliance with the sampling plan scope.

The Site Engineer's responsibilities will be handled by Dan Conover. Mr. Conover will be responsible for tracking all engineering and technical documentation, including submittals.

Mr. Conover will also perform the duties of the Site QC Manager (SQCM) and will act on behalf of the Program QC Manager, Tom Kelly, for all site-related quality control activities. The SQCM will be responsible for ensuring compliance with the QC Plan, Test Plan and Log, and Submittal Register and tracking of all field documentation.

Mr. Conover will serve as the Site Health and Safety Officer (SHSO) on behalf of the Program Health and Safety Manager, Grey Coppi, for all site-related health and safety issues, and will ensure adherence to the *Final Master Site Health And Safety Plan for Gould Island, Naval Station Newport, 1999* (SHSP) by all on-site personnel.

Field activities will be supported by Tom Teeling in Regulatory Compliance.

Note that the Contract Task Order Manager will communicate directly with the Project Superintendent. All of the aforementioned personnel are dedicated to this project full-time.

During the site activities, field staff will receive assistance from procurement and project controls personnel at both the Gould Island Site and Langhorne, PA offices. These personnel will provide part-time assistance to the site for field procurement, scheduling, and status reports .

Prior to mobilization, procurement activities will be supported on a part-time basis through the Program Procurement Department located in Langhorne, PA. On-site, Brian Delaney will act as the Project Controls Engineer. The Project Controls function will include updating the project schedule and cost tracking databases and preparing status reports. Maura McCandless will be responsible for procurement of materials at the site and field accounting activities.

4.0 QUALITY CONTROL

4.1 General

Foster Wheeler's RAC Program Organization is specifically designed to control work performed by the Foster Wheeler Team in accordance with the contract requirements. Foster Wheeler will manage this contract through the dedicated Program Management Office (PMO) located in Langhorne, Pennsylvania.

The RAC Program is organized into four elements under the Program Management Team: 1) Contract Administration; 2) Quality Control; 3) Health and Safety; and 4) Senior Project Engineer/Manager, along with the PMO support groups which will provide assistance to the Project Management Team on an as-needed basis. The PMO support groups themselves are organized into five elements: 1) Procurement; 2) Planning and Scheduling; 3) Cost Estimating; 4) Budgeting and Accounting; and 5) Technical and

Regulatory Compliance. Five of the above elements (Contract Administration, Planning and Scheduling, Cost Estimating, Budgeting and Accounting, and Technical and Regulatory Compliance) are non-remediation related and are not addressed in this Quality Control Plan (QC Plan). The major function of the QC Plan is to ensure that all organizational elements perform the assigned actions in compliance with the contract.

The QC plan provides for monitoring, auditing and conducting field inspections to ensure compliance is being maintained. Maintenance of the project records and required reports and logs is also addressed. A program to assure all submittals are correct and complete before forwarding to the Contracting Officer's Representative is included in this Plan.

4.2 Quality Control Group

The Quality Control Group assigned to the Program is technically responsible to the Foster Wheeler Corporate Quality Assurance Officer. The Group is headed by the Program Quality Control Manager (PQCM) who receives administrative direction from the Program Manager for the purpose of coordinating QC activities with the Contract Task order operations, testing sequences and schedule, and achieving timely resolution of quality issues.

The Quality Control Group consists of the Program Quality Control Manager (PQCM) and the Site QC Manager (SQCM). The QC organization is shown on Figure 4-1.

4.2.1 Program Quality Control Manager (PQCM)

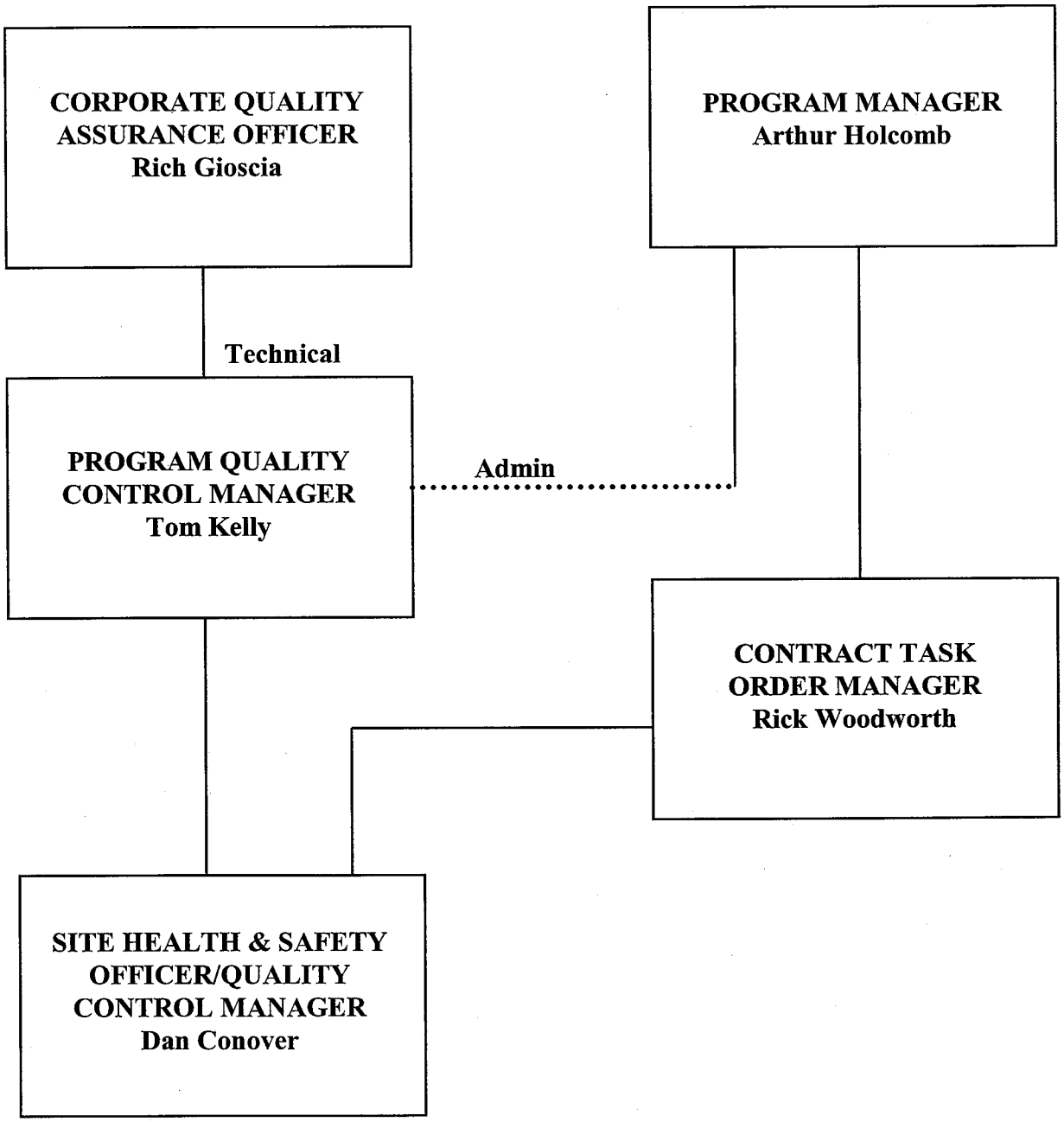
The PQCM is responsible for approving the QC Plan for the project; assuring that all relevant portions of the QC Plan are implemented during the project through audits and surveillance of project activities; issuing report to the Contract Task Order Manager on any deviation from the approved plans; reviewing, modifying or correcting all contract submittals prior to forwarding to the Contracting Officer's Representative; and authorizing the SQCM to act on his behalf for all site related quality control activities. As stated in Section 2.0, the PQCM is Mr. Tom Kelly.

4.2.2 Site Quality Control Manager (SQCM)

The SQCM will implement the Foster Wheeler Quality Control Program and will have the sole responsibility of ensuring compliance with contract documents. He shall have the authority to reject material or workmanship that do not comply. A pictorial representation of SQCM responsibilities is shown in Figure 4-1. The SQCM, or a designated representative acceptable to the Navy, will be present at the project site whenever sampling or testing is in progress. The major responsibilities of the SQCM as outlined in Section C Part 6.0 of the basic contract (Quality Control) of the contract include:

- Managing and implementing an effective Quality Control Program;
- Notifying the Contracting Officer's Representative, EPA, and RIDEM at least one (1) week in advance of any field activities and at least 24 hours in advance of any cancellations of work;
- Conducting QC meetings at the site with the Project Superintendent on a bi-weekly basis, notifying the Contracting Officer's Representative at least 48 hours in advance of each meeting, and preparing and submitting the meeting minutes to the Contracting Officer's Representative within two (2) working days after the meeting;
- Ensuring submission of bi-weekly QC meeting minutes to EPA and RIDEM;
- Providing documentation of daily field activities in the Contractor Production Report and the Contractor Quality Control Report (see Appendix A);

Figure 4-1
Quality Control Organizational Chart



- Identifying, controlling, and assuring resolution of deficiencies, including corrective action implementation;
- and
- Maintaining project records as required by the contract or statute.

As stated in Section 3.0, the SQCM is Dan Conover.

4.3 Submittals Processing

4.3.1 General

The number and type of submittals to be issued are outlined in the Submittal Register (Table 4-1). Foster Wheeler shall use and maintain the Submittal Register to track submittals from issue to approval, and to comply with the Navy requirement for submittals under Section C, Part 7.0 of the Basic Contract. Foster Wheeler shall forward submittals requiring Navy approval and shall submit these items as listed on the Submittal Register.

Each submittal shall be complete and in sufficient detail for ready determination with the contract requirements. All items shall be checked and approved by the PQCM and shall be transmitted via a submittal form (see Appendix A) which shall be initialed and dated by the PQCM indicating that the accompanying submittal conforms or does not conform to contract requirements.

4.3.2 Receipt of Submittals

Submittals shall be electronically submitted to:

Department of the Navy
Engineering Field Activity - Northeast
Ms. Christi Davis
Mr. Todd Bober
Mr. Paul Burgio

Naval Station Newport
Mr. Bob Krivinskas (Resident Officer in Charge of Construction)
Ms. Melissa Griffin (Environmental)

U.S. EPA Region I
Ms. Kim Tisa

Rhode Island Department of Environmental Management
Mr. Paul Kulpa

Each submittal shall be identified with:

- Contract Number and Contract Task Order Number;
- Unique sequential transmittal number
- Name and address of the submitting organization;
- Date of submittal;
- Description of item being submitted; and
- Approval of submitting organization indicating conformance to requirements.

Table 4-1
Submittal Register

Item No.	SD Number and Type of Submittal Material or Product	EFANE	ROICC	A/E	Transmittal Control Number	Date Submitted
	Preconstruction Submittals					
1	Draft SHSP	X	X			
2	Responses to Comments on SHSP	X	X			
3	Final SHSP	X	X			
4	Draft Sampling Plan (to Navy)	X	X			
5	Revised Draft Sampling Plan (to Regulators)	X	X			
6	Final Sampling Plan (to Navy)	X	X			
7	Revised Final Sampling Plan (to Regulators)	X	X			
8	Draft Work Plan (to Navy)	X	X			
9	Revised Draft Work Plan (to Regulators)	X	X			
10	Final Work Plan (to Navy)	X	X			
11	Revised Final Work Plan (to Regulators)	X	X			
	SD-08 Statements					
12	Training Certification		X			
13	Transporter Certification of EPA Notification		X			
14	Sampling Data	X	X			
15	Certificate of Disposal	X	X			
	SD-10 Test Reports					
16	Immunoassay Test Results	X	X			
	SD-18 Records					
17	Miscellaneous Documentation		X			
	EFANE & ROICC: Specific Submittal Review/Approval - Lester, PA /Newport, RI					

The PQCM shall enter each submittal into the Submittal Register, determine if approval is required by the Contracting Officer's Representative, and proceed with review of submitted material.

4.3.3 Review and Processing of Submittals Which Do Not Require Navy Approval

Material submitted for review by the PQCM or designated representative shall indicate that it either conforms to established requirements or does not conform to established requirements. The PQCM shall advise the submitter of the results of the review. The submittal log will be updated to indicate status. Note: conforming submittals shall be transmitted to Project and Navy personnel as determined by the distribution schedule; non-conforming submittals shall be returned to the submitter for correction, resolution of comments, and re-submittal.

4.3.4 Review and Processing of Submittals Which Require Navy Approval

Material submitted for review by the PQCM or designated representative shall be signed to indicate that it conforms to requirements. The submittal will then be transmitted in accordance with the project distribution schedule for review and approval. All items sent to the Navy shall be done so through a submittal form, which will indicate each item transmitted, the date reviewed by the PQCM, and its review status. Upon completion of review, the Contracting Officer's Representative shall return the submittal form to the SQCM for further action.

The PQCM will advise the submitter of the results of the review in writing and include any comments. The submittal log will be updated to indicate status. Note: non-conforming submittals shall be returned to the submitter for correction, resolution of comments, and re-submittal if required.

4.3.5 Revised Submittals

Revised submittals shall be logged, reviewed, and processed in a manner identical with the initial submittal and will comply with the Navy requirements under Section C, Part 7.0 of the Basic Contract.

4.4 Testing and Sampling Methodology

4.4.1 Analytical and Field Screening Testing and Sampling

Analytical testing, field screening and sampling activities shall be carried out using approved methodology and at the frequency indicated in Table 4-2. Field screening tests and analytical testing and sampling shall be performed using the methodologies presented in Section 5.0.

The Contracting Officer's Representative must approve the independent analytical laboratory to be used. The SQCM shall be the responsible person, supervising all testing and sampling activities and reviewing the qualifications of the independent testing laboratories prior to submitting any samples for test. Samples to be tested by the independent laboratory shall be collected by either laboratory or trained field personnel (i.e., employees with prior experience in field sampling who are familiar with the field sampling and laboratory testing section of this Sampling Plan).

Table 4-2
Test Plan and Log

CONTRACT NUMBER N62472-99-D-0032	PROJECT TITLE AND LOCATION CHARACTERIZATION OF PCB CONTAMINATED SOILS, GOULD ISLAND, RI								CONTRACTOR FOSTER WHEELER		
		ACCREDITED/ APPROVED LAB				LOCATION OF TEST					
ITEM OF WORK	TEST REQUIRED	YES	NO	SAMPLED BY	TESTED BY	ON SITE	OFF SITE	FREQUENCY	DATE COMPLETED	DATE FORWARDED TO CONTR. OFF.	REMARKS
WASTE CHARACTERIZATION SAMPLING	SOIL/CONCRETE/DEBRIS - VOCs; SVOCs; RCRA METALS; PCBs; CORROSIVITY; IGNITABILITY; REACTIVITY (CYANIDE & SULFIDE)	X					X	ONE PER 20 YD^3 ROLLOFF (MIN)			
	WATER - VOCs; SVOCs; PCBs; RCRA METALS; CORROSIVITY; IGNITABILITY; REACTIVITY (CYANIDE & SULFIDE)	X					X	ONE PER 20,000 GAL FRAC TANK (MIN)			
PRELIMINARY IMMUNOASSAY SAMPLING	SOIL – PCBs		X			X		PER SAMPLING GRID			
LABORATORY CONFIRMATORY SAMPLING	SOIL – PCBs	X					X	ONE PER 10 IMMUNOASSAY SAMPLES + DUPLICATES OF THE LOWEST SAMPLE BELOW THE GOAL(S) (SEE TABLE 5-1) + SAMPLES FROM WITHIN OR BELOW WATER TABLE			
	CONCRETE - PCBs	X					X	PER SAMPLING GRID			
	SEDIMENT - PCBs	X					X	PER SAMPLING GRID			

4.4.2 Chain of Custody

A chain of custody (COC) record will be used to provide evidence that samples for chemical analysis are under continuous control up until the analysis or test is complete. The COC record for laboratory chemical analysis is included in Appendix A.

4.4.3 Equipment Calibration

All laboratory analysis will be performed by Analytics Environmental Laboratory (Analytics) of Portsmouth, New Hampshire. Analytics' equipment calibration procedures and frequencies have been provided in their Standard Operating Procedures (SOPs), included as Appendix C. Calibration frequencies of real-time dust monitoring instruments are described in Section 7.0 of the SHSP.

4.4.4 Outside Organizations

Qualifications for the drilling and waste disposal subcontractors, to be employed by Foster Wheeler to perform on-site services will be provided to the Navy at the time of service.

4.5 Quality Control Site Activities

4.5.1 Introduction

This section addresses all aspects of Quality Control Site Activities, including: QC inspection activities; the identification of work features to be inspected; and the control of subcontractors and vendors.

4.5.2 Quality Control Inspection Activities

Three Phases of Control

The SQCM shall perform three phases of control to ensure that work complies with the Sampling Plan, the SHSP (Foster Wheeler, 1999), and all applicable federal, state, and local rules and regulations. The three phases of control, as defined below, shall adequately cover both on-site and off-site activities for each definable feature of work. A definable feature of work is a task that is separate and distinct from other tasks and requires separate control requirements. The definable features of work and the QC inspection and verification activities for the major technical work elements are identified in Table 4-3.

Table 4-3

Inspection Schedule

ACTIVITY	PREPARATORY	DONE	INITIAL	DONE	FOLLOW-UP	DONE
Mobilization	Operations to be carried out in accordance with approved procedures		• Establish equipment and material laydown areas		On-going inspection of site to assure compliance with OSHA and SHSP	
Site Preparation	Operations to be carried out in accordance with approved procedures		• Establish site zones • Establish decon areas • Install erosion controls • Assure that all Freedom of Information and emergency information is posted		On-going inspection of areas	
Sampling	Operations to be carried out in accordance with approved procedures		• All sample collection performed in accordance with Sampling Plan		Provide sample results in accordance with submittal register	
Removal and Disposal of Wastes	Operations to be carried out in accordance with approved procedures		• All removal activities performed in accordance with Sampling Plan • Photographic documentation • Manifests completed		On-going inspection of all removal activities and proper disposal of materials	
Demobilization	Operations to be carried out in accordance with approved procedures		• Breakdown of temporary facilities • Removal of erosion controls		On-going inspection of site to assure compliance with OSHA and SHSP	

1. Preparatory Phase

The SQCM shall notify the Contracting Officer's Representative at least two (2) workdays in advance of each preparatory phase inspection to allow for his/her participation in the inspection if desired. Preparatory phase inspections shall be documented on the Preparatory Phase Inspection Checklist (see Appendix A) and in the Contractor Quality Control Report. The SQCM shall perform the following prior to the commencement of each definable feature of work:

- Review the testing plan and ensure that provisions have been made to provide the required Quality Control testing;
- Examine the work area to ensure that the required preliminary work has been completed;
- Review the SHSP and appropriate activity hazard analysis to ensure that applicable safety requirements are met and that required Material Safety Data Sheets (MSDSs) are submitted; and
- Discuss sampling methods.

2. Initial Phase

The SQCM shall notify the Contracting Officer's Representative at least two (2) work days in advance of each initial phase inspection. When sampling crews are ready to start a definable feature of work, an initial phase inspection shall be conducted with the Project Superintendent.. The initial segment of the definable feature of work shall be observed to ensure that the work complies with contract requirements. Initial phase inspections shall be documented on the Initial Inspection Checklist (see Appendix A) and in the Daily Quality Control Report. The SQCM shall perform the following for each definable feature of work:

- Review the SHSP and the appropriate activity hazard analysis to ensure that applicable safety requirements are met; and
- Ensure that testing is performed by the approved laboratory.

3. Follow-up Phase

Follow-up phase inspections are similar in content and approach to initial phase inspections, and shall be performed as needed during on-going work. Follow-up phase inspections shall be documented in the Daily Quality Control Report. The SQCM shall perform the following for each definable feature of work:

- Ensure the work is in compliance with contract requirements;
- Ensure that testing is performed by an approved laboratory; and
- Ensure that rework items are being corrected.

Work Features Requiring Inspection or Testing

Prior to implementation of individual phases of work activities, the SQCM and Project Superintendent shall meet to identify specific work requirements, including submittal information, scheduling, and Quality Control requirements. This joint review allows close coordination of work and maximizes efficiency of operations. Project roles, potential problems, and procedures for resolving issues shall be established up-front at these discussions to allow for clarification of direction and immediate response to

any problems that may arise. As a result of this approach, Quality Control activities shall be maintained as an integral component of the overall project approach.

Completion Inspection

At the completion of all work or any increment thereof, the SQCM shall conduct a completion inspection of the work.

4.5.3 Control of Subcontractors and Vendors

Construction subcontractor's qualifications to perform the required work shall be evaluated by the SQCM. He shall be responsible for ensuring compliance with the Sampling Plan in all work performed by these subcontractors. All subcontractor activities shall be subject to QC inspection in accordance with Section C, Part 6.0 of the Basic Contract.

4.6 Documentation

4.6.1 General

All inspection and testing activities performed shall be documented by the SQCM. In addition to the forms provided in this Sampling Plan in Appendix A, forms required by the Navy shall be completed and submitted to the Contracting Officer's Representative as applicable. Also included in this Sampling Plan is a Quality Assurance Project Plan (QAPP) Addendum which can be found as Appendix B.

4.6.2 Daily Quality Control Report

The SQCM or his designee shall record his inspection activities in the Daily Quality Control Report. The Daily Quality Control Report shall be submitted to the Contracting Officer's Representative daily. Reports for weekends and holidays shall be included on the first working day's report following those periods.

5.0 FIELD SAMPLING AND LABORATORY TESTING

5.1 Analytical Sampling Programs

The characterization to be performed on Gould Island will require field sampling and analysis data in support of the decision making process. To be valuable, this data must accurately describe the characteristics and concentrations of constituents in the samples analyzed. The field sampling program will ensure that the chemical data meet the *Navy Installation Restoration Chemical Data Quality Manual*, dated September 1999, as well as *Region I, EPA-New England Compendium of Quality Assurance Project Plan Guidance, Final*, dated October 1999 requirements for completeness, precision, accuracy, representativeness, comparability, dependability, and legal defensibility. The analytical sampling programs for this project will include the following:

- Sampling and analysis of various media to determine the extent of PCB contamination.
- Sampling and analysis of various media for off-site disposal waste characterization.
- Real-time dust monitoring

5.2 Data Quality Objectives

Chemical data generated for this project will primarily be used to characterize materials in order to determine the extent of PCB contamination on the site and to determine applicable regulatory and disposal requirements. All analyses will be performed in strict accordance with EPA approved methodologies. Detection limits will be sufficiently below action levels and regulatory criteria so as to provide accurate enough data to determine the extent of PCB contamination. Sample collection will utilize approved EPA techniques that will ensure that the sample will be representative of current environmental conditions. As described in Section 5.5, QA/QC samples will be collected and analyzed for the purpose of assessing the quality of the field screening effort and the analytical data. Table 5-1 indicates the number of field samples, number of QA/QC samples, and analytical methods. All laboratory analysis will be performed by Analytics Environmental Laboratory (Analytics) of Portsmouth, New Hampshire. Analytics is a certified laboratory using EPA approved methodologies. Analytics lab certification will remain current throughout the duration of the project.
Data Quality Objectives (DQOs) for this project were developed based on the EPA Guidance for the Data Quality Objectives Process, EPA-QA/G-4, August 2000. This guidance replaces earlier guidance, referencing the five analytical levels describing data quality. The process of establishing data quality objectives is intended to provide projects with the data necessary to make decisions in a cost-effective manner. The data categories (replacing the previously referenced analytical levels) are summarized in the following subsections.

5.2.1 Data Categories

Data collected for the project can be classified into two descriptive categories. These data categories replace previous references to analytical levels, quality assurance objectives, and data use categories. The two categories are summarized below.

- Screening Data: Screening data are generated by rapid, generally less precise methods of analysis with less rigorous sample preparation than conventional laboratory methods. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent instead of elaborate extraction/digestion and cleanup. Screening data provide tentative analyte identification and partial quantitation, although the quantitation may be relatively representative in nature. At least 10 percent of the screening data will be confirmed using 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.

- Definitive Data: Definitive data are generated using rigorous analytical methods, such as approved EPA referenced methods. Data are analyte specific with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatogram, spectra, and 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.

Table 5-1
Summary of Analytical Procedures and QC Requirements

Sampling Program/Analysis	Method	Field Samples	Field Dup.	MS/ MSD	Equip. Blank
Transformer Vault Soil					
PCB Immunoassay Field Screen	SW846 4020	974[1]	40	0	0
PCBs (extraction/analysis)	SW846 3550/8082	460[1]	23	23	25
Roadway Soil (Concrete previously removed)					
PCB Immunoassay Field Screen	SW846 4020	111[1]	6	0	0
PCBs (extraction/analysis)	SW846 3550/8082	14[1]	1	0	0
Sediment Sampling (Building 54 shoreline)					
PCBs (extraction/analysis)	SW846 3550/8082	40[1]	2	0	0
Roadway Soil (Assume concrete has PCBs in lower sample – 20% of locations)					
PCB Immunoassay Field Screen	SW846 4020	127[1]	6	0	0
PCBs (extraction/analysis)	SW846 3550/8082	14[1]	1	0	0
Concrete Roadway					
PCBs (extraction/analysis)	SW846 3550/8082	212	11	11	11
Wipe Sampling					
PCBs (extraction/analysis)	SW846 3550/8082	4	0	0	0
Waste Characterization - Solid					
PCBs (extraction/analysis)	SW846 3550/8082	1[2]	0	0	0
TAL Metals (Total)	SW846 6010/7000	1[2]	0	0	0
VOCs	SW846 8260B	1[2]	0	0	0
SVOCs	SW846 3550B/8270C	1[2]	0	0	0
Reactive Cyanide	SW846 Sec.7.3.3.2/9010	1[2]	0	0	0
Reactive Sulfide	SW846 Sec.7.3.4.2/9030	1[2]	0	0	0
Corrosivity	SW846 9045	1[2]	0	0	0
Ignitability	SW846 1010 or 1020	1[2]	0	0	0
Waste Characterization - Liquid					
PCB	SW846 8082	1[2]	0	0	0
TAL Metals (Total)	SW846 6010/7000	1[2]	0	0	0
VOCs	SW846 8260B	1[2]	0	0	0
SVOCs	SW846 3510C/8270C	1[2]	0	0	0
Reactive Cyanide	SW846 Sec.7.3.3.2/9010	1[2]	0	0	0
Reactive Sulfide	SW846 Sec.7.3.4.2/9030	1[2]	0	0	0
Corrosivity	SW846 9045	1[2]	0	0	0
Ignitability	SW846 1010 or 1020	1[2]	0	0	0

[1] Proposed potential # of samples – if the upper depth and/or interior samples are below cleanup goals, the lower depths and/or exterior samples will not be analyzed.
[2] Minimum quantity of samples anticipated. Actual number will be determined based on disposal facility requirements.

5.3 Sample Collection Procedures

Several sampling programs for various media around the site will be conducted as part of this characterization. Specific sampling protocols are identified below. A summary of the analytical sampling programs, numbers of samples, analytical methods, and QA/QC sample quantities anticipated for this project is included in Table 5-1. QA/QC samples will be collected as identified in Section 5.5. All sampling equipment will be decontaminated prior to use by procedures identified in Section 5.6. All sample identification and labeling, custody procedures, sample tracking, and sample packing and shipping will be performed in accordance with procedures identified in Section 5.7. Sample containers, preservatives, and holding time requirements are included in Table 5-2.

5.3.1 Waste Characterization Sampling

Waste generated during site activities will be sampled in order to characterize the waste for disposal. All wastes other than the contaminated water will be disposed as TSCA for this activity. Wastes may include, but not be limited to, concrete debris, wastewater, soil, decontamination water, PPE and PCB field test kits. Table 5-1 indicates the anticipated numbers of samples required for this activity. Additional testing and testing frequencies may be required by the disposal facility.

Concrete Dust, Soil Cuttings and Debris/ PPE

If required by the disposal facility, one composite solid sample will be collected from containerized concrete dust, soil cuttings, and Debris/PPE. The composite sample will be analyzed for PCB, VOC, SVOC, RCRA Metals, Reactivity, Corrosivity, and Ignitability.

Wastewater

A composite sample of the water pumped from the transformer vaults and stored for off-site disposal will be collected for waste characterization. The water sample will be collected using a disposable bailer and analyzed for PCB, VOC, SVOC, RCRA metals, Reactivity, Corrosivity, and Ignitability.

Decontamination Water

Decontamination water will be combined with the wastewater and sampled as described above.

5.3.2 Concrete Roadway Sampling

Concrete samples will be collected using EPA Standard Operating Procedure for Sampling Concrete in the Field, December 1997. A 20-foot by 20-foot sampling grid will be established on the concrete roadway and pad to locate and identify samples. In addition, areas with visual staining will be sampled for PCB analysis. The top one inch of the concrete will be drilled into with a one and one-half inch drill bit and the concrete powder will be collected in a labeled sample jar. When the thickness of the concrete is known for a sample location, the one and one-half inch hole will be advanced until approximately one inch of the concrete slab remains. The hole will be vacuumed out to remove all concrete dust. Without touching the sides of the hole, a one-inch drill bit will be advanced to collect the lower sample. "Punch through" will be avoided, if possible. If a concrete sample cannot be obtained from the initial hole, a second boring will be made directly adjacent to the first one.

Table 5-2
Sample Containers, Preservatives, and Holding Times

Media/Analysis	Container	Preservative	Holding Time (1)
Soil, Concrete, Sediment & Debris Sampling			
PCB	4 oz. wide mouth jar	Ice to 4° C	Extract within 14 days; analyze extract within 40 days
VOC	2 oz. wide mouth jar	Ice to 4° C	Analyze within 14 days
TAL Metals (Total)	4 oz. wide mouth jar	Ice to 4° C	Analyze mercury within 28 days; analyze all other metals within 6 months
SVOC	4 oz. wide mouth jar	Ice to 4° C	Extract within 7 days; analyze extract within 40 days
Reactivity, Corrosivity, Ignitability	4 oz. wide mouth jar	Cool	Analyze within 14 days
Wastewater Sampling			
PCB	1L. amber	Cool	Extract within 7 days; analyze extract within 40 days
VOCs	2x40 ml. VOCs	pH<2,HCL, cool	Analyze within 14 days
TAL Metals	8oz. Plastic	pH<2,HNO3	Analyze within 180 days, mercury within 28 days
SVOC	1L. amber	Cool	Extract within 7 days; analyze extract within 40 days
Reactivity, Corrosivity, Ignitability	8oz. Plastic	Cool	Analyze within 14 days
Wipe Sampling			
PCBs	4 oz. wide mouth jar	Cool	Analyze within 14 days

Note: (1) Holding time is calculated from sampling date.

Care must be taken to note the moisture content of the concrete prior to sampling. The powdered concrete generated by the drill bit will be difficult to collect if it is wet. To avoid these problems, no samples will be collected if there is standing water at the sample location on the concrete pad. The field chemist will use personal judgment to determine where a valid sample may be collected.

The concrete dust from the lower one-inch sample will be composited with the concrete dust from the upper one-inch sample for a single analysis. This composited sample will be sent to an offsite laboratory for expedited 48-hour turnaround. Refer to Section 5.4 for the Analytical Methodology. If the sample demonstrates PCB concentrations greater than 1 ppm, the soil beneath the concrete at that location will be sampled and analyzed for PCBs.

5.3.3 Soil Sampling

The horizontal and vertical extent of PCB contamination in the soil will be determined using immunoassay field analysis and off site confirmation. Soil samples will be collected at one-foot intervals to a depth of approximately two feet below the water table. Since the immunoassay screening kits are sensitive to moisture content in the sample, they may not give accurate results if the sample contains greater than 30% moisture. Therefore, samples from one foot above the water table and higher will be analyzed on site using immunoassay testing, while samples within or below the water table will be sent off site to the fixed laboratory. Accounting for water table fluctuations, it is anticipated that a depth of 2-feet below the water table will allow sampling of the entire smear zone. Should data indicate that the deepest sample collected still contains PCBs above the target levels, the boring will be deepened at that location in 4-foot increments. The core will be sampled at one-foot intervals.

Transformer Vault Perimeter sample locations have been focused on areas of known contamination based on previous sampling data and expanded upon by using a ten-foot grid. Figures 2-2 and 2-2a provide the proposed potential sample locations around each vault. Also shown on the figures, are the approximate locations of conduit runs that exit the vaults. The locations have been selected to provide a minimum of one (1) additional sample location extending outward from each exterior vault sample location in the horizontal plane. Should the lateral extent of PCB contamination extend beyond the sampling area, the sample grid will be extended by 10-foot increments until the full extent of contamination has been determined. Where concrete is present at the boring location, a jackhammer will be used to create an opening for the Geoprobe. A HEPA vacuum will be used to remove all concrete debris from the area prior to installation of the boring.

Roadway soil sampling locations, where concrete has been removed, will be determined using a 20-foot by 20-foot sampling grid. Figure 2-2 provides the proposed potential sample locations. The locations have been selected to provide thorough analysis of the extent of PCB contamination within the soil around the southside of Buildings 32 and 33.

5.3.4 Sediment Sampling

Sediment sample locations, along the east shoreline adjacent to Building 54, will be determined based on previous sampling data and by using a ten-foot grid. Best professional field judgment will be used to maximize collection of organic sediment as opposed to rocky sandy sediment. Figure 2-2 provides the proposed potential sample locations. The locations have been selected to provide a thorough analysis of the extent of PCB contamination toward the shoreline. Should the lateral extent of PCB contamination extend beyond the sampling area, further into the bay, a determination will be made by the Navy whether additional sampling beyond the shoreline will be conducted during this sampling activity.

Samples will be retrieved using a Geoprobe with an acetate sleeve to contain the sample (Macrosampler). The sleeve will be cut into four 1-foot lengths for sample collection. Based on previous experience at the site, it is anticipated that nearly full recovery of the 4-foot length will be possible with this method. If recovery is less than required for sampling purposes, field adjustments to the sampling methodology will be made. These adjustments may include minor relocation of the sample, hand augering of the sample, or other methods to allow increased sediment recovery.

5.3.5 Wipe Sampling

Wipe samples will be collected, prior to demobilization, from all non-disposable equipment that comes in contact with potential PCB contamination. The results will be compared against a 1 ppm standard. Samples will be obtained as follows:

- The sample technician will wear clean disposable gloves for each wipe sample obtained.
- A collection area of 100 cm^2 will be marked at each sample location.
- A gauze wipe moistened with hexane will be placed on the surface to be sampled and rubbed vertically over the entire sample area.
- The wipe will then folded and rubbed horizontally (or at a 90° angle to the first wipe pattern) over the entire sample area.
- The folded wipe will be placed in a labeled 4-oz. glass jar for laboratory analysis.
- A minimum of one field blank will be submitted to the laboratory for each sampling event. A field blank will be obtained by removing an unused gauze wipe from its packaging, moistening the wipe with hexane, and then folding the wipe and immediately placing it in a labeled 4-oz. glass jar for laboratory analysis. The field blank will be collected prior to leaving the location where the wipe samples are being taken.

5.3.6 Health and Safety Dust Monitoring

Site dust monitoring activities will include on-site health and safety sampling for worker protection. This program will include on-site real-time dust monitoring, as described in Section 7.0 of the SHSP. The dust monitoring will be designed to maintain dust levels below 15 mg/m^3. This level has been calculated based on the highest known concentration of PCBs in soil at the site of 13,200 ppm, using a 2.5 factor of safety and the PEL for PCBs of 0.5 mg/m^3 in the following formula:

$$\text{Dust Action Level} = \frac{(1E+6)(\text{Exposure Limit})}{(\text{Concentration mg/kg})(\text{Safety Factor})}$$

5.4 Analytical Methodology

Because the indicator contaminant of interest is PCBs, in-field analyses will be utilized to delineate the extent of contamination in the soil. The transformer vaults and their perimeter areas will be sampled on a minimum 10-foot grid pattern to a depth of approximately 2-feet below the water table. For perimeter vault samples, the minimum depth of borings will be 2-feet below the bottom of the vault (typically a depth of approximately 8- to 10-feet). A boring will be installed in the interior of each vault in the location of the transformer (if known) or where staining is visible. If there is no staining, headspace FID readings will be taken near each corner and the center of the vault to detect the presence of carrier oils that may have contained PCBs. The boring will then be installed at the location of the highest FID reading. Additional borings will be installed outside the vault at each corner and midpoint along the

length at approximate 10-foot intervals in locations where previous sampling data has indicated concentrations less than or equal to 10 ppm PCBs. Where previous data indicates concentrations greater than 10 ppm, the boring location was moved out approximately 5-feet from the original location to attempt to identify the limits of contamination. A second row of borings will be installed 10-feet outward from the initial row. For Building 54, the 10-foot grid along the waterside will be extended to the limits of low tide. Proposed sample locations are depicted in Figures 2-2 and 2-2a.

Samples will be collected from these borings at 1-foot intervals to approximately 2-feet below the water table or 2-feet below the bottom of the vault, whichever is deeper. The surface sample at each boring location will be analyzed in the field via the immunoassay method. If the groundwater-level sample from the interior of the vault shows PCB concentrations <10 ppm, the dry samples from one foot or greater above the water table will be analyzed in the field via the immunoassay method from the surface down, until two consecutive samples meet the minimum concentration of 10 ppm. If the groundwater-level sample from the interior of the vault shows PCB concentrations >10 ppm, then the groundwater-level sample from each perimeter boring location will be sent to the lab for analysis prior to analyzing the remaining samples. A rapid turnaround (48-hour) analysis will be used for these samples to accommodate the holding time limitations for any additional samples that may require fixed laboratory analysis. If the lab analysis shows PCB concentrations >10 ppm, samples will be analyzed above and below the groundwater level. Wet samples or samples within one foot of the water table will be sent off site for analysis. When two results in each direction (above and below the groundwater table) are less than or equal to 10 ppm, the area will be considered completely delineated.

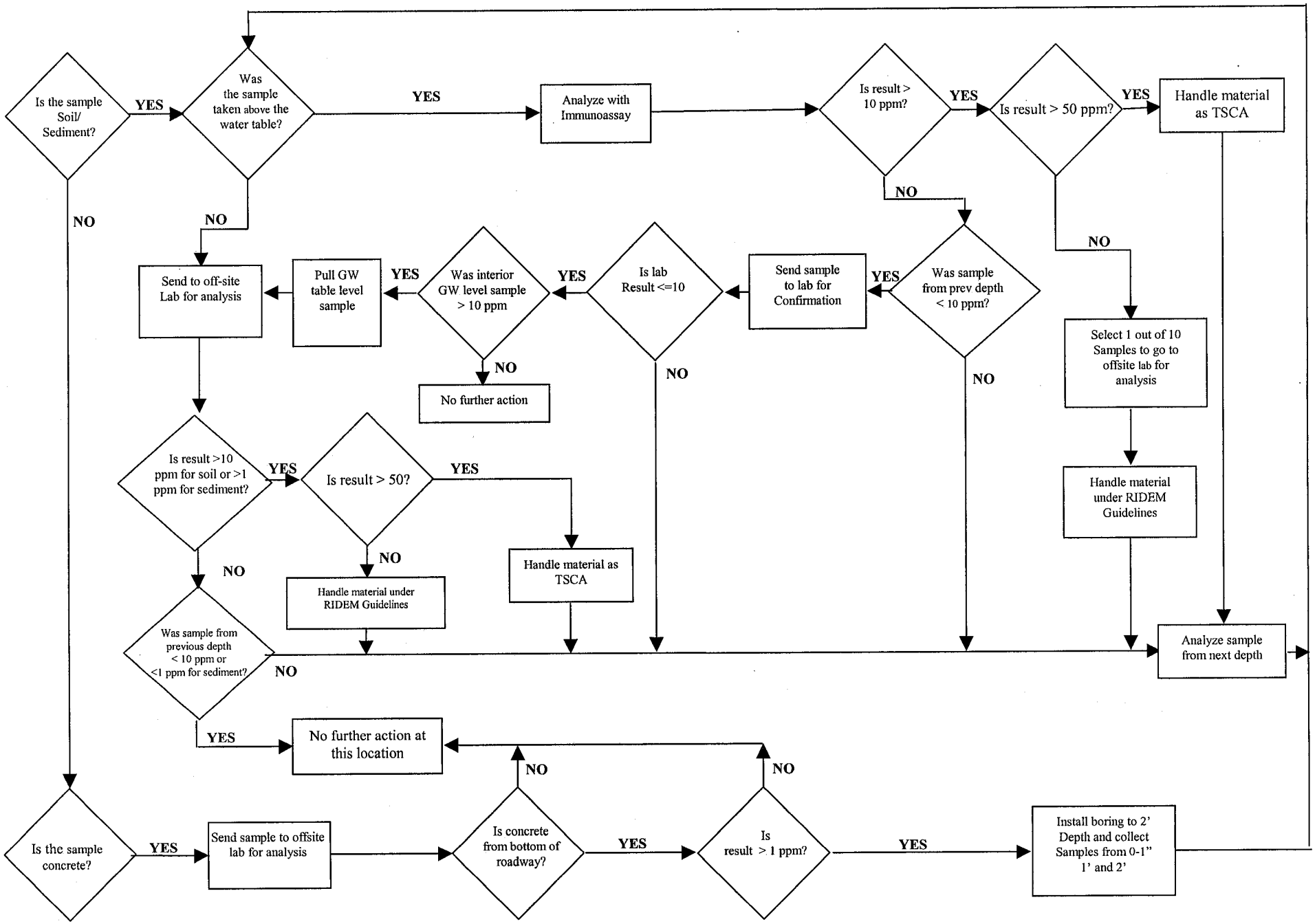
If the result at any depth is greater than 10 ppm, an additional boring will be installed another ten (or more as needed) feet out from the original boring location to the depth at which the previous boring yielded a result of less than or equal to 10 ppm. Another series of vertical samples will be collected for immunoassay or fixed lab analyses. This sequence will be repeated until the results for all depths are less than or equal to 10 ppm. If immunoassay results indicate that the soil contains less than or equal to 10 ppm, the samples will be sent to the fixed laboratory to confirm the extent of contamination has been accurately delineated.

To assist the field staff in determining the type of analyses to be performed at each location, a decision tree has been developed. This is included as Figure 5-1 of this plan.

5.4.1 Soil Sample Immunoassay Field Screening

Samples will be screened on-site using immunoassay testing procedures in accordance with EPA Method 4020 modified. Table 5-1 summarizes the number of field and quality control samples anticipated to be taken in each area. The assay uses antibodies and enzyme conjugate systems to provide the basis for the colorimetric test that is inversely proportional to the amount of analyte in the sample. The detection limit for Arochlor 1260 is 0.5 ppm. Soil samples will be analyzed by immunoassay and compared against a negative control, a 10 ppm and 25 ppm standard. The data is semi-quantitative and indicates whether the samples are greater or less than the standards (giving a concentration range). It is anticipated that a SDI Rapid Assay Field Test Kit for PCBs will be used for this screening. In that case, the data will be roughly quantitative, yielding specific concentrations to one significant digit when the sample concentration is within the calibrated range. Samples outside of the calibrated range will be reported as greater than or less than the specified limits.

Figure 5-1 Decision Tree



As discussed in the sample collection section, samples collected within one foot of the water table will not be screened via immunoassay testing but will be sent off site to the fixed laboratory.

To allow for validation of the field screening method, approximately 10% of the samples will be split and sent to the off-site laboratory. The samples selected will be biased to include soil with detectable concentrations of PCBs across the range of contamination at the site. This will provide a means of comparing the field screening to off-site laboratory data. The field screening data will be plotted against the off-site analytical data and a linear regression performed. Outliers will be individually examined to determine if they are from a different population. For example, the concentration, moisture contents, and sample source will be investigated. If the data is determined to be from a different population, it will be eliminated from the regression, or, if enough data from the second population exists, an additional regression will be performed on that data. If R^2 is greater than 0.9, the field data will be considered representative.

5.4.2 Soil, Sediment and Concrete Sample Laboratory Analysis

Samples will be analyzed at Analytics Environmental Laboratory of Portsmouth, New Hampshire. Table 5-1 summarizes the analytical methods that will be used by the laboratory. Table 5-3 summarizes the target analyte list and reporting limits for each of the methods. All analyses will be performed in strict accordance with the methods specified. Reporting limits are consistent with the CLP limits where applicable.

The solid Practical Quantitation Limits (PQLs) listed in Table 5-3 have assumed a percent solids value and have a note that the values will change based on actual percent solids. As shown in Table 5-3, the laboratory PQLs are below the PALs even when the percent solids are low. The extraction method specified 3550B uses a drying step with anhydrous Sodium Sulfate to adsorb moisture present in the sample. For very wet samples, additional Sodium Sulfate is added with mixing until a "free-flowing powder" is achieved. This step eliminates any significant problems with high percent moisture samples.

5.5 Quality Control Sample Requirements

QA/QC samples are analyzed for the purpose of assessing the quality of the sampling effort and of the analytical data. QA/QC samples will be collected as split samples. The descriptions below include sampling methodologies, sample frequencies, and the purpose of the samples.

5.5.1 QA/QC Split Samples

QA/QC duplicate samples are grab or composite samples, collected separately, that equally represent a medium at a given location and time. QA/QC split samples will be collected at a frequency of 5% per sample matrix. A minimum of one QA/QC split sample will be collected for all sampling events, except for waste characterization sampling. Each QA/QC split sample will be assigned a designated field identification number similar to the other samples collected during that sampling event. There will be no reference to the fact that the sample is a QA/QC split sample. In this manner, the sample will be submitted to the testing laboratory and analyzed as a "blind" duplicate which can then be used to assess the quality of the analytical data generated by the field screening.

Table 5-3 A

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
VOCs in Soil

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (% R)	Completeness (% valid Data)
MATRIX: SOIL PQL's assume 100% solids. PQL's will vary based on percent solids.								
VOCs: Medium Level (Methanol Preserved) 5ml purges								
Dichlorodifluoromethane	5035/8260B		µg/kg	100	24.5	70-130	60-140	
Chloromethane	5035/8260B		µg/kg	100	23.3	70-130	60-140	
Vinyl Chloride	5035/8260B		µg/kg	100	21.2	70-130	60-140	
Bromomethane	5035/8260B		µg/kg	100	27.7	70-130	60-140	
Chloroethane	5035/8260B		µg/kg	100	30.1	70-130	60-140	
t-Butyl alcohol	5035/8260B		µg/kg	4000	654.8	70-130	60-140	
Trichlorofluoromethane	5035/8260B		µg/kg	100	19.9	70-130	60-140	
Diethyl ether	5035/8260B		µg/kg	100	17.5	70-130	60-140	
Acetone	5035/8260B		µg/kg	500	92.2	70-130	60-140	
1,1-Dichloroethene	5035/8260B		µg/kg	75	13.7	70-130	60-140	
Di-isopropyl ether	5035/8260B		µg/kg	100	30.1	70-130	60-140	
Methylene Chloride	5035/8260B		µg/kg	250	32.6	70-130	60-140	
Carbon Disulfide	5035/8260B		µg/kg	100	18.8	70-130	60-140	
Methyl-tert-butyl ether	5035/8260B		µg/kg	100	18	70-130	60-140	
trans-1,2-Dichloroethene	5035/8260B		µg/kg	100	18.1	70-130	60-140	
1,1-Dichloroethane	5035/8260B		µg/kg	100	29	70-130	60-140	
Methyl ethyl ketone	5035/8260B		µg/kg	500	108.4	70-130	60-140	
Ethyl t-butyl ether	5035/8260B		µg/kg	100	13	70-130	60-140	
2,2-Dichloropropane	5035/8260B		µg/kg	100	19.5	70-130	60-140	
cis-1,2-Dichloroethene	5035/8260B		µg/kg	100	18.3	70-130	60-140	
t-Amyl methyl ether	5035/8260B		µg/kg	100	13.3	70-130	60-140	
Chloroform	5035/8260B		µg/kg	75	18.4	70-130	60-140	
Bromochloromethane	5035/8260B		µg/kg	100	17.5	70-130	60-140	
Tetrahydrofuran	5035/8260B		µg/kg	500	64.3	70-130	60-140	
1,1,1-Trichloroethane	5035/8260B		µg/kg	100	13.9	70-130	60-140	
1,1-Dichloropropene	5035/8260B		µg/kg	100	18.2	70-130	60-140	
Carbon Tetrachloride	5035/8260B		µg/kg	100	16.4	70-130	60-140	
1,2-Dichloroethane	5035/8260B		µg/kg	75	18.9	70-130	60-140	
Benzene	5035/8260B		µg/kg	100	17.4	70-130	60-140	
Trichloroethene	5035/8260B		µg/kg	100	17.7	70-130	60-140	
1,2-Dichloropropane	5035/8260B		µg/kg	75	12.6	70-130	60-140	
Bromodichloromethane	5035/8260B		µg/kg	75	15.4	70-130	60-140	
Dibromomethane	5035/8260B		µg/kg	100	14.8	70-130	60-140	
2-Hexanone	5035/8260B		µg/kg	500	109.3	70-130	60-140	
Methyl isobutyl ketone	5035/8260B		µg/kg	500	114.2	70-130	60-140	
cis-1,3-Dichloropropene	5035/8260B		µg/kg	100	16.5	70-130	60-140	
Toluene	5035/8260B		µg/kg	100	14.3	70-130	60-140	
trans-1,3-Dichloropropene	5035/8260B		µg/kg	100	16.0	70-130	60-140	
1,1,2-Trichloroethane	5035/8260B		µg/kg	75	14.8	70-130	60-140	
1,3-Dichloropropane	5035/8260B		µg/kg	100	15.3	70-130	60-140	
Tetrachloroethene	5035/8260B		µg/kg	100	14.5	70-130	60-140	
Dibromochloromethane	5035/8260B		µg/kg	75	15.8	70-130	60-140	
1,2-Dibromoethane	5035/8260B		µg/kg	75	11.1	70-130	60-140	
Chlorobenzene	5035/8260B		µg/kg	100	16.8	70-130	60-140	
1,1,1,2-Tetrachloroethane	5035/8260B		µg/kg	100	20.3	70-130	60-140	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (% R)	Completeness (% valid Data)
Ethylbenzene	5035/8260B		µg/kg	100	18.9	70-130	60-140	
m,p-Xylene	5035/8260B		µg/kg	100	11.6	70-130	60-140	
o-Xylene	5035/8260B		µg/kg	100	16.7	70-130	60-140	
Styrene	5035/8260B		µg/kg	100	18.8	70-130	60-140	
Bromoform	5035/8260B		µg/kg	75	15.2	70-130	60-140	
Isopropylbenzene	5035/8260B		µg/kg	100	17.1	70-130	60-140	
1,1,2,2-Tetrachloroethane	5035/8260B		µg/kg	75	13.4	70-130	60-140	
1,2,3-Trichloropropane	5035/8260B		µg/kg	100	10.8	70-130	60-140	
trans-1,4-Dichloro-2-butene	5035/8260B		µg/kg	100	22.8	70-130	60-140	
n-Propylbenzene	5035/8260B		µg/kg	100	19.2	70-130	60-140	
Bromobenzene	5035/8260B		µg/kg	100	23.7	70-130	60-140	
1,3,5-Trimethylbenzene	5035/8260B		µg/kg	100	22.5	70-130	60-140	
2-Chlorotoluene	5035/8260B		µg/kg	100	20.9	70-130	60-140	
4-Chlorotoluene	5035/8260B		µg/kg	100	17.7	70-130	60-140	
tert-butylbenzene	5035/8260B		µg/kg	100	16.5	70-130	60-140	
1,2,4-Trimethylbenzene	5035/8260B		µg/kg	100	19.8	70-130	60-140	
Pentachloroethane	5035/8260B		µg/kg	100	16.6	70-130	60-140	
sec-butylbenzene	5035/8260B		µg/kg	100	15.6	70-130	60-140	
p-isopropyltoluene	5035/8260B		µg/kg	100	18.7	70-130	60-140	
1,3-Dichlorobenzene	5035/8260B		µg/kg	100	19.1	70-130	60-140	
1,4-Dichlorobenzene	5035/8260B		µg/kg	100	16.3	70-130	60-140	
n-butylbenzene	5035/8260B		µg/kg	100	23.3	70-130	60-140	
1,2-Dichlorobenzene	5035/8260B		µg/kg	100	17.2	70-130	60-140	
1,2-Dibromo-3-chloropropane	5035/8260B		µg/kg	100	28.2	70-130	60-140	
1,2,4-Trichlorobenzene	5035/8260B		µg/kg	100	20.7	70-130	60-140	
Hexachlorobutadiene	5035/8260B		µg/kg	100	23.3	70-130	60-140	
Naphthalene	5035/8260B		µg/kg	100	14.1	70-130	60-140	
1,2,3-Trichlorobenzene	5035/8260B		µg/kg	100	22.7	70-130	60-140	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 5-3 B

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
VOCs in Groundwater

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% Valid Data)
MATRIX: GROUNDWATER								
VOCs 5ml purges								
Dichlorodifluoromethane	5030B/8260B		µg/l	2	0.11	70-130	70-130	
Chloromethane	5030B/8260B		µg/l	2	0.21	70-130	70-130	
Vinyl Chloride	5030B/8260B		µg/l	2	0.31	70-130	70-130	
Bromomethane	5030B/8260B		µg/l	2	0.34	70-130	70-130	
Chloroethane	5030B/8260B		µg/l	2	0.24	70-130	70-130	
t-Butyl alcohol	5030B/8260B		µg/l	80	25.06	70-130	70-130	
Trichlorofluoromethane	5030B/8260B		µg/l	2	0.21	70-130	70-130	
Diethyl ether	5030B/8260B		µg/l	2	0.38	70-130	70-130	
Acetone	5030B/8260B		µg/l	10	3.23	70-130	70-130	
1,1-Dichloroethene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Di-isopropyl ether	5030B/8260B		µg/l	2	0.27	70-130	70-130	
Methylene Chloride	5030B/8260B		µg/l	5	1.30	70-130	70-130	
Carbon Disulfide	5030B/8260B		µg/l	2	0.22	70-130	70-130	
Methyl-tert-butyl ether	5030B/8260B		µg/l	2	0.42	70-130	70-130	
trans-1,2-Dichloroethene	5030B/8260B		µg/l	2	0.26	70-130	70-130	
1,1-Dichloroethane	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Methyl ethyl ketone	5030B/8260B		µg/l	10	1.68	70-130	70-130	
Ethyl t-butyl ether	5030B/8260B		µg/l	2	0.19	70-130	70-130	
2,2-Dichloropropane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
cis-1,2-Dichloroethene	5030B/8260B		µg/l	2	0.20	70-130	70-130	
t-Amyl methyl ether	5030B/8260B		µg/l	2	0.18	70-130	70-130	
Chloroform	5030B/8260B		µg/l	2	0.14	70-130	70-130	
Bromochloromethane	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Tetrahydrofuran	5030B/8260B		µg/l	2	0.39	70-130	70-130	
1,1,1-Trichloroethane	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,1-Dichloropropene	5030B/8260B		µg/l	2	0.10	70-130	70-130	
Carbon Tetrachloride	5030B/8260B		µg/l	2	0.23	70-130	70-130	
1,2-Dichloroethane	5030B/8260B		µg/l	2	0.23	70-130	70-130	
Benzene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Trichloroethene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
1,2-Dichloropropane	5030B/8260B		µg/l	2	0.20	70-130	70-130	
Bromodichloromethane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
Dibromomethane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
2-Hexanone	5030B/8260B		µg/l	10	2.42	70-130	70-130	
Methyl isobutyl ketone	5030B/8260B		µg/l	10	0.89	70-130	70-130	
cis-1,3-Dichloropropene	5030B/8260B		µg/l	2	0.14	70-130	70-130	
Toluene	5030B/8260B		µg/l	2	0.21	70-130	70-130	
trans-1,3-Dichloropropene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
1,1,2-Trichloroethane	5030B/8260B		µg/l	2	0.13	70-130	70-130	
1,3-Dichloropropane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
Tetrachloroethene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
Dibromochloromethane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
1,2-Dibromoethane	5030B/8260B		µg/l	2	0.21	70-130	70-130	
Chlorobenzene	5030B/8260B		µg/l	2	0.10	70-130	70-130	
1,1,1,2-Tetrachloroethane	5030B/8260B		µg/l	2	0.16	70-130	70-130	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% Valid Data)
Ethylbenzene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
m,p-Xylene	5030B/8260B		µg/l	2	0.67	70-130	70-130	
o-Xylene	5030B/8260B		µg/l	2	0.15	70-130	70-130	
Styrene	5030B/8260B		µg/l	2	0.36	70-130	70-130	
Bromoform	5030B/8260B		µg/l	2	0.11	70-130	70-130	
Isopropylbenzene	5030B/8260B		µg/l	2	0.25	70-130	70-130	
1,1,2,2-Tetrachloroethane	5030B/8260B		µg/l	2	0.29	70-130	70-130	
1,2,3-Trichloropropane	5030B/8260B		µg/l	2	0.32	70-130	70-130	
trans-1,4-Dichloro-2-butene	5030B/8260B		µg/l	2	0.67	70-130	70-130	
n-Propylbenzene	5030B/8260B		µg/l	2	0.13	70-130	70-130	
Bromobenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,3,5-Trimethylbenzene	5030B/8260B		µg/l	2	0.14	70-130	70-130	
2-Chlorotoluene	5030B/8260B		µg/l	2	0.11	70-130	70-130	
4-Chlorotoluene	5030B/8260B		µg/l	2	0.11	70-130	70-130	
tert-butylbenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,2,4-Trimethylbenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
Pentachloroethane	5030B/8260B		µg/l	2	0.28	70-130	70-130	
sec-butylbenzene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
p-isopropyltoluene	5030B/8260B		µg/l	2	0.13	70-130	70-130	
1,3-Dichlorobenzene	5030B/8260B		µg/l	2	0.15	70-130	70-130	
1,4-Dichlorobenzene	5030B/8260B		µg/l	2	0.06	70-130	70-130	
n-butylbenzene	5030B/8260B		µg/l	2	0.10	70-130	70-130	
1,2-Dichlorobenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,2-Dibromo-3-chloropropane	5030B/8260B		µg/l	2	0.25	70-130	70-130	
1,2,4-Trichlorobenzene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
Hexachlorobutadiene	5030B/8260B		µg/l	2	0.31	70-130	70-130	
Naphthalene	5030B/8260B		µg/l	2	0.51	70-130	70-130	
1,2,3-Trichlorobenzene	5030B/8260B		µg/l	2	0.32	70-130	70-130	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 5-3 C

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
PCBs

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% Valid Data)
MATRIX: GROUNDWATER								
PCBS								
PCB 1016	3510C/8082		µg/l	0.5	0.05	67-121	67-121	
PCB 1221	3510C/8082		µg/l	0.5	0.04	NA	NA	
PCB 1232	3510C/8082		µg/l	0.5	0.05	NA	NA	
PCB 1242	3510C/8082		µg/l	0.5	0.05	NA	NA	
PCB 1248	3510C/8082		µg/l	0.5	0.04	NA	NA	
PCB 1254	3510C/8082		µg/l	0.5	0.07	NA	NA	
PCB 1260	3510C/8082		µg/l	0.5	0.06	80-146	80-146	
MATRIX: Wipe								
PCBS								
PCB 1016	3550B/8082		µg/wipe	0.5	0.10	52-127	52-127	
PCB 1221	3550B/8082		µg/wipe	0.5	0.20	NA	NA	
PCB 1232	3550B/8082		µg/wipe	0.5	0.10	NA	NA	
PCB 1242	3550B/8082		µg/wipe	0.5	0.10	NA	NA	
PCB 1248	3550B/8082		µg/wipe	0.5	0.10	NA	NA	
PCB 1254	3550B/8082		µg/wipe	0.5	0.20	NA	NA	
PCB 1260	3550B/8082		µg/wipe	0.5	0.20	68-159	68-159	
MATRIX: Solids/Concrete PQL's assume 100% solids. PQL's will vary based on percent solid.								
PCBS								
PCB 1016	3550B/8082		µg/kg	15	1.65	52-127	52-127	
PCB 1221	3550B/8082		µg/kg	15	5.33	NA	NA	
PCB 1232	3550B/8082		µg/kg	15	1.84	NA	NA	
PCB 1242	3550B/8082		µg/kg	15	1.65	NA	NA	
PCB 1248	3550B/8082		µg/kg	15	2.77	NA	NA	
PCB 1254	3550B/8082		µg/kg	15	5.58	NA	NA	
PCB 1260	3550B/8082		µg/kg	15	1.43	68-159	68-159	
MATRIX: Sediments PQL's assume 30% solids. PQL's will vary based on percent solid.								
PCBS								
PCB 1016	3550B/8082		µg/kg	50	1.65	52-127	52-127	
PCB 1221	3550B/8082		µg/kg	50	5.33	NA	NA	
PCB 1232	3550B/8082		µg/kg	50	1.84	NA	NA	
PCB 1242	3550B/8082		µg/kg	50	1.65	NA	NA	
PCB 1248	3550B/8082		µg/kg	50	2.77	NA	NA	
PCB 1254	3550B/8082		µg/kg	50	5.58	NA	NA	
PCB 1260	3550B/8082		µg/kg	50	1.43	68-159	68-159	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 5-3 D

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
SVOCs in Soil

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% Valid Data)
MATRIX: SOIL PQL's assume 100% solids. PQL's will vary based on percent solid.								
SVOCs:								
Pyridine	3550B/8270C		µg/kg	300	17.1	NA	NA	
N-nitrosodimethylamine	3550B/8270C		µg/kg	300	11.2	NA	NA	
Aniline	3550B/8270C		µg/kg	300	11.4	NA	NA	
Phenol	3550B/8270C		µg/kg	300	22.5	52-86	52-86	
2-Chlorophenol	3550B/8270C		µg/kg	300	19.8	51-84	51-84	
bis(2-chloroethyl) ether	3550B/8270C		µg/kg	300	10.4	NA	NA	
1,3-Dichlorobenzene	3550B/8270C		µg/kg	300	10.8	NA	NA	
1,4-Dichlorobenzene	3550B/8270C		µg/kg	300	10.4	NA	NA	
1,2-Dichlorobenzene	3550B/8270C		µg/kg	300	12.4	NA	NA	
Benzyl Alcohol	3550B/8270C		µg/kg	300	10.3	NA	NA	
2-Methylphenol	3550B/8270C		µg/kg	300	22.2	NA	NA	
bis(2-chloroisopropyl)ether	3550B/8270C		µg/kg	300	10.4	NA	NA	
3&4-Methylphenol	3550B/8270C		µg/kg	300	29.6	NA	NA	
Hexachloroethane	3550B/8270C		µg/kg	300	14	NA	NA	
N-nitroso-di-n-propylamine	3550B/8270C		µg/kg	300	8.9	21-105	21-105	
Nitrobenzene	3550B/8270C		µg/kg	300	13	NA	NA	
Isophorone	3550B/8270C		µg/kg	300	7.5	NA	NA	
2-Nitrophenol	3550B/8270C		µg/kg	300	24	NA	NA	
2,4-Dimethylphenol	3550B/8270C		µg/kg	300	20.2	NA	NA	
Bis(2-chloroethoxy)methane	3550B/8270C		µg/kg	300	7.8	NA	NA	
2,4-Dichlorophenol	3550B/8270C		µg/kg	300	20.3	NA	NA	
Benzoic Acid	3550B/8270C		µg/kg	300	23.0	NA	NA	
1,2,4-Trichlorobenzene	3550B/8270C		µg/kg	300	10.0	42-79	42-79	
Naphthalene	3550B/8270C		µg/kg	300	9.2	NA	NA	
2,6-Dichlorophenol	3550B/8270C		µg/kg	300	16.9	NA	NA	
4-Chloroaniline	3550B/8270C		µg/kg	300	10.2	NA	NA	
Hexachlorobutadiene	3550B/8270C		µg/kg	300	10.7	NA	NA	
4-Chloro-3-methylphenol	3550B/8270C		µg/kg	300	23.7	60-94	60-94	
2-Methylnaphthalene	3550B/8270C		µg/kg	300	14.5	NA	NA	
Hexachlorocyclopentadiene	3550B/8270C		µg/kg	300	19.0	NA	NA	
2,4,6-Trichlorophenol	3550B/8270C		µg/kg	300	77.3	NA	NA	
2,4,5-Trichlorophenol	3550B/8270C		µg/kg	300	36.5	NA	NA	
2-Chloronaphthalene	3550B/8270C		µg/kg	300	12.0	NA	NA	
2-Nitroaniline	3550B/8270C		µg/kg	300	16.3	NA	NA	
2,6-Dinitrotoluene	3550B/8270C		µg/kg	300	10.0	NA	NA	
Dimethyl Phthalate	3550B/8270C		µg/kg	300	10.3	NA	NA	
Acenaphthylene	3550B/8270C		µg/kg	300	8.5	NA	NA	
3-Nitroaniline	3550B/8270C		µg/kg	300	11.5	NA	NA	
Acenaphthene	3550B/8270C		µg/kg	300	11.9	49-91	49-91	
2,4-Dinitrophenol	3550B/8270C		µg/kg	300	29.8	NA	NA	
Dibenzofuran	3550B/8270C		µg/kg	300	10.8	NA	NA	
4-Nitrophenol	3550B/8270C		µg/kg	300	78.8	64-105	64-105	
2,4-Dinitrotoluene	3550B/8270C		µg/kg	300	11.0	50-104	50-104	
2,3,4,6-Tetrachlorophenol	3550B/8270C		µg/kg	300	88.3	NA	NA	
Fluorene	3550B/8270C		µg/kg	300	12.8	NA	NA	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% Valid Data)
Diethyl Phthalate	3550B/8270C		µg/kg	300	10.2	NA	NA	
4-Chlorophenyl phenyl ether	3550B/8270C		µg/kg	300	10.0	NA	NA	
4-Nitroaniline	3550B/8270C		µg/kg	300	9.4	NA	NA	
4,6-Dinitro-2-methylphenol	3550B/8270C		µg/kg	300	16.3	NA	NA	
n-nitrosodiphenylamine	3550B/8270C		µg/kg	300	9.0	NA	NA	
Azobenzene	3550B/8270C		µg/kg	300	9.7	NA	NA	
4-Bromophenyl phenyl ether	3550B/8270C		µg/kg	300	12.8	NA	NA	
Hexachlorobenzene	3550B/8270C		µg/kg	300	10.9	NA	NA	
Pentachlorophenol	3550B/8270C		µg/kg	300	17.1	47-108	47-108	
Phenanthrene	3550B/8270C		µg/kg	300	9.7	NA	NA	
Anthracene	3550B/8270C		µg/kg	300	12.4	NA	NA	
Carbazole	3550B/8270C		µg/kg	300	43.3	NA	NA	
Di-n-butyl phthalate	3550B/8270C		µg/kg	300	10.9	NA	NA	
Fluoranthene	3550B/8270C		µg/kg	300	8.4	NA	NA	
Benzidine	3550B/8270C		µg/kg	300	392.2	NA	NA	
Pyrene	3550B/8270C		µg/kg	300	7.5	52-103	52-103	
Butyl benzyl phthalate	3550B/8270C		µg/kg	300	12.3	NA	NA	
Benzo[a]anthracene	3550B/8270C		µg/kg	300	7.8	NA	NA	
Chrysene	3550B/8270C		µg/kg	300	11.9	NA	NA	
3,3'-Dichlorobenzidine	3550B/8270C		µg/kg	300	35.7	NA	NA	
Bis(2-ethylhexyl) phthalate	3550B/8270C		µg/kg	300	18.1	NA	NA	
di-n-octyl-phthalate	3550B/8270C		µg/kg	300	43.6	NA	NA	
Benzo[b]fluoranthene	3550B/8270C		µg/kg	300	36.8	NA	NA	
Benzo[k]fluoranthene	3550B/8270C		µg/kg	300	11.4	NA	NA	
Benzo[a]pyrene	3550B/8270C		µg/kg	300	7.9	NA	NA	
Indeno[1,2,3-cd]pyrene	3550B/8270C		µg/kg	300	6.7	NA	NA	
Dibenz[a,h]anthracene	3550B/8270C		µg/kg	300	8.6	NA	NA	
Benzo(g,h,i)perylene	3550B/8270C		µg/kg	300	10.9	NA	NA	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 5-3 E

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
SVOCs in Water

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% Valid Data)
MATRIX: AQUEOUS								
SVOCs:								
Pyridine	3510C/8270C		µg/l	2	0.24	NA	NA	
N-nitrosodimethylamine	3510C/8270C		µg/l	2	0.29	NA	NA	
Aniline	3510C/8270C		µg/l	2	0.30	NA	NA	
Phenol	3510C/8270C		µg/l	5	0.30	26-43	26-43	
2-Chlorophenol	3510C/8270C		µg/l	5	0.55	43-91	43-91	
Bis(2-chloroethyl) ether	3510C/8270C		µg/l	2	0.41	NA	NA	
1,3-Dichlorobenzene	3510C/8270C		µg/l	2	0.50	NA	NA	
1,4-Dichlorobenzene	3510C/8270C		µg/l	2	0.44	23-77	23-77	
1,2-Dichlorobenzene	3510C/8270C		µg/l	2	0.41	NA	NA	
Benzyl Alcohol	3510C/8270C		µg/l	5	0.46	NA	NA	
2-Methylphenol	3510C/8270C		µg/l	5	0.68	NA	NA	
Bis(2-chloroisopropyl)ether	3510C/8270C		µg/l	2	0.30	NA	NA	
3&4-Methylphenol	3510C/8270C		µg/l	5	1.02	NA	NA	
Hexachloroethane	3510C/8270C		µg/l	2	0.57	NA	NA	
N-nitroso-di-n-propylamine	3510C/8270C		µg/l	2	0.37	47-91	47-91	
Nitrobenzene	3510C/8270C		µg/l	2	0.28	NA	NA	
Isophorone	3510C/8270C		µg/l	2	0.30	NA	NA	
2-Nitrophenol	3510C/8270C		µg/l	5	0.35	NA	NA	
2,4-Dimethylphenol	3510C/8270C		µg/l	5	0.62	NA	NA	
Bis(2-chloroethoxy)methane	3510C/8270C		µg/l	2	0.27	NA	NA	
2,4-Dichlorophenol	3510C/8270C		µg/l	2	0.76	NA	NA	
Benzoic Acid	3510C/8270C		µg/l	10	1.61	NA	NA	
1,2,4-Trichlorobenzene	3510C/8270C		µg/l	2	0.45	29-81	29-81	
Naphthalene	3510C/8270C		µg/l	2	0.33	NA	NA	
2,6-Dichlorophenol	3510C/8270C		µg/l	5	0.76	NA	NA	
4-Chloroaniline	3510C/8270C		µg/l	2	0.24	NA	NA	
Hexachlorobutadiene	3510C/8270C		µg/l	2	0.34	NA	NA	
4-Chloro-3-methylphenol	3510C/8270C		µg/l	10	0.50	61-99	61-99	
2-Methylnaphthalene	3510C/8270C		µg/l	2	0.28	NA	NA	
Hexachlorocyclopentadiene	3510C/8270C		µg/l	2	0.40	NA	NA	
2,4,6-Trichlorophenol	3510C/8270C		µg/l	5	0.44	NA	NA	
2,4,5-Trichlorophenol	3510C/8270C		µg/l	5	0.63	NA	NA	
2-Chloronaphthalene	3510C/8270C		µg/l	2	0.28	NA	NA	
2-Nitroaniline	3510C/8270C		µg/l	2	0.34	NA	NA	
2,6-Dinitrotoluene	3510C/8270C		µg/l	2	0.23	NA	NA	
Dimethyl Phthalate	3510C/8270C		µg/l	2	0.75	NA	NA	
Acenaphthylene	3510C/8270C		µg/l	2	0.28	NA	NA	
3-Nitroaniline	3510C/8270C		µg/l	2	0.21	NA	NA	
Acenaphthene	3510C/8270C		µg/l	2	0.36	49-91	49-91	
2,4-Dinitrophenol	3510C/8270C		µg/l	5	2.43	NA	NA	
Dibenzofuran	3510C/8270C		µg/l	2	0.26	NA	NA	
4-Nitrophenol	3510C/8270C		µg/l	5	1.13	25-55	25-55	
2,4-Dinitrotoluene	3510C/8270C		µg/l	2	0.37	62-104	62-104	
2,3,4,6-Tetrachlorophenol	3510C/8270C		µg/l	5	0.41	NA	NA	
Fluorene	3510C/8270C		µg/l	2	0.26	NA	NA	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% Valid Data)
Diethyl Phthalate	3510C/8270C		µg/l	2	0.43	NA	NA	
4-Chlorophenyl phenyl ether	3510C/8270C		µg/l	2	0.14	NA	NA	
4-Nitroaniline	3510C/8270C		µg/l	2	0.14	NA	NA	
4,6-Dinitro-2-methylphenol	3510C/8270C		µg/l	5	1.68	NA	NA	
n-nitrosodiphenylamine	3510C/8270C		µg/l	2	0.21	NA	NA	
Azobenzene	3510C/8270C		µg/l	2	0.46	NA	NA	
4-Bromophenyl phenyl ether	3510C/8270C		µg/l	2	0.27	NA	NA	
Hexachlorobenzene	3510C/8270C		µg/l	2	0.32	NA	NA	
Pentachlorophenol	3510C/8270C		µg/l	10	0.62	56-110	56-110	
Phenanthrene	3510C/8270C		µg/l	2	0.27	NA	NA	
Anthracene	3510C/8270C		µg/l	2	0.38	NA	NA	
Carbazole	3510C/8270C		µg/l	2	0.28	NA	NA	
Di-n-butyl phthalate	3510C/8270C		µg/l	2	0.33	NA	NA	
Fluoranthene	3510C/8270C		µg/l	2	0.26	NA	NA	
Benzidine	3510C/8270C		µg/l	20	19.40	NA	NA	
Pyrene	3510C/8270C		µg/l	2	0.38	56-110	56-110	
Butyl benzyl phthalate	3510C/8270C		µg/l	2	0.78	NA	NA	
Benzo[a]anthracene	3510C/8270C		µg/l	2	0.20	NA	NA	
Chrysene	3510C/8270C		µg/l	2	0.31	NA	NA	
3,3'-Dichlorobenzidine	3510C/8270C		µg/l	20	1.36	NA	NA	
Bis(2-ethylhexyl) phthalate	3510C/8270C		µg/l	2	0.33	NA	NA	
di-n-octyl-phthalate	3510C/8270C		µg/l	2	0.45	NA	NA	
Benzo[b]fluoranthene	3510C/8270C		µg/l	2	0.25	NA	NA	
Benzo[k]fluoranthene	3510C/8270C		µg/l	2	0.50	NA	NA	
Benzo[a]pyrene	3510C/8270C		µg/l	2	0.24	NA	NA	
Indeno[1,2,3-cd]pyrene	3510C/8270C		µg/l	2	0.33	NA	NA	
Dibenz[a,h]anthracene	3510C/8270C		µg/l	2	0.22	NA	NA	
Benzo(g,h,i)perylene	3510C/8270C		µg/l	2	0.30	NA	NA	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Duplicates will be compared to a +/-50% Relative Percent Difference (RPD) as recommended in EPA guidance, according to the following formula:

$$+/-50\%RPD = \frac{V^1 - V^2}{(V^1 + V^2)/2} * 100$$

where: V^1 = Field data value
V^2 = Analytical laboratory data value

5.6 Equipment Decontamination Procedures

For the analytical sampling programs, only disposable sampling equipment will be used. Therefore, no sampling equipment decontamination procedures will be required.

5.7 Sample Identification, Chain of Custody, Packing, and Shipping

5.7.1 Sample Identification and Labeling

Sample Identification

The sample identification system that will be used for this project will assign a unique sample identifier to each sample collected. Data management will be consistent with this sample identification system. The protocols for assigning field sample numbers are described below. Each sample collected will have its own identifier, which will apply for the duration of the project. The sample identifier will consist of an alphanumeric code that will identify the site designation, sample location, sample type, and sample number. The QC sample identifier will also consist of an alphanumeric code that will identify the QC sample designation, sample location, and sample number (if applicable). Note: all sample identifiers and their corresponding locations will be carefully logged in the field notebook and may be identified on figures or drawings.

Site designation:	GI	Gould Island
Sample Location:	B53	Building 53
	B56	Building 56
	B59	Building 59
	B60	Building 60
	B61	Building 61
	RD	Concrete Roadway
Sample types:	B#	Boring Number
	WP	Wipe Sample
	CG	Contaminated Groundwater Sample
QC sample designations:	SS	Split Sample

Examples: **Soil/Sediment Sample**

Identifier:	GI-B53-B1-1		
where:	GI	=	Gould Island
	B53	=	Building 53
	B1	=	Boring No. 1 Sample
	1	=	One foot depth

Concrete Roadway Sample

Identifier:	GI-RD-B1-1		
where:	GI	=	Gould Island
	RD	=	Concrete Roadway
	B1	=	Boring No. 1 Sample
	1	=	One foot depth

Contaminated Groundwater Sample

Identifier:	GI-CG-1		
where:	GI	=	Gould Island
	CW	=	Contaminated Groundwater
	1	=	Sample Number

QC/QA Sample

Identifier:	GI-B53-SS-1		
where:	GI	=	Gould Island
	B53	=	Building 53
	SS	=	Split QC/QA Sample
	1	=	Sample Number

Sample Labeling

Sample labels will be completed by field personnel in indelible ink. Labels will include the project identification, sample identification, date and time of collection, sampler's initials, sample matrix, type of sample (grab or composite), analyses to be performed, and preservative used (if applicable).

5.7.2 Sample Chain of Custody

To maintain and document sample possession, COC procedures will be implemented. These procedures are necessary to insure the integrity of samples from the time of collection through data reporting. The COC protocol provides the ability to trace possession and handling of samples. A sample is considered under custody if it is/was:

- In a person's possession;
- In a person's view after being in possession;
- In a person's possession and locked up; or
- In a designated secure area.

Personnel collecting samples are responsible for the care and integrity of those samples until they are properly transferred or dispatched. Therefore, the number of people handling a sample will be kept to a minimum.

COC records (see Appendix A) will be completed by the sampler and shall accompany the samples at all times. The following information shall be indicated on the COC record:

- Project identification;
- Signature of samplers;
- Sample identification, sample matrix, date and time of collection, grab or composite sample designation, number of containers corresponding to that sample identification,

analyses required, remarks or sample location (if applicable), and preservation method(s);

- Signature of the individual relinquishing the samples; and
- Name of the individual(s) receiving the samples and air bill number, if applicable.

The COC preparer will then check the sample label and COC record for accuracy and completeness.

A copy of all COC records will be maintained in the field by the SQCM.

5.7.3 Sample Tracking

When transferring custody of samples, individuals relinquishing custody and individuals receiving custody will sign, date, and record the time on the COC. When samples are being shipped to the laboratory via courier or shipping company (DHL), the shipping company will be indicated as receiving custody. Upon receipt of shipment at the laboratory, a designated sample custodian will accept custody of the samples and verify that information on the sample labels matches the COC record. Pertinent information on shipment, air bill number, pickup, courier, date, and time will be recorded on the COC. It is then the laboratory's responsibility to maintain logbooks and custody records throughout sample preparation and analysis.

5.7.4 Sample Packing and Shipping

Samples for off-site laboratory analysis will be shipped via DHL or by courier for overnight delivery in waterproof coolers using the procedures outlined below. The samples taken for this project shall be considered low-level or environmental samples for packaging and shipping purposes. The sample packing procedures are as follows:

- After filling out the pertinent information on the sample label, if necessary cover the label with clear tape.
- Place about 3 inches of inert cushioning material, such as vermiculite or bubblepack, in the bottom of the cooler.
- Place containers upright in the cooler in such a way that they will not touch during shipment.
- Put in additional inert packing material to partially the cover sample containers (more than halfway).
- Place ice, sealed in plastic bags, around and on top of the containers. The temperature of the samples must be maintained at or below 4°C (but above 0°C) during shipment to the laboratory.
- Fill cooler with cushioning material.
- Put COC record in a waterproof plastic bag and tape it with masking tape to the inside lid of the cooler.
- Tape the drain shut.
- Secure the lid by taping. Wrap the cooler completely with tape at a minimum of two locations.
- Attached completed shipping label to top of the cooler.
- Affix two signed and dated custody seals on opposite corners of the cooler so that the cooler cannot be opened without breaking the seals.

Prior to packing and shipping, samples will be stored on ice from the time of sample collection.

6.0 ENVIRONMENTAL PROTECTION AND REGULATORY COMPLIANCE

The purpose of this section is to identify the applicable environmental regulatory requirements relevant to the performance of the delineation of contamination, and to establish the appropriate project controls to meet these requirements. This section also identifies Foster Wheeler's environmental compliance procedures and training requirements for this project. The Contract Task Order Manager will be responsible for verifying that all project personnel are aware of the requirements outlined in this Plan.

6.1 Background

The purpose of this delineation is to determine the extent of PCB contamination on the Gould Island Site. The screening criteria used to evaluate the data collected from each of the sampling areas include the RIDEM Division of Site Remediation Residential and Industrial/Commercial Direct Soil Exposure Criteria. These criteria are consistent with the EPA Toxic Substances Control Act (TSCA) criteria.

6.2 Environmental Regulatory Compliance

6.2.1 Characterization Criteria

The interim PCB cleanup goal for the Gould Island Site is the RIDEM Division of Site Remediation industrial/commercial direct exposure criterion of 10 ppm PCBs as set forth under the revised *Rules and Regulations for the Investigation and Remediation of Hazardous Material Releases*. Following completion of the interim removal action, a sitewide closure standard will be developed based on a risk evaluation/assessment. The proposed characterization plan will delineate contamination down to 10 ppm to assist in developing the interim removal action plan. The proposed 10-foot (approximately 3-meter) sampling grid spacing for the soils conforms with the requirements of 40CFR 761.265(a) for the characterization of PCB waste that is not in a container and porous surfaces.

6.2.2 Toxic Substances Control Act (TSCA)

PCB contaminated soil, concrete and sediment at Gould Island will be considered Remediation Waste. Remediation Waste is defined by 40 CFR 761.3 as " waste containing PCBs as a result of a spill, release, or other unauthorized disposal, at the following concentrations: materials disposed of prior to April 18, 1978 that are currently at concentrations ≥50 ppm PCBs, regardless of the concentration of the original spill; materials which are currently at any volume or concentration where the original source was ≥500 ppm PCB beginning on April 18, 1978, or ≥50 ppm PCB beginning on July 2, 1979; and materials which are currently at any concentration, if the PCBs are from a source not authorized for use under this part." The definition includes "soil, rags and other debris generated as a result of any PCB spill cleanup..." Additionally, waste containing greater than 10 ppm PCBs, are regulated by RIDEM under the Remediation Regulations, amended 1996. On-site waste management will be performed in accordance with Section 7.0 of this Work Plan.

6.2.3 Transportation

Regulatory requirements pertaining to waste transportation are addressed in Section 7.0 of this Work Plan.

6.2.4 Water Discharge

Dewatering activities will be required however all water will be stored on-site for T&D off-site and, therefore, a water discharge permit will not be required. Liquids that are generated on site may be regulated under TSCA if they came in contact with PCB-regulated materials and if they contain above 0.5 μg/l PCBs. For this action, liquids will be considered TSCA regulated unless they contain less than 0.5 μg/l PCBs.

6.2.5 Coastal Zone Management

EFANE personnel have previously obtained the necessary consistency determination from the Coastal Resource Management Committee for the work completed to date at Gould Island. Since the current scope is for sampling only, no modification of that determination will be necessary. During preparation of the Work Plan for additional clean up of the site, the determination will be reviewed to ensure that the proposed activities are in compliance with the determination. Eel grass beds have been identified along the westside of Gould Island, however, no sampling or construction activities will take place in this location; therefore, no impact to the eelgrass will occur.

6.3 Release Reporting

An Emergency Response Plan is included in Section 12.0 of the SHSP. The information contained in this section details how Foster Wheeler will address spill control, prevention, and emergency response activities on-site.

6.4 Training and Certification Requirements for Project Personnel

As indicated in Section 13.0 of the SHSP, and pursuant to 29 CFR 1910.120, site personnel performing any activities in an exclusion zone must have received a minimum of 40 hours of initial health and safety training for hazardous waste site operations. In addition to the required initial training, each employee must have received three (3) days of directly supervised on-the-job training appropriate for the activities they will be required to perform. Annual 8-hour refresher training will be required of all hazardous waste site personnel in order to maintain their qualifications for fieldwork. Both the SHSO and the Project Superintendent will also have received 24 hours of on-the-job supervised training, 8-hour supervisor training, and First Aid/CPR with blood borne pathogens training.

Pursuant to 40 CFR 172 Subpart H, Foster Wheeler site personnel involved in shipping material regulated by the U.S. DOT will be trained in U.S. DOT regulations.

6.5 Inspection and Audit Procedures

6.5.1 Inspections by Regulatory Agencies

Site personnel will notify the Project Superintendent if contacted by a regulatory agency for a site inspection. The Project Superintendent will contact the Contract Task Order Manager, who will notify the EFANE Project Manager and the Foster Wheeler Director of Regulatory Compliance. In the event of an unannounced inspection, the EFANE Project Manager and the Foster Wheeler Director of Regulatory Compliance will be contacted immediately.

Additionally, the EFANE Project Manager will provide weekly updates to RIDEM regarding the status of the work, including notification prior to the initiation of field activities.

6.5.2 Inspections by Third Parties

Any outside party requesting access to the site will be referred to the Project Superintendent who will initiate the appropriate notification of the Contract Task Order Manager and the EFANE Project Manager. Foster Wheeler personnel will not grant site access or answer questions for unauthorized personnel.

7.0 WORK APPROACH

The execution of this project will involve various distinct tasks beginning with mobilization and ending with demobilization. These tasks include:

- Mobilization
- Site Preparation
- Soil Sampling Activities
- Removal and Disposal of Waste
- Demobilization

The specific items included under these tasks, or definable features of work, are described below.

7.1 Mobilization

Mobilization for the characterization actions will incorporate the Foster Wheeler office trailer already established at Gould Island and involve the set-up of temporary equipment and material laydown areas at the work sites. All utilities at the work site will be temporary. Electricity will be provided by portable generators, and water will be obtained from Naval Station Newport and transported to the island via barge. Sanitary facilities will be available at the Foster Wheeler office trailer.

7.2 Site Preparation

7.2.1 Site Control

Site preparation will occur in accordance with Section 3.0 of this Work Plan (the Construction Quality Control Plan). Site zones will be used in preparation of intrusive activities and are intended to control the spread of contamination throughout Gould Island. Specific zones (an exclusion zone, a

contamination reduction zone, and a support zone) shall be established for each transformer vault sampling area within the site. Note that the Site Health and Safety Officer (SHSO) may need to adjust the boundaries of the specific zones prior to commencement of work. Should the configurations of the zones change, the changes will be incorporated into the SHSP. A description of each zone is as follows:

- All intrusive activities that may involve exposure to hazardous materials and/or conditions will be contained within an exclusion zone (EZ). This zone includes the area inside each transformer vault and within 5-feet around the perimeter boring locations. For the roadway borings, the EZ will be a 5-foot radius around each boring. The area will be prepared to accommodate all drilling activities, field personnel, and emergency equipment.

- The area just beyond the EZ is called the contamination reduction zone (CRZ). The CRZ contains the contamination reduction corridor (CRC) which provides an area for decontamination of heavy equipment, hand-held equipment, and personnel. The CRC will be used for EZ entry and egress in addition to access for heavy equipment and emergency support services.

- The support zone (SZ) is the uncontaminated area following the CRZ and will be the field support area for most operations. The SZ provides for field team communications and staging for emergency response. Safety equipment will be located in this zone.

As established in the SHSP, the initial level of personal protective equipment (PPE) will be modified Level D for all activities. Level C PPE may be required if action levels indicate the need to upgrade.

7.2.2 Equipment Decontamination

A temporary equipment decontamination pad will be constructed to decontaminate heavy equipment (i.e., the drill rig), as necessary, with a portable water tank or truck and a high-pressure washer. The pad will be constructed of an impervious barrier consisting of three layers of 30 mil polyethylene sheeting with hay bales forming a berm around the perimeter. The pad will be sized so as to accommodate the largest piece of equipment to be used at the site. A submersible electric pump will be placed inside the pad, and all rinsate will be pumped into the contaminated groundwater storage facility. Any solids that accumulate in the pad will be placed in a roll-off and disposed with the drill cuttings. The materials used to construct the pad will be handled and disposed of along with the PPE.

The following procedure will be used by Foster Wheeler personnel for the decontamination of equipment that has come in contact with contaminated soil:

- Place the contaminated portion of the equipment over the decontamination pad;
- Remove all visible contaminated material from that portion of the equipment that came in contact with contamination using clean water, working from top to bottom; and
- Inspect equipment to verify that all visible contamination has been removed.

7.2.3 Hand Held Equipment and Personnel Decontamination

Personnel and hand held equipment leaving the exclusion zone will be thoroughly decontaminated in accordance with the SHSP. Personnel will doff all PPE (disposable tyvek, gloves and boots) in the CRC and place it in receptacles provided. All hand tools will be decontaminated at the equipment decontamination pad. At the conclusion of the project, collected PPE and decontamination water will be sampled as required by the disposal facility and disposed of off-site. Solid waste materials generated from this sampling effort will be handled and disposed of as TSCA waste.

7.2.4 Sedimentation and Erosion Controls

Prior to any intrusive activities, Foster Wheeler will inspect and repair all sedimentation and erosion controls previously installed around the perimeter of the site. These controls will be maintained and remain in place until all site activities are completed.

7.3 Soil Sampling Activities

7.3.1 Transformer Vaults (Buildings 53, 54, 56, 59 and 60)

The interior of the Building 53, 54, 56, 59 and 60 vaults will be sampled by drilling at a selected location to a depth of approximately 2-feet below water table or 2-feet below the bottom of the vault, whichever is deeper, using a Geoprobe. In Building 59 both the transformer side and the switch house sides of the building will be sampled. Clean disposable plastic scoops will be used to collect samples from the Macrosamplers at one-foot intervals. The samples will then be analyzed starting with the sample closest to the surface. Samples from one foot above the water table and higher will be analyzed on-site using the immunoassay method, while samples within or below the water table will be sent off-site for analysis. Where PCB concentrations are found to be greater than 10 ppm, testing will continue vertically at the one-foot sampling interval until results indicate that two consecutive samples have PCB concentrations less than or equal to 10 ppm.

The perimeter of each vault will be sampled by drilling at selected locations. Locations have been determined based on previous sampling data and by using a ten-foot grid. See Figures 2-2 and 2–2a for specific sampling locations. Samples will be collected from each boring location at one-foot intervals to a depth of approximately 2-feet below water table or 2-feet below the bottom of the vault, whichever is deeper, using a Geoprobe. In locations where rubble inhibits the use of a Geoprobe rig, either the rubble will be removed or a hand auger may be used to collect the samples. Where concrete is present at the boring location, a jackhammer will be used to create an opening for the Geoprobe. A HEPA vacuum will be used to remove all concrete debris from the area prior to installation of the boring. The surface sample at each boring location will be analyzed in the field via the immunoassay method. If the groundwater-level sample from the interior of the vault shows PCB concentrations <10 ppm, the dry samples from one foot or greater above the water table will be analyzed in the field via the immunoassay method from the surface down until two consecutive samples meet the minimum concentration of 10 ppm. If the groundwater-level sample from the interior of the vault shows PCB concentrations >10 ppm then the groundwater-level sample from each perimeter boring location will be sent to the lab for analysis prior to analyzing the remaining samples. A rapid turnaround (48-hour) analysis will be used for these samples to accommodate the holding time limitations for any additional samples that may require fixed laboratory analysis. If the lab analysis shows PCB concentrations >10 ppm, samples will be analyzed above and below the groundwater level. If the result at any depth is greater than 10 ppm, an additional boring will be installed another ten (or more as needed) feet out from the original boring location to the depth at which the previous boring yielded a result of less than or equal to 10 ppm. The sample grid will

continue to be extended until the contamination has been delineated as indicated by two consecutive sample locations with all results less than 10 ppm.

Sediment samples will be collected from selected locations along the shoreline east of Building 54. Locations have been determined based on previous sampling data and by using a ten-foot grid. See Figures 2-2 for specific sampling locations. Samples will be collected from each boring location at one-foot intervals to a depth of approximately 2-feet below water table using either a Geoprobe rig or a hand auger, depending on field conditions. The samples will then be analyzed for PCBs, starting with the sample closest to the surface. It is anticipated that all samples collected from the shoreline area will be wet and therefore will be sent off-site for analysis. The grid will be extended as far as practicable, however, due to the physical constraints associated with collection of samples further from shore in this area, complete delineation may not be possible. When the results of 2 consecutive grid intervals, where possible, are less than or equal to 1 ppm, the area will be considered completely delineated.

7.3.2 Transformer Vault (Building 61)

The interior of the Building 61 vault will be sampled by drilling at a selected location to a depth of approximately 2-feet below water table using a Geoprobe. See Figure 2-2a for specific sampling locations. Clean disposable plastic scoops will be used to collect samples from the Macrosamplers at one-foot intervals for PCB immunoassay field screening. The samples will then be analyzed starting with the sample closest to the surface. Where PCB concentrations are found to be greater than 10 ppm, testing will continue vertically at the one-foot sampling interval until immunoassay field screening indicates that two consecutive samples have PCB concentrations less than or equal to 10 ppm.

Three sides (north, south and east) of the perimeter of Building 61 consist primarily of brick and concrete rubble, therefore it may not be possible to install Geoprobe borings. At these locations an excavator will be used to remove the brick and concrete rubble and attempt to expose soil for sampling. If possible, Geoprobe borings will be installed in the exposed soil and samples collected as described above. If installation of borings is not feasible, soil samples will be collected at 1-foot intervals using the bucket of the excavator until groundwater is encountered.

The westside of Building 61 does not contain significant rubble and will be sampled as described in Section 7.3.1.

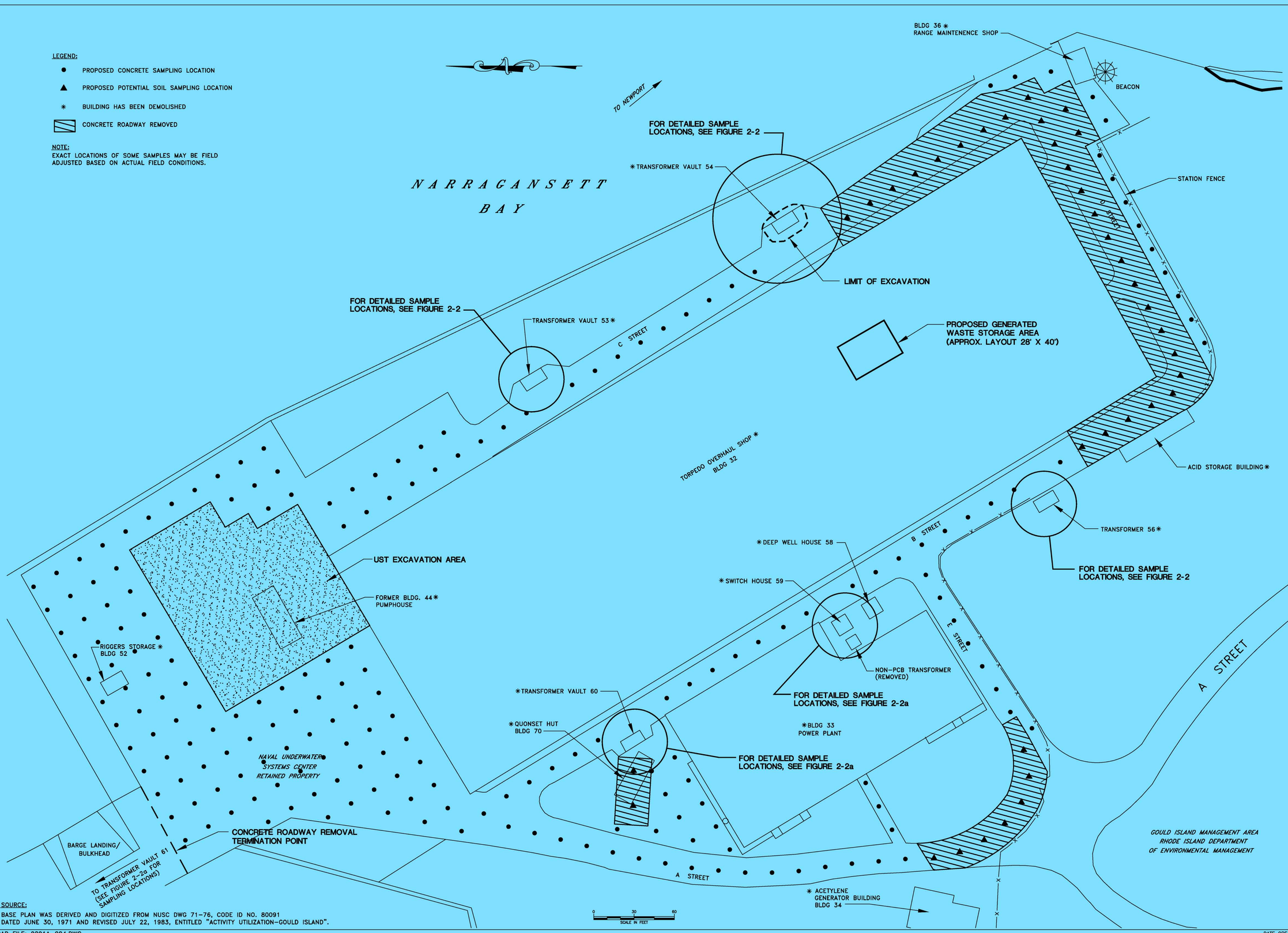
7.3.3 Concrete Roadway

The concrete roadways on the site will be sampled by drilling at selected locations using the drilling methods described in Section 5.3. The soil underlying the concrete roadways on the site will be sampled as necessary based on the results of the concrete sample collected at the bottom of the roadway slab. Concrete sample locations have been determined based on a sampling grid of approximately 20- by 20-feet within the limits of the roadways. See

LEGEND:

- ● PROPOSED CONCRETE SAMPLING LOCATION
- ▲ PROPOSED POTENTIAL SOIL SAMPLING LOCATION
- ✳ BUILDING HAS BEEN DEMOLISHED
- CONCRETE ROADWAY REMOVED

NOTE:
EXACT LOCATIONS OF SOME SAMPLES MAY BE FIELD ADJUSTED BASED ON ACTUAL FIELD CONDITIONS.



SOURCE:
BASE PLAN WAS DERIVED AND DIGITIZED FROM NUSC DWG 71-76, CODE ID NO. 80091 DATED JUNE 30, 1971 AND REVISED JULY 22, 1983, ENTITLED "ACTIVITY UTILIZATION-GOULD ISLAND".

CAD FILE: 0291A-004.DWG

DATE CREATED
LATEST CHANGE
CHANGED BY:

REV.	DESCRIPTION	PREP BY	DATE	APPRVD

FOSTER WHEELER ENVIRONMENTAL CORPORATION
133 FEDERAL STREET, BOSTON, MA 02110
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DSGN: D. CONVERS DRN: F. MONCKLU CH. ENG: CHK: E. LUCH
SUBMITTED BY: (TITLE) (DATE)
NORTHDIV DIV: ___ HD: ___ KD: ___ OR: ___ FPE: ___ OFFICER IN CHARGE
DATE
APPROVED

DEPARTMENT OF THE NAVY
NAVAL FACILITIES ENGINEERING COMMAND
ENGINEERING FIELD ACTIVITY - NORTHEAST
LESTER PENNSYLVANIA

GOULD ISLAND, NEWPORT, RHODE ISLAND
CONCRETE ROADWAY REMOVAL AND PROPOSED CONCRETE/SOIL SAMPLING BORING LOCATIONS

SEAL AREA
EFANE FOR COMMANDER, NAVFAC
DATE
APPROVED

SAT TO AUGUST 2001 DATE
CODE I.D. NO. 80091
SCALE : AS SHOWN
SPEC. NO. 04-
CONSTRN. CONTR. NO. N62472-99-D-0032
NAVFAC DRAWING NO.
SHEET 4 OF 4
SIZE: D
DIS. SH. NO. FIGURE 7-1

7-1 for specific sampling locations. Where soil samples are collected, they will be collected at the soil surface and one-foot intervals to a depth of approximately 2-feet below the soil surface. The samples will then be analyzed for PCB's, starting with the sample closest to the surface, using immunoassay field screening. Where PCB contamination is found to be greater than 10 ppm, testing will continue vertically at the one-foot sampling intervals until immunoassay field screening indicates that PCB concentrations of two consecutive samples are less than or equal to 10 ppm.

7.4 Concrete Sampling Activities - Roadway

A 20-foot by 20-foot sampling grid will be established on the concrete roadway and pad to locate and identify samples. Concrete samples will be collected using EPA Standard Operating Procedure for Sampling Concrete in the Field, December 1997. See Figure 7-1 for specific sampling locations. In addition, areas with visual staining will be sampled for PCB analysis. The top one inch of the concrete will be drilled into with a one and one-half inch drill bit and the concrete powder will be collected in a labeled sample jar. When the thickness of the concrete is known for a sample location, the one and one-half inch hole will be advanced until approximately one inch of the concrete slab remains. The hole will be vacuumed out to remove all concrete dust. Without touching the sides of the hole, a one-inch drill bit will be advanced to collect the lower sample. "Punch through" will be avoided, if possible. If a concrete sample cannot be obtained from the initial hole, a second boring will be made directly adjacent to the first one.

7.5 Removal and Disposal of Wastes

Solid wastes generated during sampling activities will be accumulated in a lined and covered roll-off. Water generated will be stored in either a Frac Tank or drums, depending on volume. At the completion of sampling activities, all accumulated waste (water, concrete, soil, PPE, PCB test kits) will be sampled for the parameters specified and at the frequency required by the waste disposal facility, removed from the island, and transported to the appropriate licensed disposal facility.

7.6 Demobilization

Following the completion of all site activities for the PCB characterization action, the site will be demobilized. Demobilization will involve the removal of all equipment, materials, portable facilities, and temporary utilities. Sedimentation and erosion control measures will remain in place.

Prior to demobilization from the site, open excavations will be stabilized. This process will involve the inspection of any plastic liner previously installed for integrity. If no liner is present at an excavation, or the previously placed liner is not intact, a new plastic liner will be placed and the excavation backfilled to grade.

8.0 TRANSPORTATION AND DISPOSAL

8.1 Objective

The objective of this section is to facilitate the proper handling, on-site management, transportation, and disposal of hazardous and non-hazardous wastes generated during sampling activities. This objective will be achieved through compliance with federal, state, and local regulations. This section identifies the waste streams and waste management responsibilities of Foster Wheeler, the Navy, transporters, and disposal facilities. This section also describes the waste management practices that will be implemented

for sampling, analyzing, classifying, segregating, staging, storing, packaging, transporting, and disposing of the generated wastes.

8.2 Naval Assistance

The ROICC will review all submittals designated for Navy approval. These submittals will include waste analysis and classifications, waste profile/approval forms, Land Disposal Restriction (LDR) certifications, manifests/shipping papers, and manifest discrepancy and exception reports. After the submittals have been approved by the ROICC, no re-submittals will be given consideration unless accompanied by a written justification as to why a change is necessary. Foster Wheeler will rely on the Navy to provide approval of final waste characterizations and sign as the generator of all waste streams leaving the site. The Navy will also make Treatment/disposal facilities and transporter approval.

8.3 Waste Streams

The estimated quantities and analyses of waste streams generated from the characterization action activities are indicated in Table 8-1. All wastes other than the contaminated water will be disposed as TSCA for this activity. Any analyses, other than those listed below; necessary for disposal of each waste stream will be performed by hazardous waste materials transporter. The results of such analyses will be forwarded to the Contracting Officer's Representative in accordance with the Submittal Register.

Table 8-1
Estimated Quantities and Analyses for Transportation and Disposal

Waste Stream	Estimated Quantity	Containerization	Planned Analyses
1. Contaminated Water	15,000 gallons	Frac Tank or Drums	PCB's, VOC, SVOC, TAL Metals, Reactivity, Corrosivity, Ignitability
2. Drill Cuttings, PPE, Immunoassay Kit Waste	15 tons	Rolloff	PCB's, VOC, SVOC, TAL Metals, Reactivity, Corrosivity, Ignitability

8.4 On-Site Waste Management

8.4.1 Containerization

All waste streams will be evaluated prior to generation to determine the most cost-effective method of handling and storage. All containerized wastes will be stored in U.S. DOT specification containers. Bulk and non-bulk containers will be considered based on estimated volumes of waste to be generated. All waste destined for off-site disposal will be stored in U.S. DOT specification containers.

Decontamination water will be stored in frac tanks or drums, depending on quantity, and solid waste will be stored in a lined and covered roll-off.

8.4.2 Toxic Substance Management

All containers will be stored in accordance with 40 CFR 761.65 and labeled in accordance with 40 CFR 761.40, 40 CFR 761.45, and Navy Specification Section 13284. Section 40 CFR 761.65(c)(9) allows for storage of bulk PCB remediation wastes at the clean-up site or site of generation for 180 days. The storage area will be on the Building 32 concrete pad (see Figure 7-1), and will comply with the following conditions:

- The waste will be placed in a pile designed and operated to control dispersal of the waste by wind, where necessary, by means other than wetting;
- The waste will be stored to avoid the generation leachate through decomposition or other reactions; and,
- The storage facility will be lined to prevent any migration of wastes off or through the liner into the adjacent subsurface soil, ground water or surface water; will be covered, and will have a run-on control system in accordance with the regulations.

8.5 Transportation and Disposal Plan

Upon characterization of each waste stream, a waste profile and manifest will be submitted to the Contracting Officer's Representative for Navy approval and signature prior to any wastes being transported off-site. The package will include the following information:

- Copies of all permits and contaminant level limitations for the receiving facilities
- Waste Transporter:
  - Name:
  - Address:
  - EPA Transporter Identification Number and/or State Permit Number:
- Out-of State Waste Transporter:
  - Name:
  - Address:
  - EPA Transporter Identification Number and/or State Permit Number:
- EPA Hazardous Waste Generator Identification Number: RI6170022036
- Proof of permit, license, or authorization to transport hazardous materials in all affected states

For every load disposed of at the approved disposal facilities, copies of the manifests, Bills of Lading, and tare and gross weight slips will be provided to the Contracting Officer's Representative.

8.6 Transportation and Disposal Requirements

8.6.1 TSCA Regulated Waste

As required by 40 CFR 761.40, PCB containers (containing material with PCB concentrations greater than 50 ppm) must be marked in accordance with 40 CFR 761.45. The off-site transportation and disposal of TSCA regulated waste must meet the PCB waste recording and reporting requirements of 40 CFR 761.202 through 761.218 (Subpart K). This Subpart specifies EPA identification number, notification, manifest, record keeping, reporting, and disposal certification requirements.

In accordance with 40 CFR 761.60(a)(4), non-liquid waste with PCB concentrations greater than 50 ppm may be either incinerated in accordance with 40 CFR 761.70 or disposed of at a TSCA waste landfill in accordance with 40 CFR 761.75. Solid waste materials generated from this sampling effort will be handled and disposed of as TSCA waste.

8.6.2 U.S. DOT Requirements

The off-site transportation of EPA designated hazardous wastes must comply with U.S. DOT regulations as set forth in 49 CFR 171 through 178. PCB contaminated waste shipments which exceed the reportable quantity (one pound) also must comply with U.S. DOT regulations 49 CFR 171 through 178. These regulations provide for training, shipping paper, packaging, marking, labeling, placarding, emergency response information, and shipper requirements.

8.6.3 EPA Hazardous Waste Generator Identification Numbers

The Navy has previously notified the EPA Region I that they are a generator under TSCA. The anticipated hazardous wastes and TSCA regulated wastes accumulated during the sampling will be disposed of using the Navy's EPA Hazardous Waste Generator Identification Number RI6170022036. The Navy will be responsible for obtaining and supplying to Foster Wheeler any additional Generator Identification Numbers required. Transporter and disposal facility identification numbers will be obtained and verified by Foster Wheeler.

8.6.4 Transportation Routes

The transportation routes that will be taken when the PCB wastes are removed from Gould Island will be dependent upon the locations of the Navy-approved disposal facilities, and will likely be developed by the transportation and disposal subcontractor.

8.6.5 Laboratory Sample Shipment

Off-site laboratory services will be used for this project. Generally, EPA designated hazardous waste is a U.S. DOT hazard class 9 waste. Samples containing greater than one pound of PCBs must also comply with U.S. DOT Hazardous Material Regulations. Although hazardous waste samples undergoing analysis are excluded from regulation under RCRA as per 40 CFR 261.4(d), U.S. DOT Hazardous Material Regulations still would apply in cases where a carrier is used to transport the samples, the laboratory picks up the samples at the site, or Foster Wheeler transports the samples in personal or company vehicles to the off-site laboratory. Site personnel should coordinate with Foster Wheeler waste management personnel approximately five days prior to shipping the first set of samples off-site and two days prior to shipping all subsequent samples. Pursuant to 40 CFR 172 Subpart H, Foster Wheeler policy requires site personnel involved in shipping U.S. DOT regulated material be trained in U.S. DOT regulations.

8.6.6 Land Disposal Restrictions

LDRs prohibit placement of untreated hazardous wastes on or in the land, except in an EPA approved management unit. LDRs specify treatment technologies and treatment standards for hazardous wastes. Foster Wheeler will identify LDRs for site generated wastes and will prepare Generator Land Disposal Restricted Waste Notification and Certification forms required for all off-site hazardous waste shipments. The Notification and Certification forms will be provided to the Navy for review and signature prior to off-site waste shipments.

8.7 Spill Prevention

Every effort will be made, through proper planning and management of the transportation process, to prevent the potential for a spill or release of hazardous substances. However, contingency measures will be in place in the case of such an occurrence. This includes providing personnel, equipment, and materials to control, contain, and cleanup any spilled material that may adversely affect the health of the public or the environment. Transporters responsible for taking waste materials to the designated disposal facilities will be required to provide and implement their own Emergency Response Plan which will be reviewed and approved by Foster Wheeler prior to the start of work. All vehicles will be inspected prior to leaving the area of contamination for leakage or materials adhering to the wheels or undercarriage. If necessary, vehicles will be cleaned with a high-pressure water to remove contaminated material.

The following equipment will be available at both locations at all times for quick response to unexpected spills:

- Sorbents and spill cleanup materials, including spill control pillows, absorbent booms, packs, and blankets
- 55-gallon containers
- Shovels, brooms, and similar hand tools
- Pressure washer

Additional information relative to spill response can be found in Section 12.12 of the SHSP. A Spill Prevention Control and Countermeasure (SPCC) Plan, prepared according to 40 CFR Part 112, will not be required for this project. Should a spill of PCB materials occur during this activity, the spill will be cleaned up in accordance with the EPA Spill Cleanup Policy (40 CFR 761, Subpart G) that applies to PCB contaminated media resulting from a spill source having PCB concentrations equal to or greater than 50 ppm.

9.0 REPORTING

9.1 Work Plan

Following the completion of the characterization actions, Foster Wheeler will prepare a Work Plan that will document the results of the sampling and characterization work. The data obtained from this sampling effort will be used to determine the action required at each of the transformer locations and along the concrete roadway to meet the regulatory criteria. Foster Wheeler will describe the recommended removal action activities, quantities of waste to be removed, post-cleanup analytical methods, and conclusions relative to the removal actions.

10.0 REFERENCES

1. *Rules and Regulations for the Investigation and Remediation of Hazardous Material Releases*, issued by the Rhode Island Department of Environmental Management Division of Site Remediation on March 31, 1993, and amended August 1996.
2. *Final Master Site Health And Safety Plan for Gould Island, Naval Station Newport*, Foster Wheeler Environmental Corp, July 22, 1999
3. *Draft Work Plan for Gould Island Demolition of Selected Buildings - Phases I, II, & III,* Foster Wheeler Environmental Corp, July 19, 1999.
4. *Addendum 2 to Draft Work Plan for Gould Island Demolition of Selected Buildings - Phases I, II, & III,* Foster Wheeler Environmental Corp, November 15, 2000.
5. *Navy Installation Restoration Chemical Data Quality Manual*, September 1999
6. *Region I, EPA-New England Compendium of Quality Assurance Project Plan Guidance, Final*, October 1999
7. 40 CFR 172 Subpart H
8. 40 CFR 761
9. 40 CFR Part 112
10. 49 CFR 171 through 178, U.S. DOT regulations

APPENDIX A

QC FORMS

APPENDIX A

QC FORMS

Includes the following:

- Contractor Production Report
- Daily Quality Control Reort
- Preparatory Inspection Checklist
- Initial Inspection Checklist
- Rework Items List
- Test Plan and Log
- Chain of Custody Record
- Submittal Form
- Change Request Form
- Request for Information

CONTRACTOR PRODUCTION REPORT

		DATE
CONTRACT NO	TITLE AND LOCATION	REPORT NO
CONTRACTOR Foster Wheeler Environmental Corp.	SUPERINTENDENT	
WEATHER	MAX TEMP F	MIN TEMP F

WORK PERFORMED TODAY

WORK LOCATION AND DESCRIPTION	EMPLOYER	#	TRADE	HRS

JOB SAFETY

WAS A JOB SAFETY MEETING HELD THIS DATE? ☐ YES ☐ NO
(if YES attach copy of the meeting minutes)

WERE THERE ANY LOST TIME ACCIDENTS THIS DATE? ☐ YES ☐ NO
(if YES attach copy of completed OSHA report)

WAS TRENCHING SCAFFOLD HV ELECTRICAL/HIGH WORK DONE? ☐ YES ☐ NO
(if YES attach statement of checklist showing inspection performed)

WERE THERE ANY HEAR MISS ACCIDENTS THIS DATE? ☐ YES ☐ NO
(if YES attach description of incident and proposed action)

TOTAL WORK HOURS ON JOB SITE THIS DATE	
CUMULATIVE TOTAL OF WORK HOURS FROM PREVIOUS REPORT	
TOTAL WORK HOURS FROM START OF CONSTRUCTION	

LIST SAFETY ACTIONS TAKEN TODAY, SAFETY INSPECTIONS CONDUCTED.

EQUIPMENT, MATERIAL / RECEIVED TODAY TO BE INCORPORATED IN JOB.

CONSTRUCTION AND PLANNED EQUIPMENT ON JOB SITE TODAY. INCLUDE NUMBER OF HOURS USED TODAY.

REMARKS:

CONTRACTOR SUPERINTENDENT _____ DATE _____

CONTRACTOR QUALITY CONTROL REPORT

(ATTACH ADDITIONAL SHEETS IF NECESSARY)

DATE

PHASE IDENTIFY DEFINABLE FEATURE OF WORK, LOCATION AND LIST PERSONNEL PRESENT.

Y - YES N - NO SEE REMARKS
BLANK NOT APPLICABLE

PREPARATORY

	THE PLANS & SPECS HAVE BEEN REVIEWED.
	THE SUBMITTALS HAVE BEEN APPROVED.
	MATERIALS COMPLY WITH APPROVED SUBMITTALS.
	MATERIALS ARE STORED PROPERLY.
	PRELIMINARY WORK IS DONE CORRECTLY.
	SAFETY REQUIREMENTS HAVE BEEN MET.
	TESTING PLAN HAS BEEN REVIEWED.
	WORK METHOD SCHEDULE DISCUSSED.

INITIAL

TESTING PERFORMED & WHO PERFORMED TEST.

	PRELIMINARY WORK IS DONE CORRECTLY.
	SAMPLE HAS BEEN APPROVED/PREPARED.
	SAFETY REQUIREMENTS HAVE BEEN MET.
	TEST RESULTS ARE ACCEPTABLE.
	WORK IS IN COMPLIANCE WITH THE CONTRACT.

FOLLOW-UP

TESTING PERFORMED & WHO PERFORMED TEST.

	WORK COMPLIES WITH CONTRACT AS APPROVED IN INITIAL PHASE.

REWORK ITEMS IDENTIFIED TODAY (NOT CORRECTED BY CLOSE OF BUSINESS).

REWORK ITEMS CORRECTED TODAY (FROM REWORK LOG).

REMARKS

On behalf of the contractor, I certify that the report is complete and correct and all equipment and material used and work performed during this reporting period is in compliance with the contract plans and specifications to the best of my knowledge except as noted above.

AUTHORIZED CQC MANAGER AT SITE DATE

GOVERNMENT QUALITY ASSURANCE REPORT

DATE

QUALITY ASSURANCE REPRESENTATIVES REMARKS AND/OR EXCEPTIONS TO THE REPORT

GOVERNMENT QUALITY ASSURANCE REPRESENTATIVE DATE

Sheet ____ of ____

FOSTER WHEELER ENVIRONMENTAL CORPORATION

PREPARATORY INSPECTION CHECKLIST

CONTRACT No: N62472-99-D-0032 CTO#0000 DATE: _____

TITLE: _____ SPEC SECTION: _____

MAJOR DEFINABLE SEGMENT OF WORK: _____

A. PERSONNEL PRESENT:

NAME	POSITION	COMPANY
1.		
2.		
3.		
4.		
5.		
6.		

(List additional personnel on reverse side)

B. SUBMITTALS INVOLVED REVIEW - SPECIFICATIONS AND DRAWINGS:

NUMBER & ITEM	COC APPROVAL	NAVY APPROVED
1.		
2.		
3.		
4.		
5.		
6.		

B.1 HAVE ALL ITEMS BEEN APPROVED? YES: _____ NO: _____

B.2 WHAT ITEMS HAVE NOT BEEN APPROVED?

ITEM	STATUS
1.	
2.	
3.	
4.	
5.	

C. ARE ALL MATERIALS ON HAND? YES: _____ NO: _____

C.1 ARE ALL MATERIALS ON HAND IN ACCORDANCE WITH APPROVALS?
YES: _____ NO: _____

C.2 ITEMS NOT ON HAND OR NOT IN ACCORDANCE WITH SUBMITTALS:

1.
2.
3'.

D. TESTS REQUIRED IN ACCORDANCE WITH CONTRACT REQUIREMENTS:

TEST	PARAGRAPH
1.	
2.	
3.	

E. ACCIDENT PREVENTION PREPLANNING - HAZARD CONTROL MEASURES:

E.1 WHAT ARE THE HAZARDS AND CONTROLS? (or attach activity hazard analysis)

1.
2.
3.
4.

F. EXAMINED THE WORK AREA TO ENSURE THAT THE REQUIRED PRELIMINARY WORK HAS BEEN COMPLETED. YES: _____ NO: _____

G. DISCUSSED THE PROCEDURES FOR CONTROLLING QUALITY OF THE WORK INCLUDING REPETITIVE DEFICIENCIES. YES: _____ NO: _____

H. DISCUSSION OF THE INITIAL CONTROL PHASE.
YES: _____ NO: _____

_____
QUALITY CONTROL MANAGER

FOSTER WHEELER ENVIRONMENTAL CORPORATION

INITIAL INSPECTION CHECKLIST

OBSERVE THE INITIAL SEGMENT OF THE DEFINABLE FEATURE OF WORK TO ENSURE THAT THE WORK COMPLIES WITH CONTRACT REQUIREMENTS.

CONTRACT No: N62472-99-D-0032 CTO#0000 DATE: _____

TITLE: _____ SPEC SECTION: _____

MAJOR DEFINABLE SEGMENT OF WORK: _____

DESCRIPTION AND LOCATION OF WORK INSPECTED: _____

REFERENCE CONTRACT DRAWINGS: _____

A. PERSONNEL PRESENT:

NAME	POSITION	COMPANY
1.		
2.		
3.		
4.		
5.		
6.		

(List additional personnel on reverse side)

B. MATERIALS BEING USED ARE IN STRICT COMPLIANCE WITH THE CONTRACT PLANS AND SPECIFICATIONS: YES: _____ NO: _____

IF NOT, EXPLAIN _____

C. PROCEDURES AND/OR WORK METHODS WITNESSED ARE IN STRICT COMPLIANCE WITH THE REQUIREMENTS OF THE CONTRACT SPECIFICATIONS:

YES: _____ NO: _____

IF NOT, EXPLAIN _____

D. WORKMANSHIP IS ACCEPTABLE: YES: _____ NO: _____

STATE AREAS WHERE IMPROVEMENT IS NEEDED: _____

E. ENSURE THAT TESTING HAS BEEN PERFORMED BY APPROVED LABORATORY:

YES: _____ NO: _____

F. SAFETY VIOLATIONS AND CORRECTIVE ACTION TAKEN: _____

REPEAT THE INITIAL PHASE FOR EACH NEW CREW TO WORK ON-SITE, OR WHEN ACCEPTABLE LEVELS OF SPECIFIED QUALITY ARE NOT MET.

_____

QUALITY CONTROL MANAGER

REWORK ITEMS LIST

Contract No. and Title: _____

Contractor: _____

NUMBER	DATE IDENTIFIED	DESCRIPTION	CONTRACT REQUIREMENT (Spec. Section and Par. No., Drawing No., and Detail No., etc.	ACTION TAKEN BY QC MANAGER	RESOLUTION	DATE COMPLETED

TEST PLAN and LOG

CONTRACT NUMBER
N62472-99-D-0032/CTO#0000

PROJECT TITLE AND LOCATION
XXXXXX

CONTRACTOR
FOSTER WHEELER ENVIRONMENTAL CORP.

SPECIFICATION SECTION & PARAGRAPH NO.	ITEM OF WORK	TEST REQUIRED	ACCREDITED/ APPROVED LAB		SAMPLED BY	TESTED BY	LOCATION OF TEST		FREQUENCY	DATE COMPLETED	DATE FORWARDED TO CO	REMARKS
			YES	NO			ON-SITE	OFF-SITE				

analytics environmental laboratory LLC

195 Commerce Way Suite E
Portsmouth, NH 03801
Phone (603) 436-5111
Fax (603) 430-2151

For Analytics Use Only Rev 2/00

Samples were:

1) Shipped or hand-delivered

2) Temp blank °C

3) Received in good condition Y or N

4) pH checked by:

5) Labels checked by:

Project#: Proj. Name:

Company:

Contact:

Address:

Phone: PO# Quote #

Sampler (Signature):

Matrix Key:

WW=Wastewat
SW=Surfacewat
GW=Groundw
DW=Drinkingwat
S=Soil/Sludg
O=Oil
E=Extract
X=Other

Container Key

P=plastic G=glass

Station Identification	Sample Date	Sample Time	Analysis	Preservation: Unpres	4° C	HNO_3	H_2SO_4	HCL	Other	Matrix	Container number/typ		pH	Analytics Sample #

Comments / Instructions:

FAX RESULTS? YES NO

Fax # _____

Turnaround Request

Standard ☐ Due Date _____

Priority ☐ Due Date _____

Prepare in quintuplicate (original and 4 copies)

CONTRACTOR DRAWINGS & INFORMATION SUBMITTAL
EFANENAVFACENGCOM 4335/3 (Rev. 6/80)

CONTROL NO. 01C

CONTRACT NO.	CONTRACT TASK ORDER NO.	ACTIVITY LOCATION
N62472-99-D-0032	**0069**	**Gould Island – Newport, RI**

PROJECT TITLE:
PCB Characterization and Removal – Gould Island

FROM:	DATE
Foster Wheeler Environmental Corp. - Program QCM: Thomas Kelly	November 21, 2001
TO:	DATE
C. Davis (E-Copy)	November 21, 2001

1. THE CONTRACTOR SUBMITTALS LISTED BELOW ARE FORWARDED FOR YOUR REVIEW AND RECOMMENDATIONS.
(a) APPLY APPROPRIATE STAMP IMPRINT TO EACH SUBMITTAL AND INDICATE REVIEW COMMENTS, AS REQUIRED.
(b) RETAIN ONE (1) COPY OF THIS TRANSMITTAL FORM AND RETURN REMAINING COPIES WITH REVIEWED SUBMITTALS TO ROICC.

2. THESE SUBMITTALS SHOULD BE RETURNED TO THIS OFFICE BY _____

3. _____

E-COPY TO:
EFANE: B. Krivinskas , P. Burgio, J. Shafer, D. Barclift, T. Bober
NSNPT: M. Griffin
USEPA: K. Tisa, K. Keckler
RIDEM: P. Kulpa
GF: J. Stump

☐ ROICC ☐ RPM ☐ CSO

Thomas Joseph Kelly NOVEMBER 21, 2001
SIGNATURE AND DATE

FROM: DESIGNER	DATE
TO: ROICC	DATE

1. THE SUBMITTALS LISTED BELOW HAVE BEEN REVIEWED AND ARE RETURNED, WITH ACTION TAKEN AS INDICATED.

2. _____

COPY TO:
☐ ROICC ☐ DESIGNER

SIGNATURE AND DATE

FROM: ROICC	DATE
TO: CONTRACTOR	DATE

1. THE SUBMITTALS LISTED BELOW HAVE BEEN REVIEWED AND ARE APPROVED/DISAPPROVED AS SHOWN BELOW AND ON EACH STAMP IMPRINT.

COPY TO:
☐ ROICC ☐ OTHER

FOR COMMANDING OFFICER, ENGINEERING FIELD ACTIVITY NORTHEAST - NAVAL FACILITIES ENGINEERING COMMAND DATE

ITEM NO.	SUBMITTAL DESCRIPTION	PREPARED/ SUBMITTED BY	APPROVED	DISAPPROVED	REMARKS
1	SD-08, Statements; Draft Sampling Plan for the Characterization of PCB Contaminated Soils and Concrete – Gould Island (R2)	Thomas Kelly			

Foster Wheeler Environmental Corporation
Change Request Form

Section 1 through 4 to be filled out by Foster Wheeler, Section 5 to be filled out by Navy

PROJECT:	OFS No.:	Change Request Form:
Navy RAC CTO NO. XX	**2282-00XX**	**CRF - XXX** **Rev.** X

To: _____ Dept.: _____ Location: _____ Date: _____

Re: ☐ Drawing No. _____ Title _____

☐ Spec. No. _____ Title _____

☐ Other _____

1. DESCRIPTION *(Items involved, submit sketch if applicable)*

2. REASONS FOR CHANGE *(If from disposition of nonconformance report, list report number)*

3. RECOMMENDED DISPOSITION

☐ Technical Clarification [NTR & COTR approval required]
☐ In Scope Adjustment [COTR approval required]
☐ Out of Scope [CO & COTR approval required]
☐ Cost Growth
☐ ROM Estimate (If Applicable) $ _____
☐ Schedule Impact (describe below)

FWENC Initiator (Signature/Title):

4. FWENC Project Manager (Signature)	Date	Project Superintendent Concurrence (Signature)	Date

5. NAVY DISPOSITION

☐ Approved per recommended disposition
☐ Not approved (give reason)
☐ Approved with modification(s) [describe below]

NTR Concurrence *(signature)*	Date	ROICC Concurrence *(Signature)*	Date

Contracting Officer Technical Representative Approval *(Signature)*	Contracting Officer Approval *(Signature)*	Date

Engineer signs and transmits to Resident Engineer with copies to:

_____ Project Manager
_____ Project Superintendent
_____ Quality Control

Others as Required _____
File: _____
_____

REQUEST FOR INFORMATION

TO: ROICC
XXXXXXXXXX
XXXXXXXXXX

RFI: 000

DATE: XX/XX/XX

Contract: N62472-99-D-0032
CTO # 00XX

Attn: XXXXXXXX

FROM: Xxxxx Xxxxxx
Site QC Manager

REFERENCES:
DRAWING:
SPEC SECTION:

Reply Needed By: xx/xx/xx

QUESTION (#1):

Name: _____

REPLY:

Date:_____

This reply is given with the expressed understanding that it does not constitute the basis for a change to the price or time for the contract.

If you do not concur, DO NOT PROCEED, and notify the writer immediately.

Name: _____

Title: _____

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN (QAPP) ADDENDUM

U.S. NAVY ENGINEERING FIELD ACTIVITY - NORTHEAST
REMEDIAL ACTION CONTRACT (RAC)
CONTRACT NO. N62472-99-D-0032
CONTRACT TASK ORDER NO. 069

QUALITY ASSURANCE PROJECT PLAN (QAPP) ADDENDUM FOR THE CHARACTERIZATION OF PCB CONTAMINATED SOILS GOULD ISLAND NAVAL STATION NEWPORT MIDDLETOWN, RHODE ISLAND

Issued:
November 20, 2001

Prepared for:
ENGINEERING FIELD ACTIVITY - NORTHEAST
NAVAL FACILITIES ENGINEERING COMMAND
10 Industrial Highway
Lester, PA 19113

Prepared by

Foster Wheeler Environmental Corporation
2300 Lincoln Highway
One Oxford Valley, Suite 200
Langhorne, Pennsylvania 19047

1.0 INTRODUCTION

Foster Wheeler Environmental Corporation is submitting this Quality Assurance Project Plan (QAPP) Addendum to the Naval Facilities Engineering Command to support the remediation activities at Gould Island, Naval Station Newport, Middletown, RI. This QAPP Addendum was developed to contain all information that would be in a Field Sampling Plan and a Quality Assurance Project Plan.

This QAPP Addendum was developed as an addition to the Final Sampling Plan For the Characterization of PCB Contaminated Soils and follows the Region I, EPA-New England Compendium of Quality Assurance Project Plan Guidance (USEPA Region I, 1999). Only information pertaining to the site activities to support the current Remedial Activities are included in this document and additional information is provided in the Final Sampling Plan.

2.0 PROJECT DEFINITION

Gould Island is located in Narragansett Bay approximately 1.5 miles from Naval Station, Newport. As described in the Work Plan, Foster Wheeler performed asbestos abatement (Section 4.3 of the Work Plan), hazardous waste removal activities (Section 4.4 of the Work Plan), demolition of designated buildings (Section 4.5 of the Work Plan), and removal of designated building slabs and foundations (Section 5.0 of the Work Plan and Work Plan Addendum No. 3) on Gould Island. Foster Wheeler is performing this work in three phases. Refer to Figure 2-1 for the Gould Island Site Plan.

Phases I and II included asbestos abatement, hazardous waste removals, and demolition of the buildings to the slab elevation only. Phase I and II were completed in May 2001. In support of the building demolition, Foster Wheeler performed concrete chip sampling of the interior floor and wall surfaces of the transformer vaults and the switch house. Phase III activities included the removal and crushing of selected building slabs and foundations. The results of that sampling indicated the presence of contamination in some of the floor locations. This Sampling Plan is being prepared to assist in delineating the extent of contamination in support of the Phase III activities.

Concrete samples will be collected using EPA Standard Operating Procedure for Sampling Concrete in the Field, December 1997. A 20 foot by 20 foot sampling grid will be established on the concrete pad to locate and identify samples. In addition, areas with visual staining will be sampled for PCB analysis. The top one inch of the concrete will be sampled using a hammer drill with a one and one-half inch drill bit and the concrete powder will be collected in a labeled sample jar. When the thickness of the concrete is known for this sample location, the one and one-half inch hole will be advanced until approximately one inch of the concrete slab remains. The hole will be vacuumed out to remove all concrete dust. Without touching the sides of the hole, a one inch drill bit will be advanced to collect the lower sample. "Punch through" will be avoided, if possible. If a concrete sample cannot be obtained from the initial hole, a second boring will be made directly adjacent to the first one.

Care must be taken to note the moisture content of the concrete. The powdered concrete generated by the drill bit will be difficult to collect if it is wet. All concrete samples will be sent off site to the fixed laboratory. The field chemist will use personal judgment to determine where a valid sample may be collected. In addition, if the "bottom" concrete sample demonstrates PCB concentrations greater than 1 ppm, the soil beneath the concrete at that location will be sampled and analyzed for PCB.

The horizontal and vertical extent of PCB contamination in the soil will be determined using immunoassay field analysis and off site confirmation. Soil samples will be collected at one-foot intervals to a depth of approximately two feet below the water table. Samples one foot above the water table will

be analyzed on site while samples within or below the water table will be sent off site to the fixed laboratory. Accounting for water table fluctuations, it is anticipated that a depth of 2-feet below the water table will allow sampling of the entire smear zone. Should data indicate that the deepest sample collected still contains PCBs above the target levels, the boring will be deepened at that location in 4-foot increments. The core will be sampled at one-foot intervals.

Perimeter sample locations will be determined based on previous sampling data and by using a ten-foot grid. Should the lateral extent of PCB contamination extend beyond the sampling area, the sample grid will be extended by 10-foot increments until the full extent of contamination has been determined. Where concrete is present at the boring location, a jackhammer will be used to create an opening for the Geoprobe. A HEPA vacuum will be used to remove all concrete debris from the area prior to installation of the boring.

Samples will be retrieved using a Geoprobe with an acetate sleeve to contain the sample (Macrosampler). The sleeve will be cut into four 1-foot lengths for sample collection. Based on previous experience at the site, it is anticipated that nearly full recovery of the 4-foot length will be possible with this method. If recovery is less than required for sampling purposes, field adjustments to the sampling methodology will be made.

2.1 Project Description

Sampling Tasks:

A total of approximately 500 concrete samples will be collected from the concrete pad surrounding the buildings on a twenty by twenty foot sampling grid. Concrete will be sampled at intervals of 0 – 1.0 inch and 7.0 – 8.0 inch, or the bottom one inch of the pad, below the pad surface. If the bottom concrete sample contains PCB concentrations above 1 ppm, the soil beneath the concrete pad will be sampled at intervals of one foot.

Analysis Tasks:

Pulverized concrete samples will be sent to the Analytics Environmental Laboratory for PCBs analysis using Method SW-846 8082. The compounds of concern, project action limits and analytical contract required quantitation limits are given in Table 1.

Table 1 A

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
VOCs in Soil

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (% R)	Completeness (% valid Data)
MATRIX: SOIL PQL's assume 100% solids. PQL's will vary based on percent solids.								
VOCs: Medium Level (Methanol Preserved) 5ml purges								
Dichlorodifluoromethane	5035/8260B		µg/kg	100	24.5	70-130	60-140	
Chloromethane	5035/8260B		µg/kg	100	23.3	70-130	60-140	
Vinyl Chloride	5035/8260B		µg/kg	100	21.2	70-130	60-140	
Bromomethane	5035/8260B		µg/kg	100	27.7	70-130	60-140	
Chloroethane	5035/8260B		µg/kg	100	30.1	70-130	60-140	
t-Butyl alcohol	5035/8260B		µg/kg	4000	654.8	70-130	60-140	
Trichlorofluoromethane	5035/8260B		µg/kg	100	19.9	70-130	60-140	
Diethyl ether	5035/8260B		µg/kg	100	17.5	70-130	60-140	
Acetone	5035/8260B		µg/kg	500	92.2	70-130	60-140	
1,1-Dichloroethene	5035/8260B		µg/kg	75	13.7	70-130	60-140	
Di-isopropyl ether	5035/8260B		µg/kg	100	30.1	70-130	60-140	
Methylene Chloride	5035/8260B		µg/kg	250	32.6	70-130	60-140	
Carbon Disulfide	5035/8260B		µg/kg	100	18.8	70-130	60-140	
Methyl-tert-butyl ether	5035/8260B		µg/kg	100	18	70-130	60-140	
trans-1,2-Dichloroethene	5035/8260B		µg/kg	100	18.1	70-130	60-140	
1,1-Dichloroethane	5035/8260B		µg/kg	100	29	70-130	60-140	
Methyl ethyl ketone	5035/8260B		µg/kg	500	108.4	70-130	60-140	
Ethyl t-butyl ether	5035/8260B		µg/kg	100	13	70-130	60-140	
2,2-Dichloropropane	5035/8260B		µg/kg	100	19.5	70-130	60-140	
cis-1,2-Dichloroethene	5035/8260B		µg/kg	100	18.3	70-130	60-140	
t-Amyl methyl ether	5035/8260B		µg/kg	100	13.3	70-130	60-140	
Chloroform	5035/8260B		µg/kg	75	18.4	70-130	60-140	
Bromochloromethane	5035/8260B		µg/kg	100	17.5	70-130	60-140	
Tetrahydrofuran	5035/8260B		µg/kg	500	64.3	70-130	60-140	
1,1,1-Trichloroethane	5035/8260B		µg/kg	100	13.9	70-130	60-140	
1,1-Dichloropropene	5035/8260B		µg/kg	100	18.2	70-130	60-140	
Carbon Tetrachloride	5035/8260B		µg/kg	100	16.4	70-130	60-140	
1,2-Dichloroethane	5035/8260B		µg/kg	75	18.9	70-130	60-140	
Benzene	5035/8260B		µg/kg	100	17.4	70-130	60-140	
Trichloroethene	5035/8260B		µg/kg	100	17.7	70-130	60-140	
1,2-Dichloropropane	5035/8260B		µg/kg	75	12.6	70-130	60-140	
Bromodichloromethane	5035/8260B		µg/kg	75	15.4	70-130	60-140	
Dibromomethane	5035/8260B		µg/kg	100	14.8	70-130	60-140	
2-Hexanone	5035/8260B		µg/kg	500	109.3	70-130	60-140	
Methyl isobutyl ketone	5035/8260B		µg/kg	500	114.2	70-130	60-140	
cis-1,3-Dichloropropene	5035/8260B		µg/kg	100	16.5	70-130	60-140	
Toluene	5035/8260B		µg/kg	100	14.3	70-130	60-140	
trans-1,3-Dichloropropene	5035/8260B		µg/kg	100	16.0	70-130	60-140	
1,1,2-Trichloroethane	5035/8260B		µg/kg	75	14.8	70-130	60-140	
1,3-Dichloropropane	5035/8260B		µg/kg	100	15.3	70-130	60-140	
Tetrachloroethene	5035/8260B		µg/kg	100	14.5	70-130	60-140	
Dibromochloromethane	5035/8260B		µg/kg	75	15.8	70-130	60-140	
1,2-Dibromoethane	5035/8260B		µg/kg	75	11.1	70-130	60-140	
Chlorobenzene	5035/8260B		µg/kg	100	16.8	70-130	60-140	
1,1,1,2-Tetrachloroethane	5035/8260B		µg/kg	100	20.3	70-130	60-140	
Ethylbenzene	5035/8260B		µg/kg	100	18.9	70-130	60-140	
m,p-Xylene	5035/8260B		µg/kg	100	11.6	70-130	60-140	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (% R)	Completeness (% valid Data)
o-Xylene	5035/8260B		µg/kg	100	16.7	70-130	60-140	
Styrene	5035/8260B		µg/kg	100	18.8	70-130	60-140	
Bromoform	5035/8260B		µg/kg	75	15.2	70-130	60-140	
Isopropylbenzene	5035/8260B		µg/kg	100	17.1	70-130	60-140	
1,1,2,2-Tetrachloroethane	5035/8260B		µg/kg	75	13.4	70-130	60-140	
1,2,3-Trichloropropane	5035/8260B		µg/kg	100	10.8	70-130	60-140	
trans-1,4-Dichloro-2-butene	5035/8260B		µg/kg	100	22.8	70-130	60-140	
n-Propylbenzene	5035/8260B		µg/kg	100	19.2	70-130	60-140	
Bromobenzene	5035/8260B		µg/kg	100	23.7	70-130	60-140	
1,3,5-Trimethylbenzene	5035/8260B		µg/kg	100	22.5	70-130	60-140	
2-Chlorotoluene	5035/8260B		µg/kg	100	20.9	70-130	60-140	
4-Chlorotoluene	5035/8260B		µg/kg	100	17.7	70-130	60-140	
tert-butylbenzene	5035/8260B		µg/kg	100	16.5	70-130	60-140	
1,2,4-Trimethylbenzene	5035/8260B		µg/kg	100	19.8	70-130	60-140	
Pentachloroethane	5035/8260B		µg/kg	100	16.6	70-130	60-140	
sec-butylbenzene	5035/8260B		µg/kg	100	15.6	70-130	60-140	
p-isopropyltoluene	5035/8260B		µg/kg	100	18.7	70-130	60-140	
1,3-Dichlorobenzene	5035/8260B		µg/kg	100	19.1	70-130	60-140	
1,4-Dichlorobenzene	5035/8260B		µg/kg	100	16.3	70-130	60-140	
n-butylbenzene	5035/8260B		µg/kg	100	23.3	70-130	60-140	
1,2-Dichlorobenzene	5035/8260B		µg/kg	100	17.2	70-130	60-140	
1,2-Dibromo-3-chloropropane	5035/8260B		µg/kg	100	28.2	70-130	60-140	
1,2,4-Trichlorobenzene	5035/8260B		µg/kg	100	20.7	70-130	60-140	
Hexachlorobutadiene	5035/8260B		µg/kg	100	23.3	70-130	60-140	
Naphthalene	5035/8260B		µg/kg	100	14.1	70-130	60-140	
1,2,3-Trichlorobenzene	5035/8260B		µg/kg	100	22.7	70-130	60-140	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 1 B

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
VOCs in Groundwater

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% valid Data)
MATRIX: GROUNDWATER								
VOCs 5ml purges								
Dichlorodifluoromethane	5030B/8260B		µg/l	2	0.11	70-130	70-130	
Chloromethane	5030B/8260B		µg/l	2	0.21	70-130	70-130	
Vinyl Chloride	5030B/8260B		µg/l	2	0.31	70-130	70-130	
Bromomethane	5030B/8260B		µg/l	2	0.34	70-130	70-130	
Chloroethane	5030B/8260B		µg/l	2	0.24	70-130	70-130	
t-Butyl alcohol	5030B/8260B		µg/l	80	25.06	70-130	70-130	
Trichlorofluoromethane	5030B/8260B		µg/l	2	0.21	70-130	70-130	
Diethyl ether	5030B/8260B		µg/l	2	0.38	70-130	70-130	
Acetone	5030B/8260B		µg/l	10	3.23	70-130	70-130	
1,1-Dichloroethene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Di-isopropyl ether	5030B/8260B		µg/l	2	0.27	70-130	70-130	
Methylene Chloride	5030B/8260B		µg/l	5	1.30	70-130	70-130	
Carbon Disulfide	5030B/8260B		µg/l	2	0.22	70-130	70-130	
Methyl-tert-butyl ether	5030B/8260B		µg/l	2	0.42	70-130	70-130	
trans-1,2-Dichloroethene	5030B/8260B		µg/l	2	0.26	70-130	70-130	
1,1-Dichloroethane	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Methyl ethyl ketone	5030B/8260B		µg/l	10	1.68	70-130	70-130	
Ethyl t-butyl ether	5030B/8260B		µg/l	2	0.19	70-130	70-130	
2,2-Dichloropropane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
cis-1,2-Dichloroethene	5030B/8260B		µg/l	2	0.20	70-130	70-130	
t-Amyl methyl ether	5030B/8260B		µg/l	2	0.18	70-130	70-130	
Chloroform	5030B/8260B		µg/l	2	0.14	70-130	70-130	
Bromochloromethane	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Tetrahydrofuran	5030B/8260B		µg/l	2	0.39	70-130	70-130	
1,1,1-Trichloroethane	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,1-Dichloropropene	5030B/8260B		µg/l	2	0.10	70-130	70-130	
Carbon Tetrachloride	5030B/8260B		µg/l	2	0.23	70-130	70-130	
1,2-Dichloroethane	5030B/8260B		µg/l	2	0.23	70-130	70-130	
Benzene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Trichloroethene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
1,2-Dichloropropane	5030B/8260B		µg/l	2	0.20	70-130	70-130	
Bromodichloromethane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
Dibromomethane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
2-Hexanone	5030B/8260B		µg/l	10	2.42	70-130	70-130	
Methyl isobutyl ketone	5030B/8260B		µg/l	10	0.89	70-130	70-130	
cis-1,3-Dichloropropene	5030B/8260B		µg/l	2	0.14	70-130	70-130	
Toluene	5030B/8260B		µg/l	2	0.21	70-130	70-130	
trans-1,3-Dichloropropene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
1,1,2-Trichloroethane	5030B/8260B		µg/l	2	0.13	70-130	70-130	
1,3-Dichloropropane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
Tetrachloroethene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
Dibromochloromethane	5030B/8260B		µg/l	2	0.22	70-130	70-130	
1,2-Dibromoethane	5030B/8260B		µg/l	2	0.21	70-130	70-130	
Chlorobenzene	5030B/8260B		µg/l	2	0.10	70-130	70-130	
1,1,1,2-Tetrachloroethane	5030B/8260B		µg/l	2	0.16	70-130	70-130	
Ethylbenzene	5030B/8260B		µg/l	2	0.16	70-130	70-130	
m,p-Xylene	5030B/8260B		µg/l	2	0.67	70-130	70-130	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% valid Data)
o-Xylene	5030B/8260B		µg/l	2	0.15	70-130	70-130	
Styrene	5030B/8260B		µg/l	2	0.36	70-130	70-130	
Bromoform	5030B/8260B		µg/l	2	0.11	70-130	70-130	
Isopropylbenzene	5030B/8260B		µg/l	2	0.25	70-130	70-130	
1,1,2,2-Tetrachloroethane	5030B/8260B		µg/l	2	0.29	70-130	70-130	
1,2,3-Trichloropropane	5030B/8260B		µg/l	2	0.32	70-130	70-130	
trans-1,4-Dichloro-2-butene	5030B/8260B		µg/l	2	0.67	70-130	70-130	
n-Propylbenzene	5030B/8260B		µg/l	2	0.13	70-130	70-130	
Bromobenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,3,5-Trimethylbenzene	5030B/8260B		µg/l	2	0.14	70-130	70-130	
2-Chlorotoluene	5030B/8260B		µg/l	2	0.11	70-130	70-130	
4-Chlorotoluene	5030B/8260B		µg/l	2	0.11	70-130	70-130	
tert-butylbenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,2,4-Trimethylbenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
Pentachloroethane	5030B/8260B		µg/l	2	0.28	70-130	70-130	
sec-butylbenzene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
p-isopropyltoluene	5030B/8260B		µg/l	2	0.13	70-130	70-130	
1,3-Dichlorobenzene	5030B/8260B		µg/l	2	0.15	70-130	70-130	
1,4-Dichlorobenzene	5030B/8260B		µg/l	2	0.06	70-130	70-130	
n-butylbenzene	5030B/8260B		µg/l	2	0.10	70-130	70-130	
1,2-Dichlorobenzene	5030B/8260B		µg/l	2	0.17	70-130	70-130	
1,2-Dibromo-3-chloropropane	5030B/8260B		µg/l	2	0.25	70-130	70-130	
1,2,4-Trichlorobenzene	5030B/8260B		µg/l	2	0.19	70-130	70-130	
Hexachlorobutadiene	5030B/8260B		µg/l	2	0.31	70-130	70-130	
Naphthalene	5030B/8260B		µg/l	2	0.51	70-130	70-130	
1,2,3-Trichlorobenzene	5030B/8260B		µg/l	2	0.32	70-130	70-130	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 1 C

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
PCBs

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% valid Data)
MATRIX: GROUNDWATER								
PCBS								
PCB 1016	3510C/8082		µg/l	0.5	0.05	67-121	67-121	
PCB 1221	3510C/8082		µg/l	0.5	0.04	NA	NA	
PCB 1232	3510C/8082		µg/l	0.5	0.05	NA	NA	
PCB 1242	3510C/8082		µg/l	0.5	0.05	NA	NA	
PCB 1248	3510C/8082		µg/l	0.5	0.04	NA	NA	
PCB 1254	3510C/8082		µg/l	0.5	0.07	NA	NA	
PCB 1260	3510C/8082		µg/l	0.5	0.06	80-146	80-146	
MATRIX: Wipe								
PCBS								
PCB 1016	3550B/8082		µg/wipe	0.5	0.10	52-127	52-127	
PCB 1221	3550B/8082		µg/wipe	0.5	0.20	NA	NA	
PCB 1232	3550B/8082		µg/wipe	0.5	0.10	NA	NA	
PCB 1242	3550B/8082		µg/wipe	0.5	0.10	NA	NA	
PCB 1248	3550B/8082		µg/wipe	0.5	0.10	NA	NA	
PCB 1254	3550B/8082		µg/wipe	0.5	0.20	NA	NA	
PCB 1260	3550B/8082		µg/wipe	0.5	0.20	68-159	68-159	
MATRIX: Solids/Concrete PQL's assume 100% solids. PQL's will vary based on percent solid.								
PCBS								
PCB 1016	3550B/8082		µg/kg	15	1.65	52-127	52-127	
PCB 1221	3550B/8082		µg/kg	15	5.33	NA	NA	
PCB 1232	3550B/8082		µg/kg	15	1.84	NA	NA	
PCB 1242	3550B/8082		µg/kg	15	1.65	NA	NA	
PCB 1248	3550B/8082		µg/kg	15	2.77	NA	NA	
PCB 1254	3550B/8082		µg/kg	15	5.58	NA	NA	
PCB 1260	3550B/8082		µg/kg	15	1.43	68-159	68-159	
MATRIX: Sediments PQL's assume 30% solids. PQL's will vary based on percent solid.								
PCBS								
PCB 1016	3550B/8082		µg/kg	50	1.65	52-127	52-127	
PCB 1221	3550B/8082		µg/kg	50	5.33	NA	NA	
PCB 1232	3550B/8082		µg/kg	50	1.84	NA	NA	
PCB 1242	3550B/8082		µg/kg	50	1.65	NA	NA	
PCB 1248	3550B/8082		µg/kg	50	2.77	NA	NA	
PCB 1254	3550B/8082		µg/kg	50	5.58	NA	NA	
PCB 1260	3550B/8082		µg/kg	50	1.43	68-159	68-159	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 1 D

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
SVOCs in Soil

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% valid Data)
MATRIX: SOIL PQL's assume 100% solids. PQL's will vary based on percent solid.								
SVOCs:								
Pyridine	3550B/8270C		µg/kg	300	17.1	NA	NA	
N-nitrosodimethylamine	3550B/8270C		µg/kg	300	11.2	NA	NA	
Aniline	3550B/8270C		µg/kg	300	11.4	NA	NA	
Phenol	3550B/8270C		µg/kg	300	22.5	52-86	52-86	
2-Chlorophenol	3550B/8270C		µg/kg	300	19.8	51-84	51-84	
bis(2-chloroethyl) ether	3550B/8270C		µg/kg	300	10.4	NA	NA	
1,3-Dichlorobenzene	3550B/8270C		µg/kg	300	10.8	NA	NA	
1,4-Dichlorobenzene	3550B/8270C		µg/kg	300	10.4	NA	NA	
1,2-Dichlorobenzene	3550B/8270C		µg/kg	300	12.4	NA	NA	
Benzyl Alcohol	3550B/8270C		µg/kg	300	10.3	NA	NA	
2-Methylphenol	3550B/8270C		µg/kg	300	22.2	NA	NA	
bis(2-chloroisopropyl)ether	3550B/8270C		µg/kg	300	10.4	NA	NA	
3&4-Methylphenol	3550B/8270C		µg/kg	300	29.6	NA	NA	
Hexachloroethane	3550B/8270C		µg/kg	300	14	NA	NA	
N-nitroso-di-n-propylamine	3550B/8270C		µg/kg	300	8.9	21-105	21-105	
Nitrobenzene	3550B/8270C		µg/kg	300	13	NA	NA	
Isophorone	3550B/8270C		µg/kg	300	7.5	NA	NA	
2-Nitrophenol	3550B/8270C		µg/kg	300	24	NA	NA	
2,4-Dimethylphenol	3550B/8270C		µg/kg	300	20.2	NA	NA	
Bis(2-chloroethoxy)methane	3550B/8270C		µg/kg	300	7.8	NA	NA	
2,4-Dichlorophenol	3550B/8270C		µg/kg	300	20.3	NA	NA	
Benzoic Acid	3550B/8270C		µg/kg	300	23.0	NA	NA	
1,2,4-Trichlorobenzene	3550B/8270C		µg/kg	300	10.0	42-79	42-79	
Naphthalene	3550B/8270C		µg/kg	300	9.2	NA	NA	
2,6-Dichlorophenol	3550B/8270C		µg/kg	300	16.9	NA	NA	
4-Chloroaniline	3550B/8270C		µg/kg	300	10.2	NA	NA	
Hexachlorobutadiene	3550B/8270C		µg/kg	300	10.7	NA	NA	
4-Chloro-3-methylphenol	3550B/8270C		µg/kg	300	23.7	60-94	60-94	
2-Methylnaphthalene	3550B/8270C		µg/kg	300	14.5	NA	NA	
Hexachlorocyclopentadiene	3550B/8270C		µg/kg	300	19.0	NA	NA	
2,4,6-Trichlorophenol	3550B/8270C		µg/kg	300	77.3	NA	NA	
2,4,5-Trichlorophenol	3550B/8270C		µg/kg	300	36.5	NA	NA	
2-Chloronaphthalene	3550B/8270C		µg/kg	300	12.0	NA	NA	
2-Nitroaniline	3550B/8270C		µg/kg	300	16.3	NA	NA	
2,6-Dinitrotoluene	3550B/8270C		µg/kg	300	10.0	NA	NA	
Dimethyl Phthalate	3550B/8270C		µg/kg	300	10.3	NA	NA	
Acenaphthylene	3550B/8270C		µg/kg	300	8.5	NA	NA	
3-Nitroaniline	3550B/8270C		µg/kg	300	11.5	NA	NA	
Acenaphthene	3550B/8270C		µg/kg	300	11.9	49-91	49-91	
2,4-Dinitrophenol	3550B/8270C		µg/kg	300	29.8	NA	NA	
Dibenzofuran	3550B/8270C		µg/kg	300	10.8	NA	NA	
4-Nitrophenol	3550B/8270C		µg/kg	300	78.8	64-105	64-105	
2,4-Dinitrotoluene	3550B/8270C		µg/kg	300	11.0	50-104	50-104	
2,3,4,6-Tetrachlorophenol	3550B/8270C		µg/kg	300	88.3	NA	NA	
Fluorene	3550B/8270C		µg/kg	300	12.8	NA	NA	
Diethyl Phthalate	3550B/8270C		µg/kg	300	10.2	NA	NA	
4-Chlorophenyl phenyl ether	3550B/8270C		µg/kg	300	10.0	NA	NA	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% valid Data)
4-Nitroaniline	3550B/8270C		µg/kg	300	9.4	NA	NA	
4,6-Dinitro-2-methylphenol	3550B/8270C		µg/kg	300	16.3	NA	NA	
n-nitrosodiphenylamine	3550B/8270C		µg/kg	300	9.0	NA	NA	
Azobenzene	3550B/8270C		µg/kg	300	9.7	NA	NA	
4-Bromophenyl phenyl ether	3550B/8270C		µg/kg	300	12.8	NA	NA	
Hexachlorobenzene	3550B/8270C		µg/kg	300	10.9	NA	NA	
Pentachlorophenol	3550B/8270C		µg/kg	300	17.1	47-108	47-108	
Phenanthrene	3550B/8270C		µg/kg	300	9.7	NA	NA	
Anthracene	3550B/8270C		µg/kg	300	12.4	NA	NA	
Carbazole	3550B/8270C		µg/kg	300	43.3	NA	NA	
Di-n-butyl phthalate	3550B/8270C		µg/kg	300	10.9	NA	NA	
Fluoranthene	3550B/8270C		µg/kg	300	8.4	NA	NA	
Benzidine	3550B/8270C		µg/kg	300	392.2	NA	NA	
Pyrene	3550B/8270C		µg/kg	300	7.5	52-103	52-103	
Butyl benzyl phthalate	3550B/8270C		µg/kg	300	12.3	NA	NA	
Benzo[a]anthracene	3550B/8270C		µg/kg	300	7.8	NA	NA	
Chrysene	3550B/8270C		µg/kg	300	11.9	NA	NA	
3,3'-Dichlorobenzidine	3550B/8270C		µg/kg	300	35.7	NA	NA	
Bis(2-ethylhexyl) phthalate	3550B/8270C		µg/kg	300	18.1	NA	NA	
di-n-octyl-phthalate	3550B/8270C		µg/kg	300	43.6	NA	NA	
Benzo[b]fluoranthene	3550B/8270C		µg/kg	300	36.8	NA	NA	
Benzo[k]fluoranthene	3550B/8270C		µg/kg	300	11.4	NA	NA	
Benzo[a]pyrene	3550B/8270C		µg/kg	300	7.9	NA	NA	
Indeno[1,2,3-cd]pyrene	3550B/8270C		µg/kg	300	6.7	NA	NA	
Dibenz[a,h]anthracene	3550B/8270C		µg/kg	300	8.6	NA	NA	
Benzo(g,h,i)perylene	3550B/8270C		µg/kg	300	10.9	NA	NA	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Table 1 E

LABORATORY ANALYSIS METHODS AND DATA QUALITY OBJECTIVES
SVOCs in Water

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% valid Data)
MATRIX: AQUEOUS								
SVOCs:								
Pyridine	3510C/8270C		µg/l	2	0.24	NA	NA	
N-nitrosodimethylamine	3510C/8270C		µg/l	2	0.29	NA	NA	
Aniline	3510C/8270C		µg/l	2	0.30	NA	NA	
Phenol	3510C/8270C		µg/l	5	0.30	26-43	26-43	
2-Chlorophenol	3510C/8270C		µg/l	5	0.55	43-91	43-91	
bis(2-chloroethyl) ether	3510C/8270C		µg/l	2	0.41	NA	NA	
1,3-Dichlorobenzene	3510C/8270C		µg/l	2	0.50	NA	NA	
1,4-Dichlorobenzene	3510C/8270C		µg/l	2	0.44	23-77	23-77	
1,2-Dichlorobenzene	3510C/8270C		µg/l	2	0.41	NA	NA	
Benzyl Alcohol	3510C/8270C		µg/l	5	0.46	NA	NA	
2-Methylphenol	3510C/8270C		µg/l	5	0.68	NA	NA	
bis(2-chloroisopropyl)ether	3510C/8270C		µg/l	2	0.30	NA	NA	
3&4-Methylphenol	3510C/8270C		µg/l	5	1.02	NA	NA	
Hexachloroethane	3510C/8270C		µg/l	2	0.57	NA	NA	
N-nitroso-di-n-propylamine	3510C/8270C		µg/l	2	0.37	47-91	47-91	
Nitrobenzene	3510C/8270C		µg/l	2	0.28	NA	NA	
Isophorone	3510C/8270C		µg/l	2	0.30	NA	NA	
2-Nitrophenol	3510C/8270C		µg/l	5	0.35	NA	NA	
2,4-Dimethylphenol	3510C/8270C		µg/l	5	0.62	NA	NA	
Bis(2-chloroethoxy)methane	3510C/8270C		µg/l	2	0.27	NA	NA	
2,4-Dichlorophenol	3510C/8270C		µg/l	2	0.76	NA	NA	
Benzoic Acid	3510C/8270C		µg/l	10	1.61	NA	NA	
1,2,4-Trichlorobenzene	3510C/8270C		µg/l	2	0.45	29-81	29-81	
Naphthalene	3510C/8270C		µg/l	2	0.33	NA	NA	
2,6-Dichlorophenol	3510C/8270C		µg/l	5	0.76	NA	NA	
4-Chloroaniline	3510C/8270C		µg/l	2	0.24	NA	NA	
Hexachlorobutadiene	3510C/8270C		µg/l	2	0.34	NA	NA	
4-Chloro-3-methylphenol	3510C/8270C		µg/l	10	0.50	61-99	61-99	
2-Methylnaphthalene	3510C/8270C		µg/l	2	0.28	NA	NA	
Hexachlorocyclopentadiene	3510C/8270C		µg/l	2	0.40	NA	NA	
2,4,6-Trichlorophenol	3510C/8270C		µg/l	5	0.44	NA	NA	
2,4,5-Trichlorophenol	3510C/8270C		µg/l	5	0.63	NA	NA	
2-Chloronaphthalene	3510C/8270C		µg/l	2	0.28	NA	NA	
2-Nitroaniline	3510C/8270C		µg/l	2	0.34	NA	NA	
2,6-Dinitrotoluene	3510C/8270C		µg/l	2	0.23	NA	NA	
Dimethyl Phthalate	3510C/8270C		µg/l	2	0.75	NA	NA	
Acenaphthylene	3510C/8270C		µg/l	2	0.28	NA	NA	
3-Nitroaniline	3510C/8270C		µg/l	2	0.21	NA	NA	
Acenaphthene	3510C/8270C		µg/l	2	0.36	49-91	49-91	
2,4-Dinitrophenol	3510C/8270C		µg/l	5	2.43	NA	NA	
Dibenzofuran	3510C/8270C		µg/l	2	0.26	NA	NA	
4-Nitrophenol	3510C/8270C		µg/l	5	1.13	25-55	25-55	
2,4-Dinitrotoluene	3510C/8270C		µg/l	2	0.37	62-104	62-104	
2,3,4,6-Tetrachlorophenol	3510C/8270C		µg/l	5	0.41	NA	NA	
Fluorene	3510C/8270C		µg/l	2	0.26	NA	NA	
Diethyl Phthalate	3510C/8270C		µg/l	2	0.43	NA	NA	
4-Chlorophenyl phenyl ether	3510C/8270C		µg/l	2	0.14	NA	NA	

ANALYTES OF CONCERN	METHOD	PAL'S	UNITS	PQL	MDL	LCS (% R)	MS/MSD (%R)	Completeness (% valid Data)
4-Nitroaniline	3510C/8270C		µg/l	2	0.14	NA	NA	
4,6-Dinitro-2-methylphenol	3510C/8270C		µg/l	5	1.68	NA	NA	
n-nitrosodiphenylamine	3510C/8270C		µg/l	2	0.21	NA	NA	
Azobenzene	3510C/8270C		µg/l	2	0.46	NA	NA	
4-Bromophenyl phenyl ether	3510C/8270C		µg/l	2	0.27	NA	NA	
Hexachlorobenzene	3510C/8270C		µg/l	2	0.32	NA	NA	
Pentachlorophenol	3510C/8270C		µg/l	10	0.62	56-110	56-110	
Phenanthrene	3510C/8270C		µg/l	2	0.27	NA	NA	
Anthracene	3510C/8270C		µg/l	2	0.38	NA	NA	
Carbazole	3510C/8270C		µg/l	2	0.28	NA	NA	
Di-n-butyl phthalate	3510C/8270C		µg/l	2	0.33	NA	NA	
Fluoranthene	3510C/8270C		µg/l	2	0.26	NA	NA	
Benzidine	3510C/8270C		µg/l	20	19.40	NA	NA	
Pyrene	3510C/8270C		µg/l	2	0.38	56-110	56-110	
Butyl benzyl phthalate	3510C/8270C		µg/l	2	0.78	NA	NA	
Benzo[a]anthracene	3510C/8270C		µg/l	2	0.20	NA	NA	
Chrysene	3510C/8270C		µg/l	2	0.31	NA	NA	
3,3'-Dichlorobenzidine	3510C/8270C		µg/l	20	1.36	NA	NA	
Bis(2-ethylhexyl) phthalate	3510C/8270C		µg/l	2	0.33	NA	NA	
di-n-octyl-phthalate	3510C/8270C		µg/l	2	0.45	NA	NA	
Benzo[b]fluoranthene	3510C/8270C		µg/l	2	0.25	NA	NA	
Benzo[k]fluoranthene	3510C/8270C		µg/l	2	0.50	NA	NA	
Benzo[a]pyrene	3510C/8270C		µg/l	2	0.24	NA	NA	
Indeno[1,2,3-cd]pyrene	3510C/8270C		µg/l	2	0.33	NA	NA	
Dibenz[a,h]anthracene	3510C/8270C		µg/l	2	0.22	NA	NA	
Benzo(g,h,i)perylene	3510C/8270C		µg/l	2	0.30	NA	NA	

PALs= Project Action Level
PQL= Laboratory Practical Quantitation Limit
MDL=Laboratory Method Detection Limit
LCS=Laboratory Control Sample
MS/MSD=Matrix Spike/Matrix Spike Duplicate

Quality Control Tasks:

Quality control samples, Table 2, will be collected in the field and submitted with the samples to the analytical laboratory for the analysis of PCBs. The following QC samples will be collected and analyzed for the concrete sampling: blind duplicates.

Table 2
Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	Organic		No. of Equip. Blanks	Total No. of Samples to Lab*
						No. of MS	No. of MSD		
Concrete	PCBs	Low	4020/8082	212	11	11	11	NA	245

* Soil samples will be evaluated as the concrete results are received.

The SW-846 Method 8082 analysis will perform: initial calibration, continuing calibration, reagent blanks, surrogates, laboratory control spike samples.

Secondary Data:

No secondary data will be used.

Data Management Tasks:

Data will be tabulated to be reviewed and assessed by the project team against the project action level.

Documentation and Records:

Samples collected will be noted in a field logbook. Field notebook, copies of COCs and Airbills will be kept by the field team.

Data Packages:

All data packages will include tabulated sample results and QC sample results.

Assessment/Audit Tasks:

Field sample collection and documentation audit for completeness performed after all of the sampling has taken place.

Data Verification and Validation Tasks:

Laboratory will verify that all data are complete for samples received.

Data Usability Assessment Tasks:

Data placed into tables for comparison to project action level.

3.0 PROJECT QUALITY OBJECTIVES

The data generated from the concrete samples must be of a quality to support the following decisions and requirements:

1. Determine if any PCBs are present in the samples from the concrete pad.

2. Delineate the vertical and horizontal extent of the PCB contamination in the upper portion and the lower portion of the concrete pad, and in the soil below, if necessary.

If/then decisions:

1. If PCBs are present in the concrete above the action level of 10 mg/Kg, then that particular grid will be removed and properly disposed of off site.

2. If PCB contamination exists in bottom concrete sample above 1 ppm, then the soil beneath the concrete pad will be sampled for PCBs at intervals of one foot.

4.0 SAMPLING PROCESS DESIGN

Samples of the concrete pad will be sampled from two depths: from the surface (top 1") and from a depth of approximately eight inches. The rational for this approach is two fold: delineate the degree of potential PCB contamination across the surface of the pad, and determine the depth of the potential PCB contamination within the concrete pad and sample soil beneath the pad if necessary.

Samples from the concrete pad will be collected using a 20' by 20' grid system. The grid system will allow the locations of positive PCB results to be quickly located both on a map and to locate potential soil sampling locations. The grid system will be laid out in several sections of the pad with clear documentation to be provided of each section. If staining is present on the pad, sample locations may need to be adjusted accordingly.

Approximately 250 samples will be collected, one from the top (0-1") and one from the bottom (7"-8") interval at each location. In addition, samples from visibly stained areas will be collected at the sampler's discretion. Care will be taken to avoid "recent" fluid spills that may have emanated from vehicles parked on the pad that may interfere with the PCB analysis. Photographs will be taken to document the sampling effort and the discretionary sample's locations.

4.1 Sampling Procedures and Requirements

Once the grid is laid out, the actual sample location will be cleaned with a broom or cloth to remove any surface debris. A hammer drill will be used to generate the concrete samples. A 1 and ½ inch drill bit will be advanced to one inch below the surface. The dry concrete cuttings

will be collected and labeled as the "top" sample from this location. A Scoopula® will be used to gather the loose concrete from the surface of the pad and scoop out the bottom of the hole. Samples will be collected in 2 oz glass jars with Teflon® lined lids.

The one and ½ inch drill will then be advanced to the depth of seven inches. The hole will be cleaned out with a HEPA vacuum and a one and one-quarter inch drill bit will be used to drill to a depth of an additional one inch. The dry concrete powder will be collected from the hole as with a vacuum device detailed in the EPA SOP for Concrete Sampling, and placed in a sample jar labeled "bottom". No backfilling of the concrete holes will take place. If "breakthrough" of the concrete takes place, the depth of the concrete will be measured and a one-inch sample from the bottom of the pad will be collected from an immediately adjacent hole drilled next to the original.

Drill bits and Scoopulas® will be decontaminated between locations using Alconox® detergent and distilled water. The tools will be rinsed with distilled water and dried on paper towels.

Table 3
Sampling Locations, Sampling and Analysis Method/SOP Requirements Table

Sampling Location	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc. Level	No. of Samples [2]	Sampling SOP	Analytical Method/SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
A1 – Z99	A1 – Z99 Top1/2	CT	0 – 1.0 inches	PCBs	Low	500	USEPA Region I concrete sampling	SW-846 4020/8082	20 – 25 g.	1 – 2 oz. Jar	Cool 4°C ± 2°C	7 days sample prep 40 days to analysis
A1 – Z99	A1 – Z99 Bot1/2	CT	7.0 – 8.0 inches[1]	PCBs	Low	500	USEPA Region I concrete sampling	SW-846 4020/8082	20 – 25 g.	1 – 2 oz. Jar	Cool 4°C ± 2°C	7 days sample prep 40 days to analysis

1- Bottom depth will be evaluated at each location.
2- The number of potential soil sampling locations will be based on the PCB results from the concrete samples.

4.2 Sample Handling, Tracking and Custody Requirements

Samples will be shipped on ice via overnight courier to the Navy approved laboratory. Strict Chain of Custody procedures will be followed. All coolers will be sealed with strapping tape and custody seals.

5.0 FIXED LABORATORY ANALYTICAL METHOD REQUIREMENTS

Samples will be analyzed for PCBs using the USEPA SW-846 Statement of Work for Organic Analysis. The analytical method will be modified for the limited amount that will be available and the action level requirements of the project. Approximately 10 grams of the pulverized concrete sample will be prepared for the analysis. All samples will be analyzed onsite via Method 4020, Immunoassay Screening. Approximately 5% of the samples will be analyzed using a dual capillary column Gas Chromatograph/Electron Capture Detector (GC/ECD) via Method 8082

Table 4

Fixed Laboratory Analytical Method/SOP Reference Table

Reference Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive Or Screening Data	Region I NESTS Method Code	Analytical Parameter	Instrument	Modified for Project Work Y or N
L-1	Analytics Environmental Laboratory.	SW-846 Method 8082	Definitive	8082	PCBs	GC/ECD	Y - Amount used for sample preparation is 10 grams.

6.0 QUALITY CONTROL REQUIREMENTS

Quality control samples will be collected in the field and submitted to the laboratory for analysis with the field samples. The laboratory will analyze the quality control samples collected in the field and described in the analytical SOW and verify that the results meet the requirements listed in Tables 5, field QC samples and Table 6, laboratory QC samples. If the quality control sample results are determined not meet the criteria listed in Tables 5 and 6, corrective action will be initiated to correct the error and/or to take appropriate action to note potential bias in the data.

Table 5
Field Sampling QC Table

Sampling SOP*:	Region I Concrete Sampling
Medium/Matrix:	Concrete
Analytical Parameter:	PCBs
Concentration Level:	Low
Analytical Method/SOP Reference:	Method 4020/8082
Field Sampling Organization:	Foster Wheeler
No. of Sample Locations:	500

Field QC:	Frequency/ Number	Method/ SOP QC Acceptance Limits[2]	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria[3]
Equipment Blanks/ Rinsate Blanks	NA	< Laboratory Reporting Limit	Evaluate with sample data	Data Reviewer	Accuracy/ Bias – contamination	No Target Compounds > QL
Cooler Temperature Blanks	1/cooler shipment	4°C ∀ 2°C	Contact field team Field team to add more ice for shipments	Foster Wheeler Field Team	Accuracy/ Bias - preservation	4°C ± 2°C
Field Duplicate Pairs (Duplicate Subsamples)	5 %	< 50% D	Evaluate all Duplicate samples	Data Reviewer	Precision	RPD < 50% where target compounds in both field samples are > QL

Table 6
Fixed Laboratory Analytical QC Sample Table

Medium/Matrix Concrete
Sampling SOP Region I Concrete Sampling
Analytical Parameter PCBs
Concentration Level Low
Analytical Method/ SOP Reference Method 8082
Laboratory Name Analytics Environmental Laboratory
No. of Sample Locations 25

Laboratory QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria[3]
Method Blank	1/20 samples	No target compounds > QL	Reanalyze /Reextract	Analytics Env. Lab	Accuracy/ Bias - contamination	No target compounds > QL
Reagent Blank	1/20 samples	No target compounds > QL	Reanalyze /Reextract	Analytics Env. Lab	Accuracy/ Bias - contamination	No target compounds > QL
Laboratory Matrix Spike	1/20 samples	NA	Reanalyze	Analytics Env. Lab	Accuracy Precision	
Matrix Spike Duplicates	1/20 samples	NA	Reanalyze	Analytics Env. Lab	Accuracy Precision	
Surrogates	Tetrachloro-m-xylene Decachlorobiphenyl	30 – 150 % recovery	Reextract	Analytics Env. Lab	Accuracy	Rec. 30 – 150 %

7.0 DOCUMENTATION, RECORDS AND DATA MANAGEMENT

Table 7 lists the project documentation and records that will be kept during the sampling and analysis program by the field sampling team and the analytical laboratory.

The field sampling team will maintain a field file during the collection of samples. The field file will include field notes kept in a field logbook, copies of the COCs and air bills for samples submitted to the laboratory.

The analytical laboratory will maintain logs for sample receipt, custody and tracking. The laboratory will record the standards preparation so as to be able to trace the origin of the standards used during the sample preparation and analysis. Sample preparation logs also will be kept. Maintenance, calibration and run logs will be kept for all of the instruments that are used to perform the analysis of samples. Sample results will be reported on sample result forms and will be submitted in a data package containing QC sample result forms, raw data and a data package completeness check form. All logs, raw data and reports will be kept on file by the laboratory for a minimum of 10 years from the date of project completion.

Table 7

Project Documentation and Records Table

Sample Collection Records	Fixed Laboratory Records
Field Notes	Sample Receipt logs, Laboratory custody and tracking records
COC Records	Standards Tracability logs
Air Bills	Instrument Calibration Logs
	Sample Preparation Logs
	Instrument Run Logs
	Instrument Maintenance Logs
	Sample Result Forms
	QC sample Result Forms
	Raw Data
	Data Packages Completeness Check Form

8.0 VERIFICATION AND VALIDATION REQUIREMENTS/PROCEDURES

To document data verification the procedures listed in Table 8 will be followed during the collection and the analysis of samples.

The field team will verify the information on the COC(s) for each cooler that is shipped to the analytical laboratory to ensure accuracy and completeness. Copies of all sample shipping documents will be kept in the project field file. The laboratory will complete a data completeness audit of all data packages prior to their submittal. A copy of the audit will be included in the data package and in the laboratory project file.

Table 8
DATA VERIFICATION PROCESS

Verification Task	Description	I/E	Responsible for Verification (Name, Organization)
COC and shipping forms	Chain-of –custody forms and shipping documentation will be reviewed by the sampling team after completion and prior to shipping of the samples to verify the contents of the packed sample cooler(s). When the check is complete, the field team will sign the COC(s), a copy of the COC(s) will be kept with the field files, and the original COC(s) will be placed inside a sealable bag and taped to the inside of the sample cooler.	I	Foster Wheeler
Laboratory Data	All laboratory data packages will be verified by the laboratory performing the analyses for completeness prior to the submittal of the final data package(s). A completeness audit form (CSF) will be completed and included in the data package to document that the data package(s) are complete.	E	Analytics Environmental Laboratory.

I/E: Internal/External

9.0 REFERENCES

Environmental Protection Agency. 1999. Region I, New England Compendium of Quality Assurance Project Plan Requirements and Guidance, Final. Prepared by USEPA-New England Quality Assurance Unit Staff, Office of Environmental Measurement and Evaluation, Lexington, MA. October 1999.

Environmental Protection Agency. 1997. Region I, Draft Standard Operating Procedure for Concrete Sampling in the Field. Prepared by USEPA-New England Quality Assurance Unit Staff, Office of Environmental Measurement and Evaluation, Lexington, MA. December 1997.

APPENDIX C

STANDARD OPERATING PROCEDURES

PROCEDURE	**ANALYTICS** SOP DESCRIPTION	**ANALYTICS** SOP NUMBER
PCB IN SOIL, CONCRETE, SEDIMENT ANALYSIS		
PCB Analysis EPA SW-846 8082	Laboratory Method for Determination of PCBs in All Matrices	QA-P012
PCB Extraction EPA SW-846 3550B	Sonication Extraction For the Determination of Pesticides and PCBs in Solid Samples	QA-P006
PCB Clean-up EPA SW-846 3665A	Acid Clean-up for PCBs	QA-P009
Solid Percent EPA SW-846 Method 3550B	Laboratory Method the Determination of Percent Solids	QA-M002

PCB Wipe		
PCB Analysis EPA SW-846 8082Analysis	Laboratory Method for Determination of PCBs in All Matrices	QA-P012
PCB Extraction EPA SW-846 3550B	PCB Wipe Sample Preparation	QA-P011
PCB Clean-up EPA SW-846 3665A	Acid Cleanup For PCB	QA-P009

Waste Characterization-Solid		
PCB Extraction EPA SW-846 3550B	See Above	QA-P012
PCB Clean-up EPA SW-846 3665A	See Above	QA-P006
PCB Analysis EPA SW-846 8082	See Above	QA-P009
VOC Preparation EPA SW-846 5035	Preparation of Solid Samples (Medium Level) for Volatile Organic Analysis	QA-V002
VOC Analysis EPA SW-846 8260B	Laboratory Method for the Determination of Volatile Compounds-Capillary Column, GC/MS Detection	QA-V005
SVOC Extraction EPA SW-846 3550B	Sonication Extraction For the Determination of Semi-Volatiles in Solid Matrices	QA-S007
SVOC Analysis EPA SW-846 8270C	Laboratory Method for the Determination of Acid and Base/Neutrals in All Matrices	QA-S005
Reactive Cyanide &Reactive Sulfide EPA SW-846 Sec. 7.3.3.2, Sec 7.3.4.2	Laboratory Method for the Determination of Reactive Sulfide and Cyanide	QA-M010
Corrosivity EPA SW-846 9045	Laboratory Method for the Determination of pH in Solid and Liquid Samples	QA-M006
Ignitability EPA SW-846 1010	Laboratory Method for the Determination of Flashpoint Using the Pensky-Martin Closed Cup Tester and the Ignitability Charateristic Using a Direct Flame	QA-M005
Solid Percent EPA SW-846 Method 3550B	Laboratory Method the Determination of Percent Solids	QA-M002

PROCEDURE	**ANALYTICS** SOP DESCRIPTION	**ANALYTICS** SOP NUMBER
WASTE CHARACTERIZATION-LIQUID		
PCB Extraction EPA SW-846 3510C	Separatory Funnel Liquid/Liquid Extraction for the Determination of Pesticides and PCBs in Aqueous Samples	QA-P002
PCB Clean-up EPA SW-846 3665A	See Above	QA-P009
PCB Analysis EPA SW-846 8082	See Above	QA-P012
VOC Preparation EPA SW-846 5030B	Laboratory Method for Sample Preparation and Extraction of Volatile Organic Compounds by Purge and Trap	QA-V009
VOC Analysis EPA SW-846 8260B	See Above	QA-V005
SVOC Extraction EPA SW-846 3510C	Separatory–Funnel Liquid/Liquid Extraction For the Determination of Semi-Volatiles in Aqueous Samples	QA-S001
SVOC Analysis	See Above	QA-S005
Reactive Cyanide &Reactive Sulfide EPA SW-846 Sec. 7.3.3.2, Sec 7.3.4.2	See Above	QA-M010
Corrosivity EPA SW-846 9045	See Above	QA-M006
Ignitability EPA SW-846 1010	See Above	QA-M005

PROCEDURE	**FOSTER WHEELER** SOP DESCRIPTION	**FOSTER WHEELER** SOP NUMBER
GeoProbe Sampling	Hollow-Stem Auger Drilling/Split-Barrel Sampling/ Direct Push Sampling	A-1

ANALYTICS ENVIRONMENTAL LABORATORY

STANDARD OPERATING PROCEDURES

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RE: Gould Island Sampling and Analysis Plan
Analytics SOPs

Dear Mr. Woodworth;

Enclosed please find the Standard Operating Procedures (SOPs) required for your Sampling and Analysis Plan for the Gould Island Project. Attached is a table that describes the SOPs in this submittal.

If you have any questions or I can be of further assistance, please do not hesitate to contact me.

Sincerely,
ANALYTICS Environmental Laboratory, LLC

Stephen L. Knollmeyer
Laboratory Director

LIST OF STANDARD OPERATING PROCEDURES (SOPS)

Procedure	**ANALYTICS** SOP Description	**ANALYTICS** SOP Number
PCB in Soil, Concrete, Sediment Analysis		
PCB Analysis EPA SW-846 8082	Laboratory Method for Determination of PCBs in All Matrices	QA-P012
PCB Extraction EPA SW-846 3550B	Sonication Extraction For the Determination of Pesticides and PCBs in Solid Samples	QA-P006
PCB Clean-up EPA SW-846 3665A	Acid Clean-up for PCBs	QA-P009
Solid Percent EPA SW-846 Method 3550B	Laboratory Method the Determination of Percent Solids	QA-M002

PCB Wipe		
PCB Analysis EPA SW-846 8082Analysis	Laboratory Method for Determination of PCBs in All Matrices	QA-P012
PCB Extraction EPA SW-846 3550B	PCB Wipe Sample Preparation	QA-P011
PCB Clean-up EPA SW-846 3665A	Acid Cleanup For PCB	QA-P009

Waste Characterization-Solid		
PCB Extraction EPA SW-846 3550B	See Above	QA-P012
PCB Clean-up EPA SW-846 3665A	See Above	QA-P006
PCB Analysis EPA SW-846 8082	See Above	QA-P009
VOC Preparation EPA SW-846 5035	Preparation of Solid Samples (Medium Level) for Volatile Organic Analysis	QA-V002
VOC Analysis EPA SW-846 8260B	Laboratory Method for the Determination of Volatile Compounds-Capillary Column, GC/MS Detection	QA-V005
SVOC Extraction EPA SW-846 3550B	Sonication Extraction For the Determination of Semi-Volatiles in Solid Matrices	QA-S007
SVOC Analysis EPA SW-846 8270C	Laboratory Method for the Determination of Acid and Base/Neutrals in All Matrices	QA-S005
Reactive Cyanide &Reactive Sulfide EPA SW-846 Sec. 7.3.3.2, Sec 7.3.4.2	Laboratory Method for the Determination of Reactive Sulfide and Cyanide	QA-M010
Corrosivity EPA SW-846 9045	Laboratory Method for the Determination of pH in Solid and Liquid Samples	QA-M006
Ignitability EPA SW-846 1010	Laboratory Method for the Determination of Flashpoint Using the Pensky-Martin Closed Cup Tester and the Ignitability Charateristic Using a Direct Flame	QA-M005
Solid Percent EPA SW-846 Method 3550B	Laboratory Method the Determination of Percent Solids	QA-M002

LIST OF STANDARD OPERATING PROCEDURES (SOPS)

Procedure	**ANALYTICS** SOP Description	**ANALYTICS** SOP Number
Waste Characterization-Liquid		
PCB Extraction EPA SW-846 3510C	Separatory Funnel Liquid/Liquid Extraction for the Determination of Pesticides and PCBs in Aqueous Samples	QA-P002
PCB Clean-up EPA SW-846 3665A	See Above	QA-P009
PCB Analysis EPA SW-846 8082	See Above	QA-P012
VOC Preparation EPA SW-846 5030B	Laboratory Method for Sample Preparation and Extraction of Volatile Organic Compounds by Purge and Trap	QA-V009
VOC Analysis EPA SW-846 8260B	See Above	QA-V005
SVOC Extraction EPA SW-846 3510C	Separatory--Funnel Liquid/Liquid Extraction For the Determination of Semi-Volatiles in Aqueous Samples	QA-S001
SVOC Analysis	See Above	QA-S005
Reactive Cyanide &Reactive Sulfide EPA SW-846 Sec. 7.3.3.2, Sec 7.3.4.2	See Above	QA-M010
Corrosivity EPA SW-846 9045	See Above	QA-M006
Ignitability EPA SW-846 1010	See Above	QA-M005

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Laboratory Method For The Determination Of Solid Percent

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Approved By:

QA Officer: _____ Date: 11/06/01

Laboratory Director: _____ Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Laboratory method for the determination of Percent Solid.

Reference: Test Methods For Ultrasonic Extraction Method 3350B, Revision 2 December 1996.

Comprehensive Quality Assurance Plan for Analytics Environmental laboratory, LLC, January 2001.

2. Scope and Application

This method is used to determine the percent solid of a sample. All solid samples are reported on a dry weight basis unless requested otherwise.

3. Interferences

3.1 Possible method interferences would be low flashpoint, plastic substances or materials that will melt, materials that change state or become hazardous materials with exposure to heat. Any materials that are suspected to present a danger shall be brought to the attention of Laboratory Director for determination of the need to proceed.

4. Safety

Each sample shall be treated as a potential health hazard. Potential exposure to hazardous substances should be minimized by wearing safety goggles, lab coat, gloves, and working in a fume hood. The oven must be vented through a fume hood to avoid inhaling unknown substances released by heating the sample.

5. Method Summary

Approximately 10 grams of a pre-weighed solid sample is heated at a minimum of 105°C (not to exceed 110°C) for not less than 4 hours. The sample is then re-weighed and the Percent Solid is calculated based on the difference between the wet and dry weights.

6. Sample Collection, Handling and Preservation

6.1 All samples should be collected in glass jar with a screw top Teflon lid.

6.2 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling instructions.

6.3 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and login procedures.

6.4 Solid percent determination must be performed within 14 days of collection.

7. Definitions

7.1 °C = Degrees Celsius

7.2 NIST= National Institute of Standards & Technology

8. Apparatus and Materials

8.1 Aluminum weighing pan

8.2 Balance capable of accurately weighing to 0.01 grams, calibrated per QA-M009.

8.2 Oven - 105°C heating capacity

8.4 Fume Hood

8.5 Spatula

8.6 Desiccators Cabinet

8.7 Thermometer (-20 - 150°C), checked per QA-M018

9. Standards and Reagents

7.1 Silica gel-6-16 mesh indicating desiccant

10. Procedure

10.1 Ascertain if the sample is available for percent solid determination. Containers for samples requiring volatile organic analysis are sealed with red tape and those requiring flash point are sealed with purple tape to ensure that the samples remain headspace free prior to analysis. Percent Solid determination must not be performed on these samples until the seal has been broken, thus indicating that volatile organic analysis or flashpoint has been performed. Percent Solid determination on samples that are not sealed with tape may be performed at any time. Some sample matrices are not amenable to Percent Solid determination, for example: oil and plastic. Note in the Percent Solid notebook that the sample was not amenable to this procedure.

10.2 Percent Solid Notebook

All information pertinent to the Percent Solid determination is recorded in a bound paginated laboratory notebook. Header information will include the following: Initial oven temperature and start time, and final oven temperature and end time. Pertinent data are entered in a column format. Column format should include the following headings: Date, sample number, pan weight, initial sample and pan weight, wet weight, dry sample and pan weight, dry weight, Percent Solid, and analyst (Figure 1). All weights shall be recorded to the nearest 0.01 grams.

10.3 Calibrate balance per QA-M009.

10.4 Label an aluminum pan with the corresponding sample number. Tare the balance. Place the pan on the balance and record the "pan weight".

10.5 Weigh approximately 10 grams of "wet" sample into pan and record "initial sample and pan weight". Determine the "wet weight" of the sample using Equation 1 and record in the notebook.

10.6 Record the initial oven temperature to the nearest degree Celsius **(being sure to use any correction factor)** and the time the temperature is taken.

10.7 Place pan with sample into oven and heat at a temperature of at least 105°C for a minimum of 4 hours.

10.8 Prior to removing samples from the oven record the final oven temperature **(be sure to use any correction factor)** to the nearest degree Celsius and the time the temperature is taken.

10.9 Remove the sample from the oven and place in the desiccator to cool to room (approximately 10 minutes). The silica gel in the desiccators must be blue. If not, replace with fresh silica gel.

10.10 Weigh the sample and the pan and record this number under "dry sample and pan weight". Determine the dry weight using Equation 2 and record in the notebook.

10.11 Determine the Percent Solid using Equation 3 and record in the notebook.

10.12 Dispose of sample according to QA-W001.

10.13 Calculations

10.13.1 EQUATION 1:
Wet weight = (Initial pan and sample weight) - (pan weight)

10.13.2 EQUATION 2:

Dry weight = (Dry sample and pan weight) - (pan weight)

10.13.3 EQUATION 3:
Percent Solid = (Dry weight / Wet weight) x 100

10.14 Upon completion of the logbook the analyst must enter the results into the computer.

10.14.1 Open "ANALYTICS LLC" then "Login LLC", "ANALYSIS" under analysis select "begin Solid percent". Enter required information at the prompts. When the required fields are entered "PANORAMA" will calculate the solid percent. The Analyst should verify that the hand calculated value and "PANORAMA's" values match.

11. Quality Control

11.1 A Thermometer checked annually against a NIST thermometer, shall be used in the oven.

11.2 The oven temperature is measured and the time is recorded prior to introduction of the samples and before removal to ensure an appropriate temperature and time is maintained.

11.2 The balance is checked for calibration at a minimum of daily.

12.0 Pollution Prevention and Waste Management

12.1 See SOP W001 for handling of waste and unused portions of sample.

Figure 1

SOILD PERCENT LOGBOOK

Date	Sample No.	Pan Wgt.	Init. Sample & Pan Wgt.	Wet Wgt. (A)	Dry Sample & Pan Wgt.	Dry Wgt. (B)	%Solid (B/A)*100	Analyst

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Laboratory Method for the Determination of Flashpoint Using the Pensky-Martens Closed Cup Tester and the Ignitability Characteristic Using a Direct Flame

Approved By:

QA Officer: Melissa A Sullivan Date: 11/7/01

Laboratory Director: Steve L. Knollyn Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Laboratory method for the Determination of Flashpoint Using the Pensky-Martens Closed Cup Tester and the Ignitability Characteristic Using a Direct Flame.

EPA Reference: Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Method 1010, Revision 0, September 1986

D 93-80, Test Methods for Flash Point by Pensky-Martens Closed Tester, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103, 04.09,1986

New Hampshire Department of Environmental Services, Ignitability Characteristic for a Soil Sample, April, 1994.

Comprehensive Quality Assurance Plan for Analytics Environmental Laboratory, LLC, January 2001

2. Scope and Application

This method is used to evaluate samples for flashpoint and ignitability.

3. Interferences

Not Applicable

4. Safety

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard. Exposure should be minimized by wearing safety goggles, solvent-resistant gloves, and a laboratory coat when standards or samples are handled, and by working in a fume hood. Material Safety Data Sheets for all of the reagents used in this method are on file in the laboratory and should be read by any personnel prior to initiating this method. All flammable materials must be removed from the immediate work area (hood) as an open flame with a propane source is used throughout the test. A fire extinguisher should be within quick and easy access of the test site, as well as any other fire safety equipment the tester may deem appropriate; i.e. fire blanket, face shield, etc.

5. Method Summary

To determine flashpoint, the sample is heated at a slow, constant rate. A small flame is directed into the cup at regular intervals. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite. To determine ignitability, a flame is applied directly to the sample. A sample exhibits the characteristic of ignitability if it burns persistently and vigorously.

6. Sample Collection, Handling and Preservation

6.1 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling.

6.2 Samples should be refrigerated (1.1°-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-l001 for laboratory receipt and log-in procedures

7. Definitions

7.1 °F = Degrees Fahrenheit

7.2 °C = Degrees Celsius

7.3 g = grams

7.4 ml = milliliter

8. Apparatus and Materials

8.1 Pensky-Martens Flash Tester Precision Scientific 74537, 74540 or equivalent

8.2 Propane source

8.3 Scoops or spatulas

8.4 Matches or other source of ignition

8.5 Timer - to check rate of temperature increase

8.6 Flexible rubber tubing

8.7 Mercury Thermometer (°F) encompassing the temperature range from 60°F to 165°F

8.8 Non-combustible surface area

8.9 400 ml beakers

8.10 Flashpoint logbook

9. Standards and Reagents

9.1 p-Xylene - Reagent grade (Flash Point = 27°C/81°F)

9.2 Silica sand

10. Procedure

10.1 Samples must be analyzed within 14 (fourteen) days of collection. Inspect the sample container to see that it has not been opened and that there is no head space in the container. The container must be glass. Plastic and polyethylene containers must not be used as volatiles may diffuse through the material. If any of these conditions exist, notify the supervisor **and** make a note in the Miscellaneous sample prep notebook, as well as on the chain of custody (if it hasn't already been noted).

10.2 All precautions must be observed to avoid loss of volatiles. Flashpoint should always be the first test performed on the sample. The sample container must not be opened until the analyst is ready to perform the test. Keep the time that the sample is exposed to air to a minimum.

10.3 Data will be recorded in the bound, paginated Flashpoint Logbook (Figure 1) under the following columns: Date, AEL Sample #, Analyst, Matrix, Range (temperature in °F), Flashpoint, Av. Flash (duplicate analysis must agree within 4° C, Ignitability.

10.4 Inspect tubing for signs of wear or cracking. Replace tubing if necessary. Connect tubing from propane source to flash point tester .

10.5 Prior to analyzing any samples, a p-Xylene standard must be tested. The flashpoint of the p-Xylene must be 81°F ±2°F to pass. Record the temperature in the Flashpoint Logbook (Figure 1). After the p-Xylene passes, a blank consisting of clean silica sand must be analyzed yielding no flash to prove that all residual volatiles have been removed from the flashpoint system. If the blank flashes, successive blanks must be run until no flashes are observed before analyzing client samples.

10.6 Thoroughly clean and dry all parts of the flash cup before starting each test. Be sure test cup is not warmer than room temperature before adding sample. Transfer sample to test cup and fill to line inside of cup. (For liquid samples it is not necessary to fill the cup all the way to the line if insufficient sample is provided.) Immediately install test cup on flashpoint tester, place flashpoint cover on top and tighten mounting posts.

10.7 Insert thermometer in test cup until stop collar seats thermometer. Mix sample in a downward direction using the stirrer.

10.8 Turn on propane and light the test flame. Adjust flame to approximately 1/4" in height. Apply the test flame to the test cup by opening the shutter. The shutter mechanism should operate smoothly enough to allow the test flame to enter the test cup in about 1/2 second. The test flame should be exposed to the test cup for at least a full second. Record the temperature at the first application of the test flame. Initial temperature should not be warmer than room temperature (75°F maximum).

10.9 Plug in the flashpoint tester and set dial to 55-60. Rate of temperature increase should not exceed 9-11°F / minute. Check the rate of temperature increase at least once with the timer for one minute during each sample run.

10.10 Continue to apply the test flame every 4°F until a flash occurs or until the temperature reaches 165°F. If application of the test flame causes a distinct flash, record the temperature at which it flashed and discontinue heating. If temperature reaches 165°F (+/- 1°F) without flashing, apply test flame for a final time. If no flashed occurred, then the sample has a flashpoint >165°F.

***Note**: A flash has occurred when a large distinct green flame propagates itself away from the test flame and over the entire surface of the sample. This is not to be confused with a 'water halo', which is a dull bluish or greenish halo that may surround test light.

10.11 **If a flash occurs the sample must be re-tested by a different analyst.** A blank must be run after every sample that flashes to prove that the flashpoint system is clean. Fresh sample must be used for the re-test since all the volatiles will have been consumed at the time of ignition. If a flash occurs on the second trial the average of the two temperatures shall be reported. Duplicate measurements should agree within 4°F. If no flash occurs the second time or readings differ by ±4°F, another test must be performed to confirm a flash or no flash. No flash on the third test would presume a 'water halo' on the first test. If the third test is not within ±4°F of the other trials, then more tests must be performed until two readings agree within ±4°F.

10.12 Results shall be recorded in the Flashpoint Logbook (Figure 1) and filed in the appropriate client folder.

10.13 If a sample tests positive for a flashpoint an ignitability test must be performed, proceed to section 10.14. If it is determined that a sample has a flashpoint greater that 165° proceed to section 11.

10.14 Procedure - Ignitability

10.14.1 To determine ignitability, a fresh aliquot of solid sample (approximately 50 g) is placed on a non-combustible surface such as a sheet of aluminum foil.

10.14.2 Flame is applied directly to the fresh aliquot.

10.14.3 If no flash or flame occurs, report ignitability as PASS. If a flash does occur, place a 400 ml beaker over the solid to determine if the burn can be sustained. If the burn is vigorous and persistent, report ignitability as FAIL and document your observation in the miscellaneous sample prep notebook. If the flame is extinguished, remove the beaker and determine if the sample will reignite. If the sample does not reignite, report ignitability as Pass. If the sample reignites, report ignitability characteristic as FAIL and document your observation in the miscellaneous sample prep notebook. Refer to attachment 1.

10.14.4 Make a copy of the notebook page and file in the appropriate client folder.

11. Quality Control

11.1 The flashpoint tester is calibrated at the beginning of each test session. The flash point of p-Xylene is tested by filling the test cup to about 1/2" and proceeding in the same manner as a standard flashpoint test. The acceptable values are 81°F +/- 2°F. The temperature of the flashpoint of p-Xylene is recorded in the "Flashpoint Calibration" log as well as the miscellaneous sample prep notebook.

11.2 **A blank needs to be analyzed after each observed flashpoint to show that there are no residues that will yield a false positive.** Silica sand will be used for blanks. One blank will be run at least every 10 samples, following the procedures as described in Section 8. In the event that a blank flashes, successive blanks are analyzed in the cup until the blank no longer flashes.

11.3 All flashes are confirmed by a second analyst, except the p-Xylene standard.

Figure 1

EPA Method 1010 (FLASHPOINT)

DATE: _____

	AEL Sample #	Analyst	Matrix	Range (°F)	Flashpoint	Avg. Flash**	Ignitability
	p-Xylene*		STD				
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							

*Criteria for flashpoint of p-Xylene=81°F +/- 2°F

Duplicate analysis must agree within 4°F. Report average of the two results.

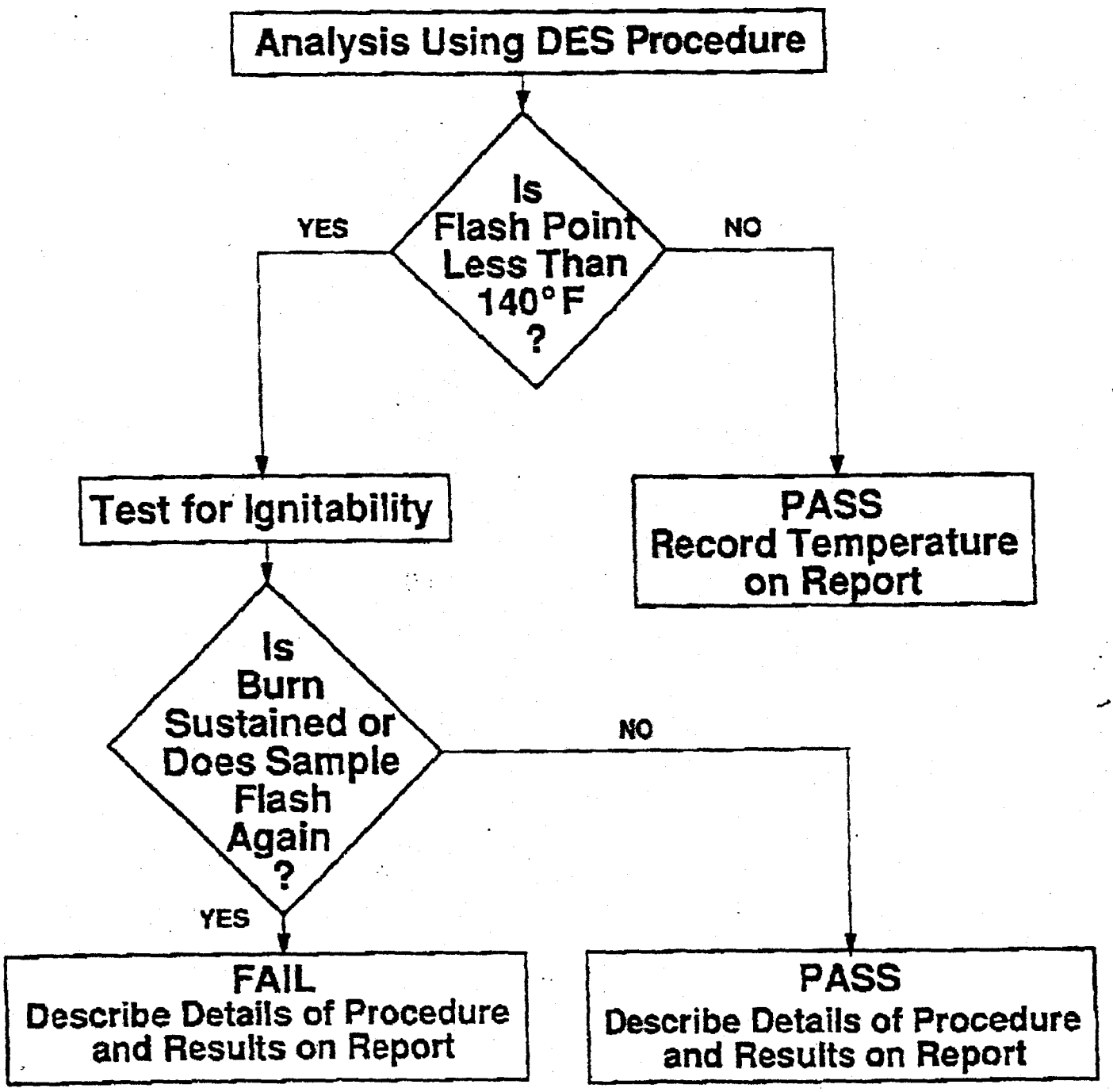
NR=Not required

ATTACHMENT 1

NEW HAMPSHIRE DEPARTMENT OF ENVIRONMENTAL SERVICES

IGNITABILITY CHARACTERISTIC FOR A SOIL SAMPLE

ie ..owing flow chart shows the decision path to take when determining whether a soil sample exhibits the ıitability characteristic and how to report the result. Since there is no available documented methodology for ıitability of soils, DES is providing the attached guidance procedure which may be used as a test method. This ocedure follows the Pensky-Martens Closed Cup method for flashpoint (EPA reference SW846, Method 1010) llowed, if necessary, by a determination of ignitability characteristic using a flame test. The owner/ consultant ıould be advised that the methodology serves as guidance only and that they may, with prior DES approval, use :her equivalent testing methods to determine results.



The ignitability characteristic for all matrices is described in EPA's "Test Methods for Evaluating Solid Waste" SW-846, chapter seven.

April, 1994

nh des lab 6032712997 02/24/00 01:50P P.002

NHDES HAZ. WASTE RULES

P2

ATTACHMENT 1

(b) For purposes of Part Env-Wm 403 and Env-Wm 405.03, the division shall consider a sample obtained using a sampling method appropriate for the waste as specified in 40 CFR 261 Appendix I, 7-1-89 edition, to be a representative sample within the meaning of Env-Wm 110.01(b).

Env-Wm 403.02 Hazardous Waste Numbers.

(a) The hazardous waste numbers assigned by EPA for characteristic hazardous wastes shall be as set forth in Env-Wm 403.03, Env-Wm 403.04, Env-Wm 403.05, and Env-Wm 403.06.

(b) EPA hazardous waste numbers or NH hazardous waste numbers for characteristic hazardous wastes shall be used in complying with the notification, labeling, manifest and recordkeeping and reporting requirements of Chapters Env-Wm 500, Env-Wm 600, Env-Wm 700, and Env-Wm 800.

Env-Wm 403.03 Ignitability.

(a) A waste that exhibits the characteristic of ignitability but is not listed as a hazardous waste in Env-Wm 402.04(b), Env-Wm 402.05(b), Env-Wm 402.06(a) or Env-Wm 402.07 and is not a mixture under Part Env-Wm 404.01(a) shall be assigned the EPA hazardous waste number of D001.

(b) A waste shall be classified under these rules as ignitable if a representative sample of the waste has any of the following characteristics:

(1) It is a liquid, other than an aqueous solution containing less than 24 percent alcohol by volume, and has a flash point less than 60°C (140°F) as determined by a Pensky-Martens Closed Cup Tester, using the test method specified in ASTM Standard D-93-79 or D-93-80, incorporated by reference at Env-Wm 110.03, or a Setaflash Closed Cup Tester, using the test method specified in ASTM Standard D-3278-78, or as determined by an equivalent test method approved by EPA pursuant to 40 CFR 260.20 and 260.21, 7-1-89 edition;

(2) It is not a liquid and is capable, under standard temperatures and pressure, of causing fire through friction, absorption of moisture, or spontaneous chemical changes, and when ignited, it burns so vigorously and persistently that it creates a hazard;

(3) It is an ignitable compressed gas as defined by the U.S. Department of Transportation at 49 CFR 173.300, 7-1-89 edition; and as determined by the test method described in that regulation or an equivalent test method approved by the Administrator pursuant to 40 CFR 260.20 and

260.21, 7-1-89 edition; or

(4) It is an oxidizer as defined by 49 CFR 173.151, 10-1-92 edition (U.S. Department of Transportation).

Env-Wm 403.04 Corrosivity.

(a) A waste that exhibits the characteristic of corrosivity but is not listed as a hazardous waste in Env-Wm 402.04(b), Env-Wm 402.05(b), Env-Wm 402.06(a) or Env-Wm 402.07(a) and is not a mixture under Part Env-Wm 404.01(a) shall be assigned the EPA hazardous waste number of D002 if it meets the criteria set forth in Env-Wm 403.04(b)(1) or (2) and the NH hazardous waste number of NH02 if it meets the criteria set forth in Env-Wm 403.04(b)(3).

(b) A waste shall be classified under these rules as corrosive if a representative sample has any of the following characteristics:

(1) It is aqueous and has a pH of less than or equal to 2, or greater than or equal to 12.5, as determined by a pH meter using either Method 5.2 in SW-846 as defined in Env-Wm 110.01(b) and incorporated by reference at Env-Wm 110.03, or an equivalent test method approved by the Administrator of EPA under the procedures set forth in 40 CFR 260.20 and 40 CFR 260.21, 7-1-89 edition;

(2) It is a liquid and corrodes steel (SAE 1020) at a rate of greater than 6.35 mm or 0.250 inch per year at a test temperature of 55°C (130°F) as determined by the test method specified in NACE (National Association of Corrosion Engineers) Standard TM-01-69 as standardized in SW-846, or an equivalent test method approved by the Administrator of the EPA under the procedures set forth in 40 CFR 260.20 and 40 CFR 260.21, 7-1-89 edition; or

(3) It is a non-aqueous waste which when mixed 50% by weight with distilled water, or a gaseous material which when mixed with distilled water to form a 2 molar solution, yields a pH less than or equal to 2 or greater than or equal to 12.5 as measured with a pH meter using the protocol specified in SW-846. Such wastes shall have the NH hazardous waste number of NH02.

Env-Wm 403.05 Reactivity.

(a) A waste that exhibits the characteristic of reactivity but is not listed in Env-Wm Env-Wm 402.04(b), Env-Wm 402.05(b), Env-Wm 402.06(a) or Env-Wm 402.07(a) and is not a mixture under Part Env-Wm 404.01(a) shall be assigned the EPA hazardous waste number of D003.

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Laboratory Method for the Determination of pH In Liquid and Solid Samples

Approved By:

QA Officer: _____ Date: 03/09/01

Laboratory Director: _____ Date: 3/9/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Laboratory Method for the Determination of pH in Liquid and Solid Samples.
EPA Reference:

EPA 600, Method 150.1

Test Methods for Evaluating Solid Waste Physical/Chemical Methods- Method 9045 C, Revision 3 January 1995

Test Methods for Evaluating Solid Waste Physical/Chemical Methods- Method 9040B,Revision 2 January 1995

Comprehensive Quality Assurance Plan For ANALYTICS Environmental Laboratory, LLC. January 2001

2. Scope and Application

This is a procedure used to measure the pH of aqueous wastes,(multiphase wastes where the aqueous phase constitutes at least 20 % of the total volume) soils and wastes. Wastes may be solids, sludges or non-aqueous liquids. If water is present, it must constitute less than 20 % of the total volume of the sample to be treated as a soil or waste.

3. Interferences

3.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or moderate salinity.

3.2 Coatings of oily material of particulate matter can impair electrode response. Gentle wiping or detergent washing, followed by a Distilled water rinse can usually remove these coatings.

3.3 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in the electrode output at various temperatures. This interference should be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This interference is sample dependant and cannot be controlled. It is therefore required to document the pH and temperature at the time of analysis.

3.4 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of > 10, the measures pH may be incorrectly low. Using a low-sodium-error electrode can minimize this error.

4. Safety

Wearing a laboratory coat, safety goggles and solvent resistant gloves while working with samples should minimize potential exposure to hazardous materials. Samples should be covered with a watch glass while stirring to prevent splashing of sample onto work area.

5. Method Summary

5.1 Liquid samples are measured directly with a pH meter at room temperature (18-25°C).

5.2 Solid and waste samples are mixed with reagent water. The pH of the aqueous solution is then measured with a pH meter.

6. Sample Collection, Preservation and Handling

6.1 Aqueous samples should be collected in a clean plastic container.

6.2 Soil samples and wastes should be collected in clean glass containers.

6.3 All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling instructions.

6.4 Samples should be refrigerated at (1.1-4.4°C)as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and login procedures.

6.5 Samples should be analyzed as soon as possible after collection. Analysis time may not exceed 24 hours from collection.

7. Definitions

7.1. °C = Degrees Celsius

7.2 g = grams

7.3 ml= milliliters

7.4 H_2O = water

7.5 HCL = hydrochloric acid

7.6 pH = negative logarithm of the hydrogen ion concentration

7.7 N = normal

7.8 TCLP = Toxicity Characteristic Leaching Procedure

7.9 AgCl = silver chloride

8. Apparatus and Materials

8.1 pH Meter with temperature compensation, Orion model 230A or equivalent

8.2 50 ml beaker.

8.3 Watch glass.

8.4 Scoop or spatula.

8.5 Stir plate.

8.6 Magnetic stir bar.

8.7 25 ml graduated cylinder.

8.8 Balance capable of accurately weighing to 0.01 g. Calibrated per QA-M009.

8.9 Wide range pH indicator paper - pH 0-14.

8.10 pH Logbook.

8.11 pH Meter Calibration Logbook.

9. Standards and Reagents

9.1 Reagent water - Distilled then deionized with a Sybron Barnstead 3-canister filtering system or equivalent.

9.2 Beckman pH 12.45 salts for buffer solution - Prepared by emptying contents of one bottle into 1 liter erlenmeyer flask that is one quarter full with reagent water. Bring to 500 ml. Add a stir bar to the flask, place the flask on a magnetic stirrer and stir for 5 minutes. Allow solution to settle. Decant clear solution for pH measurement. The solution is stable for 6 months.

9.3 Per**pH**ect Buffer 4.0 - Orion Cat. No. 910425, or equivalent.

9.4 Per**pH**ect Buffer 7.0 - Orion Cat. No. 910725, or equivalent.

9.5 Per**pH**ect Buffer 10.0 - Orion Cat. No. 911025, or equivalent.

9.6 pH Electrode storage solution - Orion Cat. No. 91001, or equivalent. Ionically-balanced solution to eliminate pre-soaking and preparation procedures.

9.7 1 N Hydrochloric acid- purchased as a solution.

9.8 pH electrode filling solution - AgCl purchased from pH probe manufacturer (Orion)

9.9 Second source 7.0 buffer – VWR 7.00 cat. no. 34170-115, or equivalent.

10. Procedure

10.1 pH Meter Calibration For TCLP Extracts or Extraction Fluids - pH 4.0 - 7.0

10.1.1 The "Power" button on pH meter turns the meter on.

10.1.2 To prepare the buffer solutions, open corresponding envelopes being careful not to spill solutions inside. The lot code of each buffer solution, found at

the base of the envelope, should be noted in the logbook. **These solutions are single use solutions and are good for one day after opening.**

10.1.3 Rinse the pH meter electrode with reagent water and place in buffer solution.

10.1.4 Press "cal" button on the meter, "P1" will appear on the bottom of display. The pH meter is ready when "Ready" flashes on the display and a high-pitched tone is audible.

10.1.5 If the first calibration pH reading is **within 0.05 pH units of the buffer solution value, it is acceptable.** Press "Yes" button on pH meter to store this first point. If the pH reading is unacceptable, repeat the reading. If after several readings the pH reading is still unacceptable, press "setup" button to adjust readout to 4.00 then press "yes". Record pH value(s) in the pH logbook (Figure 1).

10.1.6 The display will show "P2" when the pH meter is ready to calibrate the second point.

10.1.7 Repeat Section 10.1.2 to 10.1.5 for pH 7.0 buffer solution (Section 9.3).

10.1.8 After the second point has been accepted the pH meter will display "Slope" followed by a temperature reading. Record both of these reading in the pH logbook (Figure 1). **Acceptable slope values are 92-102% and temperature values are 18-25°C.** If the slope is outside of the acceptance range, try re-calibrating. If the slope is still unacceptable, proceed to section 11.6 "Electrode Cleaning" then re-calibrate.

10.1.8 Once the meter is calibrated the calibration should be checked using a second source buffer solution. Pour about 10 ml of the VWR 7.00 solution into a 20 vial and measure the pH. If the pH reading does not agree within 0.05 pH units the meter should be recalibrated.

10.1.9 For measuring a pH greater than 7.0 the pH meter must be calibrated using pH buffer solutions 7.0 and 10.0 (Section 9.3 & 9.4) before an accurate reading can be taken. pH readings greater than 10.0 require verification of calibration by analyzing the Beckman buffer solution, pH 12.45 (Section 9.2).

10.2 pH Meter Calibration for Client Samples

10.2.1 Calibration is performed as described Section 10.1.

10.3 pH Measurement of Liquid Samples

10.3.1 pH readings for liquids are recorded in the pH logbook. It is required to record: date, analyst's initials, AEL sample #, pH, duplicate pH, temperature (°C), time, and pH calibration range.

10.3.2 Samples should be analyzed as soon as possible after receipt.

10.3.3 Samples must be between 18-25 °C before reading pH.

10.3.4 **For Corrosivity Characterization, the sample must be measured at 25°C +/-1°C if the pH of the waste is above 12.0.**

10.3.5 Place 20-30 ml of sample in a 50 ml beaker, making certain that there is sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stir bar. Add the magnetic stir bar. Place on the stir plate and stir at a moderate constant rate to provide homogeneity while not producing any vortexing or splashing of the sample.

10.3.6 Check pH of sample with pH indicator paper to determine which 2 buffer solutions to use for calibration.

10.3.6 When the pH meter displays "ready", read and record the pH of the sample, (to the nearest 0.01 unit),temperature (to the nearest °C), and time (to the nearest minute).

10.3.7 Duplicates are performed on each sample. **Repeat measurements on the sample until readings differ by less than 0.10 pH units.** The average of the duplicate measurements is reported.

10.3.8 pH results are transferred to the pH Logbook (Figure 1). File a copy in the in the client's folder.

10.3.9 Rinse the probe thoroughly with reagent water between samples, and if necessary gently wipe electrode with a kim-wipe to remove any residue. Press the "Measure" button to reset the pH meter.

10.3.10If the electrode becomes coated with an oily material that will not rinse free, the electrode can be washed (see 11.6 Electrode cleaning for details).

10.4 pH Measurement Solid Samples

10.4.1 pH data is recorded in the pH logbook, with a header of "Soil pH" .Its required to document the following: date and analyst's initials, AEL sample #, Weight, Matrix, Volume (H_2O), pH, duplicate pH, temperature (°C), time and pH calibration range.

10.4.2 Calibrate the balance (QA-M009). Weigh out 20g of sample and record the weight to the nearest 0.01g.

10.4.3 Combine the 20 g of sample and 20 ml reagent H_2O in a 50 ml beaker. Place the beaker on the stir plate, add the stir bar, cover with a watch glass and stir for **5** minutes.

10.4.4 Let the mixture stand for about one hour to allow the solid portion to settle out of the solution.

10.4.5 Check pH of water layer with pH indicator paper to determine which 2 buffer solutions to use for calibration.

10.4.6 Place the pH meter electrode in the beaker such that it is immersed in the water portion of the mixture but not in the solid portion.

10.4.7 When the pH meter displays "ready", read and record the pH value, temperature reading, and time (to the nearest minute).

10.4.7 Duplicates are performed on each sample. **Repeat the measurement until successive readings agree within 0.10 pH units.** The average of the duplicate measurements is reported.

10.4.8 Rinse the probe thoroughly with reagent water between samples, and if necessary gently wipe electrode with a kim-wipe to remove any residue. Press the "Measure" button to reset the pH meter.

10.4.9 pH results are transferred to the pH Logbook (Figure 1). File a copy in the client's folder.

11. Quality Control

11.1 The pH meter must be calibrated prior to use.

11.2 Samples will be analyzed in duplicate. Successive measurements must agree within 0.10 pH units for the results to be valid.

11.3 Samples with pH greater than 10.0 will be verified by analyzing the Beckman pH 12.45 buffer.

11.4 Electrodes are thoroughly rinsed between each measurement.

11.5 Daily and after each calibration the calibration must be checked by analyzing a second source buffer solution. The pH reading must agree within 0.05 pH units or the meter should be recalibrated.

11.6 Calibration should be verified after every 10 samples, and when the probe has been unattended for more than one hour, or the probe has powered off. The calibration should be verified by running a buffer solution in the range of calibration. The pH reading must agree within 0.05pH units.

12. Routine Maintenance

12.1 Electrode Cleaning Procedure

12.1.1 Electrodes are cleaned in 1 N HCL as needed.

12.1.2 If the electrode is used with oily waste or sludge samples, or if performance is not satisfactory (time to "ready" too long or readings are not accurate) a more thorough cleaning may be required.

12.1.3 Begin by changing the reference solution within the pH probe. Use a pipet to remove the reference solution through the small hole in the upper portion

of the probe. Remove as much of the solution as possible by inverting the probe.

12.1.4 Refill the probe with fresh solution to just below the filler hole. Do not cover filler hole except when shipping or storing probe for extended periods of time.

12.1.5 Retry pH calibration. If performance is satisfactory, probe is ready for use. If performance is not satisfactory, further cleaning is required.

12.1.6 Allow pH probe to soak in solution of 1 N HCL for no longer than 1 hour.

12.1.7 After 1 hour, remove probe from 1 N HCL solution and change reference solution (see 11.6.3).

12.1.8 Allow probe to soak in storage solution for one hour then retry calibration.

12.1.9 If performance deficiency persists, call manufacturer (Orion, Inc.) for further options, including replacement of probe.

12.2 pH meter electrode will be kept in Electrode storage solution when not in use. Solution should be refreshed monthly or as needed.

13. Pollution Prevention and Waste Management

13.1 See SOP QA-W001 for handling of waste and unused portions of sample.

EPA 600, Method 150.1
SW-846 Method 9045C
SW-846 Method 9040B

DATE _____

pH Meter Calibration	Buffers					
	4.00	7.00	10.00	Slope	Temp deg C	Analyst

				If Solid Matrix						Reporting
	AEL Sample #	Analyst	Matrix	Wt (g)	H20 (ml)	pH / Time	Temp	pH Dup / Time*	Temp dup	Ave. pH
1	QC Buffer Check 7.0									
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										

FIGURE 1

Notes - pH must be taken at ambient temperature
- QC buffer check must agree within .05 pH units.

* Duplicate pH measurements must agree within .10 pH units.

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COPY

Laboratory Method for the Determination of Reactive Sulfide and Cyanide in Solid and Liquid Samples

Approved By:

QA Officer: _____ Date: 11/7/01

Laboratory Director: _____ Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Laboratory Method for the Determination of Reactive Sulfide and Cyanide in Solid and Liquid Samples.

EPA References: Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Chapter Seven Characteristics Introduction and Regulatory Definitions, Revision 3, September 1986

Comprehensive Quality Assurance Plan for Analytics Environmental Laboratory, LLC, January 2001

2. Scope and Application

This method is used to determine the amount of hydrocyanic acid or hydrogen sulfide released from a sample when exposed to a mild acid. This test measures only the hydrocyanic acid and hydrogen sulfide evolved under these test conditions. It is applicable to all wastes, with the condition that when the waste is combined with acid it **DOES NOT** form an explosive mixture

3. Interferences

3.1 Interferences are undetermined

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard

4.2 **Hydrogen sulfide and hydrogen cyanide are deadly gases. Extreme caution must be exercised. See attached information from the Merck Index. All testing must take place in a fume hood.**

4.3 Exposure should be minimized by wearing gloves, safety goggles and a lab coat when samples or standards are handled, and by working in a fume hood. Material Safety Data Sheets for all of the compounds and reagents used in this method are on file in the laboratory and should be read by any personnel prior to initiating this method.

5. Method Summary

Approximately 10 g of solid sample or 10 ml of liquid sample is combined with 100 ml of 0.01 N H_2SO_4 in a closed, pressurized system and mixed for thirty minutes. Any hydrocyanic acid or hydrogen sulfide gas generated is forced through an indicating tube which will change color with the presence of hydrogen sulfide or hydrogen cyanide.

6. Sample Collection, Handling and Preservation

6.1 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling.

6.2 Samples should be refrigerated (1.1°-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-l001 for laboratory receipt and log-in procedures

6.3 Samples should be collected to minimize headspace. All sample containers should be filled completely. Samples should be analyzed as soon as possible but no longer than 7 days after collection.

7. Definitions

7.1 µl = micro-liter

7.2 ml = milliliter

7.3 L = liter

7.4 ng = nanogram

7.5 µg = microgram

7.6 mg = milligram

7.7 g = gram

7.8 psi = pounds per square inch

7.9 ml/min = mililiters per minute

7.10 QC = quality control

7.11 H_2S = Hydrogen sulfide

7.12 HCN = Hydrogen cyanide

7.13 ppm = parts per million

7.14 $Na_2S \cdot 9H_2O$ = Sodium sulfate nonahydrate

7.15 rpm = revolutions per minute

7.16 H_2SO_4 = Sulfuric acid

7.17 KOH = Potassium hydroxide

7.18 KCN = Potassium cyanide

7.19 N_2 = nitrogen gas

7.20 M = Molar

7.21 N = Normal

8. Apparatus and Materials

8.1 2 ring stands

8.2 2 clamps

8.3 2 stir plates capable of 30 rpm

8.4 2 magnetic stir bars

8.5 2 Large rubber stoppers

8.6 2 500ml 3-neck round bottom flasks with 20/40 ground glass joints

8.7 2 Flask stands

8.8 2 250ml addition funnels with 20/40 ground glass joints

8.9 Drager tubes: CH25701 for hydrocyanic acid 2/9 (yellow) and CH29101 for hydrogen sulfide 100/9 (white)

8.10 2 Drager tube mounting apparatus

8.11 2 Nitrogen flow gauges capable of measuring 60 ml/min of gas flow

8.12 Nitrogen split-source flow regulator

8.13 Drager tube cutter

8.14 500ml graduated cylinder

8.15 10ml graduated cylinder

8.16 syringes

8.17 Wide range pH paper

8.18 Timer

8.19 Volumetric flasks - Class A 50 ml

8.20 Balance capable of accurately weighing to the nearest 0.01g. Calibrate per QA-M009.

9. Standards and Reagents

9.1 H_2SO_4 - reagent grade.

9.2 0.1 N H_2SO_4 - Add 2.8ml conc. H_2SO_4 to 500 ml reagent water, mix well then bring volume to 1L with reagent water.

9.3 0.01 N H_2SO_4 - Add 100ml 0.05 N H_2SO_4 to 500 ml reagent water, mix well then bring volume to 1L with reagent water.

9.4 570 ppm sulfide reference solution - Add 0.20g $Na_2S \cdot 9H_2O$ to 25ml reagent water mix well then bring volume to 50 ml with reagent water.

9.5 1000 ppm(1000 mg/L) cyanide reference solution - Add 0.13 g KOH pellets and 0.13 g KCN to 25 ml reagent water, mix well and bring volume to 50 ml with reagent water.

9.6 Reagent water - Distilled then de-ionized with a Sybron Barnstead 3-canister filtering system, or equivalent.

10. Procedure

10.1 Reactivity Apparatus Setup (Figure 1)

10.1.1 Set up ring stands side by side with clamps and stir plates on each. Place a 3 neck round bottom flask on each stir plate and secure in place with a clamp. Place a stir bar inside each flask then stopper the middle neck with a large rubber stopper. Secure a 250ml addition funnel to the left neck of each three-neck flask, stopcock must be closed until reaction is ready to run. Label one setup "H_2S" and the other "HCN".

10.1.2 Drager tube mounting apparatus. Cut off the glass tips at both ends of the CH29101(H_2S) and CH25701(HCN) Drager Tubes with the tube cutter. Insert Drager tubes into plastic tubing of the mounting apparatus oriented with the flow direction arrow on tubes pointing away from the apparatus. Once reaction is ready to run, the mounted tubes will be secured to the right neck of the 3-neck flask.

10.1.3 Nitrogen delivery system. Attach N_2 split-source flow regulator to hood N_2 spigot. To each regulator output attach a nitrogen flow guage. Turn on nitrogen tank regulator to 10-20 psi. Adjust source split regulator so that both gauges read 60 ml/min. Once reaction is ready to begin, N_2 system outlets will be attached to the tops of the 250ml addition funnels. Re-check N_2 flow rates after attaching to reactivity apparatus - flow may change. Readjust as necessary.

10.2 Logbook

10.2.1 All information pertinent to reactivity will be entered into the bound, paginated Reactivity Logbook. The logbook page is set up with separate sections for H_2S and HCN reactions (Figure 2). In each corresponding box record: sample #, matrix, volume or weight of sample tested, standard# for

0.01 N H_2SO_4 and volume used, N_2 flow, reaction run time, tube reading, pH of sample solution at termination of run and sample dilution factor. Each notebook page should have the date of the reaction and analyst's initials.

10.2.2 All standards prepared by the analyst for reactivity analysis must be properly recorded in the Miscellaneous Standards Preparation Logbook. In the left margin of the logbook enter the "Standard #", date and analyst initials. In the middle section of the page record the name or description of the standard being prepared. Below this heading write out the recipe for that standard. On the right margin enter the expiration date of the new standard. Cyanide and sulfide reference solutions are good for 6 months from the date of preparation. Acid solutions are good for one year. In the index section of the miscellaneous standard prep book write in column format the date, the standard #, a description of the standard, initials of analyst and the page on which it was recorded.

10.3 Low Level Calibration. Sections 10.3.1 and 10.3.2 may be run simultaneously.

10.3.1 10 ppm H_2S: Set up reactivity apparatus (Figure 1 and Section 10.1). With a 250μl syringe, measure 175μl of the 570ppm sulfide reference solution. Deliver into the "H_2S" round bottom flask. Add 10ml reagent water and a stir rod to the flask . Stopper the right neck with the Drager tube mounting apparatus and H_2S Drager tube. Measure 250ml of 0.01 N H_2SO_4 and slowly pour into the 250ml addition funnel. Attach addition funnel to left neck. Attach N_2 gauges as explained in section 10.1.3. Open addition funnel stopcock and turn on stir plate to begin reaction. Re-check N_2 flow gauges and allow to run for 30 min. At the end of the 30 min. disassemble the set up, take reading of Drager tube and pH of the solution in flask using wide range pH paper. Record results in reactivity logbook. Dispose of solution into a carboy and clean round bottom flask thoroughly.

10.3.2 10ppm HCN: Set up reactivity apparatus (Figure 1, Section 10.1). Using a 100μl syringe measure 100μl of 1000ppm cyanide reference solution. Deliver into 3-neck round bottom flask. Add 10ml of reagent water to flask and a stir rod, stopper middle neck. Attach HCN Drager tube to Drager tube mounting apparatus and stopper right neck. Measure 250ml of 0.01 N H_2SO_4 and pour slowly into a 250ml addition funnel. Open addition funnel stopcock and turn stir plate on to begin reaction. Re-check N_2 flow gauges and allow to run for 30 min. At the end of the 30 min. disassemble the set up, take reading of Drager tube and pH of the solution in flask using wide range pH paper. Record results in reactivity logbook. Dispose of solution into a carboy and clean round bottom flask thoroughly.

10.4 High Level Calibration - Sections 10.4.1 and 10.4.2 may be run simultaneously.

10.4.1 40ppm H_2S: Use the same setup as low calibration with 700μl of 570ppm sulfide reference solution

10.4.2 220ppm HCN: Use the same set up as low calibration with 200μl of 1000ppm cyanide reference solution.

10.5 Method Blank for HCN and H_2S

10.5.1 A blank is analyzed to ensure that there is no HCN or H_2S contamination that could yield false positives for client samples. Blanks must be run once per 20 samples or eight hour shift for both H_2S and HCN.

10.5.2 In two clean round bottom flasks, pour 10ml of reagent water.

10.5.3 Set up apparatus as described in section 10.1 (Figure 1) to run separate blanks for HCN and H_2S reactions by using corresponding Drager tubes.

10.5.4 Pour 250ml of 0.01 N H_2SO_4 into addition funnels then run reactions and record results in the same manner as calibration reactions.

10.6 Sample Reaction

10.6.1 Set up according to section 10.1 (Figure 1). For each reaction weigh out approximately 10g of solid sample (calibrate balance before use according to QA-M009) or measure approximately 10ml of liquid sample (using a 10 ml graduated cylinder). Record sample weight or volume in logbook. Carefully deposit samples into 3-neck flasks. Cap middle necks with large stoppers and right necks with Drager mounting apparatus. Attach corresponding HCN or H_2S Drager tubes to apparatus. Measure out 250ml of 0.01 N H_2SO_4 solution. Pour into the addition funnels and attach them to the left neck of each flask. Attach nitrogen sources to funnels and adjust N_2 flow to 60 ml/min. Open addition funnel stopcocks and run reaction for 30 min. Record tube readings and sample pH (using wide range pH paper) at end of reaction. If there is no tube indication, note U in notebook

10.6.2 Dilution Factor - Divide 10 by amount of sample used.. Divide result by percent solid (in decimal form). Record Dilution Factor in notebook

$$DF = \frac{\text{nominal wt (10g) of vol. (10mL)}}{\text{Sample amt (g or mL)X \% solid}}$$

10.6.2 If a client sample tube reading indicates a concentration above that of the highest standard, the sample must be run at an appropriate dilution that will yield a tube reading below that of the highest standard.

10.7 Result Calculation

10.7.1 Sulfide

$$H_2S \text{ (ppm)} = \frac{\text{tube reading sample}}{\text{tube reading 40 ppm standard}} \times 40 \text{ ppm } XDF$$

Values from the 10ppm standard may be substituted in the 40ppm positions. The analyst should quanititate from the standard that has a reading closest to the sample

10.7.2 Cyanide

$$HCN \text{ (ppm)} = \frac{\text{tube reading sample}}{\text{tube reading 20 ppm standard}} \times 20 \text{ ppm X DF}$$

11. Quality Control

11.1 The system is calibrated prior to sample analysis with two standard concentrations.

11.2 Blanks

A method blank is run after the standards to show that there is no contamination either inherently in the system or as carryover from the standards. Blanks are run for H_2S and HCN at a rate of one per shift or per 20 client samples.

12. Pollution Prevention and Waste Management

12.1 See SOP QA-W001 for handling of waste and unused portions of sample.

FIGURE 1

Reactivity Apparatus

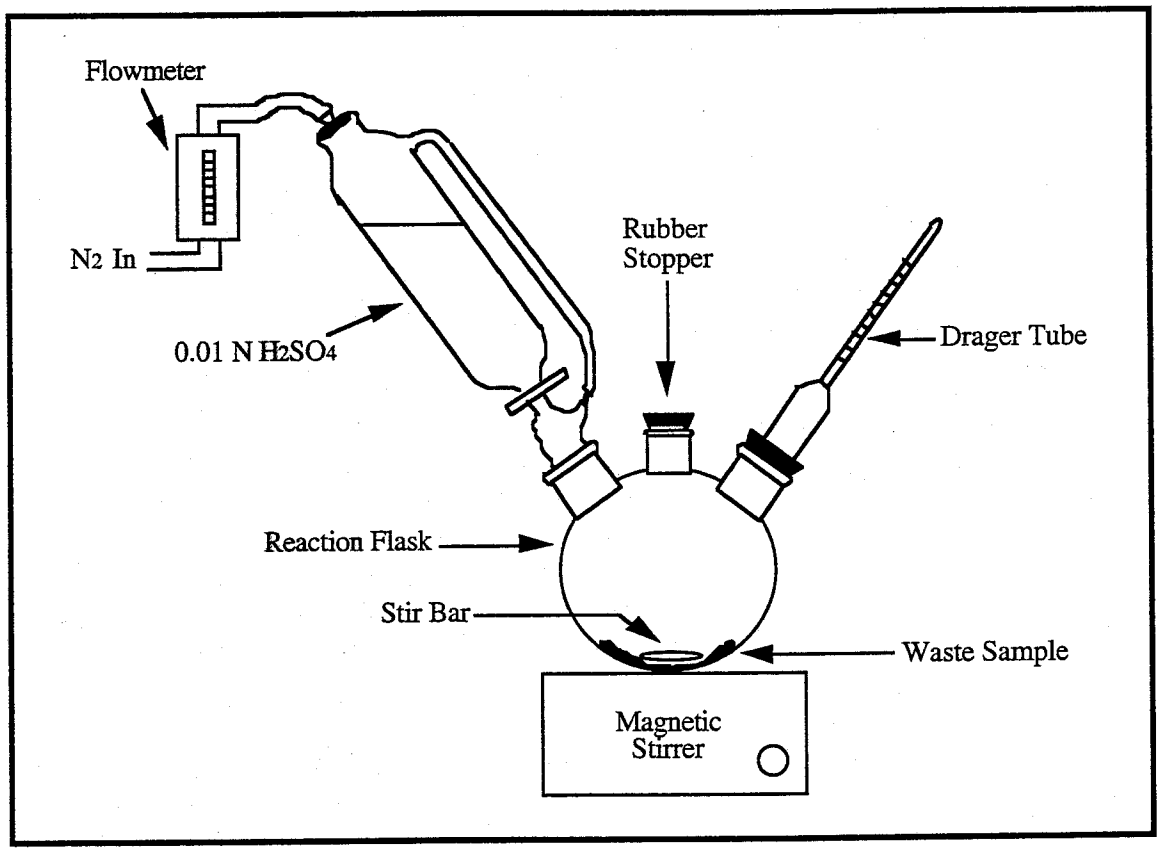

FIGURE 2

H_2S HCN Reactivity Data

DATE:
INITIALS:_____

H2S

SAMPLE #	MATRIX	ml/g	ml 0.01 N H2SO4	Std #	N2 Flow	TIME	Tube Rdg	Final pH	Dil. Fac.

HCN

SAMPLE #	MATRIX	ml/g	ml 0.01 N H2SO4	Std #	N2 Flow	TIME	Tube Rdg	Final pH	Dil. Fac.

Dilution = Nominal Wt/Volume
Factor Actual Wt/Volume x % Solid/100

REV 1/97

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COPY

Laboratory Method For Sample Preparation And Extraction Of Volatile Organic Compounds By Purge And Trap

Approved By:

QA Officer: _____ Date: 11/7/01

Laboratory Director: _____ Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Laboratory method for sample preparation and extraction of volatile organic compounds by purge and trap.

EPA Reference: EPA 600 Series-40 CFR, PART 136, Revised as of July 1, 1995 Appendix A to Part 136-Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. Method 625 –Semi-Volatile Organic Compounds

Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Methods 8260B Revision, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods-Method 8000B, Revision, December 1996

Comprehensive Quality Assurance Plan for ANALYTICS Environmental Laboratory, LLC. January 2001

2. Scope and Application

This method provides purge and trap conditions used to extract various volatile organic compounds (those that have boiling points below 200 degrees Celsius, and which are insoluble or slightly soluble in water). Purge and trap extraction is applicable to volatile organic compounds including purgeable aromatics, halocarbons and gasoline, in most sample matrices. Refer to the specific determinative Standard Operating Procedure (Table 1) for gas chromatographic conditions necessary to detect compounds of interest. This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system.

3. Interfernces

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevation in baselines. Method blanks must be routinely analyzed to show the absences of these interferences.

3.2 Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. In order to reduce carryover, the sample syringe must be rinsed with solvent between sample injections and whenever an unusually concentrated sample is analyzed, it should be followed by a solvent blank to check for contamination.

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard.

4.2 Exposure should be minimized by wearing safety goggles, solvent-resistant gloves and a laboratory jacket when samples or standards are handled, and by working in a fume hood.

4.3 **The following compounds have been tentatively classified as know or suspected human carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, 1,2-dichloroethane, 1,1,2,2-tetrachloroethane, tetrachloroethene, 1,1,2-trichlorethane and vinyl chloride.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method. (Refer to the specific determinative method for lists of chemicals and standards.)

5. Method Summary

An inert gas is bubbled through an aqueous sample contained in a purging chamber at ambient temperature(or 40°C for low soils). Medium and high level solid samples are first extracted in methanol. A portion of the methanol is diluted in reagent water prior to purging. Low level solid samples are weighed directly into specially designed purge cells or VOA vials, then diluted with reagent water prior to purging. Volatile compounds are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the compounds are adsorbed. When purging is complete, the trap is heated and backflushed with the inert gas to desorb the compounds onto a gas chromatographic column.

6. Sample Collection, Handling and Preservation

6.1 All samples should be collected with no headspace in 40 ml VOA glass jar with a screw top Teflon lid.

6.2 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling instructions.

6.3 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and log-in procedures.

6.4 Aqueous samples must be analyzed within 14 days of collection

7. Definitions

7.1 ALS = Automated Liquid Sampler

7.2 °C = degrees Celsius

7.3 GC = Gas Chromatograph

7.4 GC/MS = Gas Chromatograph/Mass Spectrometer

7.5 LSC = Liquid Sample Concentrator

7.6 QC = Quality Control

7.7 SOP = Standard Operating Procedure

7.8 µl = microliter

7.9 ml = milliliter

8. Apparatus and Materials (Equivalents may be used.)

8.1 Purge and Trap system:

8.1.1 Liquid Sample Concentrator: Tekmar LSC 2, Tekmar LSC 2000, Tekmar LSC 3000, OI 4560, or equivalent.

Conditions:	Purge gas (FID)	Nitrogen
	Purge gas (GC/MS)	Helium
	Purge temperature	ambient≤35°C (except low soils =40°C)
	Purge flow	25-40 ml/minute
	Purge time	11 minutes
	Dry purge time (optional)	0-2 minutes
	Desorb preheat temperature	175°C (250°C 5971GC/MS)(225°C 5970GC/MS)
	Desorb temperature	185°C (GAS/GRO), 250°C (5971GC/MS) 225°C (5970GC/MS)

Desorb time	4 minutes (6 minutes 5971
GC/MS)Backflush flow	20-60 ml/minute
Bake time	5 minutes, or greater
Bake temperature	200°C or greater
Trap components	See determinative SOP

8.1.2 Automated Liquid Sampler: Tekmar ALS, Tekmar ALS 2016, or OI MPM-16 (or equivalent) equipped with 5 ml or 25 ml purge cells or a Tekmar Precept.

8.2 5 ml Luerlok Syringe Rinse twice with reagent grade water before and after use.

8.3 25 ml Luerlok Syringe Rinse twice with reagent grade water before and after use.

8.4 Gastight Syringes :1 ml, 500 µl, 100 µl, 250 µl, 50 µl, 25 µl, and 10 µl. Rinse three times with methanol before and after use.

8.5 50 ml plastic syringes with Luerlok tips (for rinsing purge cells.)

9. Standards and Reagents

9.1 Reagent Water: Distilled then deionized using a Sybron Barnstead 3-canister filtering system, or equivalent.

9.2 Methanol: Purge and Trap Grade, or equivalent.

9.3 Calibration Standards: Refer to the determinative method SOP for calibration and standard information.

10. Procedure - ALS, ALS 2016, or OI MPM-16

10.1 Aqueous samples (Non-drinking water analyses)

10.1.1 Remove the plunger from a 5 ml Luerlok syringe. Rinse the syringe barrel twice with reagent water, then once with the aqueous sample. Fill the syringe to just overflowing with the sample. (If the sample is a blank, standard, or laboratory control sample rinse and fill the syringe barrel with reagent water). Replace the plunger and adjust to the desired volume. The total purge volume required is 5 ml.

10.1.2 Sample dilution may be necessary for the following reasons: to achieve a target compound concentration within the calibration range, unusual sample matrix, the presence of high level non-target compounds, or due to sample foaming. If a dilution is necessary, use reagent water as the diluent to achieve a final volume of 5 ml. For example, if a ten fold dilution is required, fill the syringe barrel with reagent water and adjust the volume in the syringe to 4500 µl. Pull the syringe plunger back to create enough space for addition of 500 µl of the sample. Use a gastight syringe to add 500 µl of the sample directly through the syringe opening.

10.1.3 The process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one vial, the analyst should fill a second syringe at this time or aliquot into a 20 ml VOA vial the remaining sample to protect against possible loss of sample integrity. This additional aliquot should be analyzed within 24 hours.

10.1.4 Using a gastight micro-syringe, add the appropriate volume of surrogate(s), internal standard(s), or matrix spiking solution directly to the 5 ml Luerlok syringe by inserting the needle through the syringe opening.

10.1.5 Open the three-way sample valve on the purge cell into which the sample will be loaded. Turning clockwise, screw the Luerlok end of the 5 ml syringe onto the sample valve's luerlok connector or introduce sample directly into 25 ml one-way glass tube.

10.1.6 Introduce the entire sample into the purge cell by depressing the syringe plunger until all sample has been transferred. **CLOSE** the sample valve or tighten the glass tube into place on the autosampler.

10.1.7 Turn the syringe counter clockwise to remove it from the luerlok connector.

10.1.8 To reduce the possibility of cross contamination, rinse the syringe barrel twice with reagent water after the sample has been loaded.

10.1.9 Repeat steps 10.1.1 through 10.1.8 until all desired purge cells are loaded.

10.1.10 Follow the manufacturer's instructions to program an autosampler batch to sequentially purge and trap the appropriate samples, and begin purging the first sample.

10.1.11 Refer to the determinative method SOP in Table 1 for sample analysis.

10.1.12 All standards and samples must be warmed to ambient temperature prior to analysis.

10.2 Aqueous samples (Drinking water analyses using ALS 2016 or equivalent)

10.2.1 Remove the plunger from a 25 ml Luerlok syringe. Rinse the syringe barrel twice with reagent water. Fill the syringe to just overflowing with the sample. (If the sample is a standard, blank, or laboratory fortified blank, rinse and fill the syringe barrel with reagent water).

10.2.2 Replace the plunger and adjust to the desired volume. The total purge volume required is 25 ml. If a dilution is necessary, use reagent water as the diluent to achieve a final volume of 25 ml.

10.2.2 The process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This additional aliquot must be analyzed within 24 hours. If there is no dilution and the sample is in a 40 ml VOA vial, this will not be possible.

10.2.4 Using a gastight syringe, add the appropriate volume of internal standard, surrogate, standard, or matrix spiking solution directly to the 25 ml Luerlok syringe by inserting the needle through the syringe opening.

10.2.5 Open the three-way sample valve on the purge cell into which the sample will be loaded. Turning clockwise, screw the Luerlok end of the 25 ml syringe onto the sample valve's luerlok connector or tighten the glass tubes into place on the autosampler.

10.2.6 Repeat steps 10.1.6 through 10.1.11.

10.3 Solid sample analysis (Medium and High Level)

10.3.1 Prepare solid samples according to QA-V002.

10.3.2 Rinse the barrel of a 5 ml Luerlok syringe twice with reagent water. Fill the syringe to just overflowing with the water. For modified medium soil method, replace the plunger and adjust the volume to 4.5 ml (The maximum volume of methanol extract that may be loaded is 500 µl.). The total purge volume is 5

ml. Add internal standards or surrogates. Pull the syringe plunger back to create enough space for addition of 500 µl of the sample. The unmodified medium soil method uses a maximum volume of 100 µl of methanol, with a total volume purged of 5 ml.

10.3.3 Using a gastight syringe, transfer 500 µl (100 µl for unmodified) of the methanol extract through the opening of the syringe.

10.3.4 Repeat steps 10.1.5 through 10.1.11.

10.3.4 If dilutions are performed, increase reagent water used to achieve a final volume of 5 ml.

10.4 Low level solid samples

10.4.1 Refer to QA-V003 for preparation of low level solid samples.

10.4.2 Repeat steps 10.1.10 through 10.1.11.

11. Quality Control

11.1 Instrument Blanks

The purge and trap system must be demonstrated to be free from contamination under the conditions of the analysis by analyzing reagent water (instrument) blanks. Instrument blanks are analyzed daily at a minimum and then 1 per 20 client samples.

11.1.1 Using reagent water and the appropriate internal and/or surrogate standards, purge the blank as necessary for the determinative method (*i.e.* 25 ml for drinking water, 5 ml for non-drinking water.) .

11.1.2 Refer to the specific determinative method (Table 1) for labeling convention and acceptance criteria.

11.2 Autosampler Contamination Prevention

11.2.1 If necessary, use the 50 ml plastic syringes to rinse each purge cell twice with reagent grade water after use if the previous sample was contaminated. It may also be necessary to bake the cells in an oven. Some autosamplers have automatic pre-purge and line bake methods which may

be used to help clean the system prior to sample analysis. The two Tekmar™ ALS 2016's use "Method 2" to prepurge cells and lines.

11.2.2 Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. Whenever a sample that contains any compound that is greater than the highest level of the initial calibration is analyzed, the sample should be followed by a reagent water blank to check for cross contamination.

11.2.3 The trap is baked daily prior to use.

11.3 Purge Cell Contamination Prevention
Refer to QA-M001 for the cleaning of contaminated purge cells.

11.4 Purge gas used is grade 5 or equivalent.

12. Routine Maintenance

12.1 Record any maintenance in the Instrument Maintenance Log.

12.2 Replace the sorbent trap as needed, refer to determinative method. Condition new traps for 20 minutes in the "Bake" mode prior to use.

12.3 The hydrocarbon trap on the purge gas line should be replaced annually to remove impurities from the purge gas.

13. Pollution prevention and waste management.

13.1 See SOP W001 for handling of waste and unused portions of sample.

TABLE 1

Determinative Standard Operating Procedures for Volatile Organic Compounds

SOP Number	Title
V005	Laboratory Method for the Determination of Volatile Organic Compounds - Capillary Column, GC/MS Detection
V006	Laboratory Method for the Determination of Volatile Organic Compounds in Drinking Water - Capillary Column, GC/MS Detection
V007	Laboratory Method for the Determination of Volatile Organic Compounds - Capillary Column, Gas Chromatography with PID/ELCD Detection
T009	Laboratory Method for the Determination of Total Gasoline in All Matrices
T015	Laboratory method for the Determination of Gasoline Range Organics in All Matrices

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Separatory Funnel Liquid/Liquid Extraction for the Determination of Pesticide and PCB Compounds in Aqueous Samples

COPY

Approved By:

QA Officer: _____ Date: 08/17/99

Laboratory Director: _____ Date: 8/17/99

Revision History

Revision	Description of Change	Approval	Date

1. Title

Separatory funnel liquid/liquid extraction for the determination of pesticide and PCB compounds in aqueous samples.

EPA References: Federal Register, Method 608
SW-846, Method 3510C

2. Scope and Application

This method describes the isolation and concentration of pesticides and PCB's from aqueous samples in preparation for analysis by GC/ECD. The parameters listed in Table 1 can be measured by this method.

3. Safety

The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard. Exposure should be minimized by wearing solvent-resistant gloves, a lab coat, and safety goggles when samples or standards are handled, and by working in a fume hood. **The following compounds included in this method are classified as known or suspected human carcinogens: the BHC's, the DDT series, and PCBs.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

4. Method Summary

Approximately 1 Liter of an aqueous sample is serially extracted with methylene chloride at a pH between 5-9 using a separatory funnel extraction. The methylene chloride extract is then dried, exchanged to hexane, concentrated to a volume of 10.0 ml, and analyzed by GC/ECD.

5. Definitions

5.1 ml= milliliter
5.2 L= liter
5.3 GC/ECD= gas chromatograph/electron capture detector
5.4 PCB= polychlorinated biphenyl
5.5 ^{o}C = Degrees Celsius
5.6 $MeCl_2$ = Methylene Chloride
5.7 QC = Quality Control
5.8 CAS = Chemical Abstact Service
5.9 PSI = Pounds per square inch
5.10 N_2 = nitrogen
5.11 AEL = Analytics Environmental Laboratory, Inc.

6. Apparatus and Materials

6.1 Automatic Shaker Table- Glas-Col 3-D Shaker
6.2 Separatory funnel - 2 liter glass, with Teflon stopcock and glass or Teflon cap.
6.3 Erlenmeyer flask - 250 ml glass.
6.4 Long-stem glass funnel with glass wool plug.
6.5 TurboVap II Concentration Workstation - Zymark Part Number 46368 or equivalent.
6.6 TurboVap Tubes - 1.0 ml stem.
6.7 Vials - 2ml glass autosampler with Teflon lined crimp caps.
6.8 pH indicator paper - Wide range, capable of measuring 0-14 pH units.
6.9 Graduated cylinder - 1 liter.
6.10 Graduated cylinder - 100ml.
6.11 Syringes - 1ml.

6.12 Ring stands.
6.13 Class A volumetric glassware as required for final volume determination - rinsed 3 times with hexane before and after use.
6.14 Pesticide/PCB sample prep log.
6.15 Disposable Pasteur pipettes, 9 inch borosillicate glass.
6.16 Nitrogen blow down apparatus.
6.17 125ml glass Separatory funnel, with Teflon stopcock and glass or Teflon cap.
6.18 12ml vial and Teflon lined screw caps.
6.19 Red labeling tape
6.20 Wax pencil

7. Standards and Reagents

7.1 Methylene chloride - Pesticide quality or equivalent.
7.2 Reagent water - Distilled then deionized with a Sybron Barnstead 3-canister filtering system or equivalent.
7.3 Sodium sulfate - Granular, anhydrous; suitable for environmental residue analysis. Purify by heating at 200°C for a minimum of four hours in a shallow glass baking dish and store in a closed glass container.
7.4 Hexane - Pesticide quality or equivalent
7.5 Methanol - purge and trap grade or equivalent for syringe rinsing.
7.6 Nitrogen gas-grade 5, from a liquid nitrogen tank
7.7 Pesticide/PCB surrogate. Refer to QA-P001 for standard preparation.
7.8 Pesticide matrix spiking soloution. Refer to QA-P001 for standard preparation.
7.9 PCB matrix spiking soloution. Refer to QA-P001 for standard preparation.

8. Procedure

8.1 Sample Preparation Notebook
All information pertinent to Pesticide and PCB sample preparation is recorded in a bound paginated laboratory notebook. Each entry should contain a general heading with the extraction date and the initials of the analyst(s) that performed the extraction. A column format should then include the following headings: sample number, initial volume (mls), surrogate standard number and amount added, spike standard number and amount added, volume extraction solvent recovered, final extract volume (after cookdown), and the date the concentration was completed as well as the initials of the individual(s) performing the concentration. Observations about specific samples will also be recorded in the notebook. For example, it is important to note when emulsions or precipitates form. The Lot #'s of all reagents used shall be documented with each extraction batch.
8.2 Determine Necessary Quality Control
8.2.1 One method blank must be performed for each analytical batch (twenty samples, or one eight hour shift.) The method blank consists of one liter of reagent water with 1.0 ml of surrogate that is extracted in the same manner as the samples. The blank is labeled as follows: BMMDDYP, where;

B indicates a blank
MM is the two number designation for the month
DD is a two number designation for the day
Y is a single number designation for the year
P designates Pesticide/PCB-water

8.2.2 One laboratory control sample and laboratory control sample duplicate must be performed for Pesticides and for PCB's for every twenty client samples (4 lab controls total) or within

a one month period. Fill out the QC Summary (Figure 1) to determine if it is necessary to perform laboratory control samples and laboratory control sample duplicates. Laboratory control samples and laboratory control sample duplicates are prepared by spiking 1 liter of reagent water with 1.0 ml of either pesticide or PCB spiking solution and 1.0 ml of surrogate. The laboratory control sample is labeled either: LMMDDYPT or LMMDDYPB where;

L designates a laboratory control sample
MM is the two number designation for the month
DD is a two number designation for the day
Y is a single number designation for the year
PT designates pesticide spiked water or PB designates PCB spiked water

LDMMDDY(PT or PB) is the designator for the laboratory control sample duplicate.

8.2.3 A matrix spike of an actual sample will be performed on an as-assigned basis when sufficient sample is provided. Matrix spikes will be labeled with the sample identifier followed by MS. Matrix spikes are prepared by spiking 1 liter of sample with 1.0 ml of Pesticide or PCB spiking solution and 1.0 ml of surrogate.

8.3 Glassware Preparation

8.3.1 For each sample, set up a ring stand with a ring, a 2-liter separatory funnel, and a 250ml Erlenmeyer flask. Pre-rinse all of the glassware, including the caps, with methylene chloride.

8.3.2 Place each separatory funnel in a ring and label it, using a wax pencil, with the sample number it will contain. Label (with red tape) each of the 250ml flasks with the sample number, P/P-H_2O, and the blank # that the sample batch is associated with. It is imperative that the labels be clear. The labels will be transferred throughout the extraction process.

8.3.3 Place the sample container on the ring stand supporting the appropriate funnel. The funnel label and sample are to be checked by another individual for agreement. Note that the samples were checked in the sample preparation notebook by putting a check over the initials of the individual performing the check.

8.3.4 Using a wax pencil, mark the meniscus on the outside of the sample container. Carefully pour the sample into the separatory funnel being careful not to transfer any sediment. Verify that the sample pH of the sample measures between 5 and 9 using wide range pH paper.

8.3.5 Using a 1 ml syringe, designated for P/P surrogate, add 1.0 ml of the P/P surrogate to every sample. Mark each separatory funnel with wax pencil after the addition of the surrogate.

8.3.6 If laboratory control samples and laboratory control sample duplicates are being extracted, use a 1 ml syringe to add 1.0 ml of either pesticide or PCB spiking solution to a liter of reagent water. Mark each separatory funnel with wax pencil after the addition of the spike. **The syringe used for spiking is not a dedicated syringe so it must be rinsed 3 times before and after use with Purge and Trap grade methanol.**

8.3.7 If a matrix spike is being extracted, use the 1 ml syringe in 8.3.6 above to add 1.0 ml of pesticide spiking solution or 1.0 ml of PCB spiking solution to the sample which is being spiked. Mark each separatory funnel with wax pencil after the addition of the spike.

8.4 Extraction

8.4.1 Measure 60ml methylene chloride in the 100ml graduated cylinder. Pour the first aliquot of methylene chloride into the sample container, rinse. Add the methylene chloride to the separatory funnel.

8.4.2 Seal the separatory funnel and place it in the auotomatic shaker holder. Invert the shaker holder and open the stopcock . Set the motor speed to 66 and program the timer for 2 minutes. Turn on the shaker table. **Methylene chloride creates excessive pressure rapidly and should be initially vented immediately after the funnel has been sealed and shaken once.**

8.4.3 Allow the methylene chloride to completely separate from the aqueous phase (about 10 minutes). If an emulsion occurs, mechanical techniques must be employed to try to complete phase separation. Gentle rocking or stirring may help. A 125ml separatory funnel may be useful in dispersing emulsions. Slowly drain the emulsion from the 2 liter separatory funnel into a pre-rinsed125ml separatory funnel. This action alone may be sufficient to disperse the emulsion. If it does not, return the emulsion to the separatory funnel and employ a different technique. Be sure to rinse the 125ml separatory funnel with methylene chloride to remove any residuals left behind. The emulsion may be filtered into the appropriate flask through sodium sulfate, **rinsed with methylene chloride**, in a long stem funnel plugged with a small amount of glass wool. Slowly drain the methylene chloride extract into the corresponding numbered flask by carefully opening the stopcock. Close the stopcock when all of the methylene chloride has been collected in the flask. Record all information on emulsions in the P/P preparation logbook.

8.4.4 Add a second 60ml aliquot of methylene chloride. Repeat steps 8.4.2 and 8.4.3, combining the extracts in the flask.

8.4.5 Repeat steps 8.4.2 and 8.4.3 with a third aliquot of methylene chloride. Again, combining the extracts in the flask. The extraction is now complete.

8.4.6 Note the volume of extraction solvent recovered. **The total volume recovered must be at least 140 mL or additional extractions must be performed.**

8.4.7 Determine the initial volume of the sample. Fill the sample container to the line which marked the meniscus.Carefully transfer the contents into a 1000ml graduated cylinder. Record the volume to the nearest 5mls in the sample preparation log notebook. Dispose of spent sample in the liquid waste stream.

8.5 Extract Drying and Concentration

8.5.1 If the extract is not concentrated immediately, store in the refrigerator.

8.5.2 Plug a long-stem glass funnel with a small wad of glass wool and fill funnel about 3/4 full with baked sodium sulfate. Rinse the sodium sulfate thoroughly with methylene chloride. Collect methylene chloride in a container designated as 'methylene chloride waste' and dispose of properly.

8.5.3 The TurboVap temperature should be set at 35°C for the Pesticide/PCB extract. The select button should be set on timer and the timer set to 40 minutes. The nitrogen pressure should be set to 10 psi.

8.5.4 P/P extractions are in methelyene chloride which must be exchanged to hexane so the extract will need to be concentrated in two stages. Transfer the label to the TurboVap tube and pour the extract from the flask into the tube through a funnel filled with sodium sulfate. After the sample has passed through the sodium sulfate, rinse the flask three times with methylene chloride and pour over the sodium sulfate. Pour about 20 ml of methylene chloride over the sodium sulfate to rinse out any of the remaining extract. Begin concentration by activating the TurboVap cell which contains the sample extract.

8.5.5 After 40 minutes of concentration (or when the volume of methylene chloride reaches about 10 mls) the sample extract must be exchanged with hexane. Remove the TurboVap Tube from the TurboVap. Re-set the timer to 10 minutes and the temperature to 40°C. Add 20mls of hexane directly to the TurboVap Tube. When the temperature of the TurboVap reads 40° C place the TurboVap Tube back in the TurboVap and continue concentration with nitrogen pressure still set at 10 psi.

8.5.6 Transfer concentrated sample extract with a clean 9 inch pasteur pipette into a 10 ml volumetric flask. With a clean pasteur pipette add a small amount of hexane to the TurboVap tube to rinse sides. Using the original pipette, rinse the walls of the lower portion of the Turbo tube at least three times then transfer the hexane to the volumetric flask. Repeat as many times as needed to achieve 10.0 ml volume in the flask. **(It is preferable to underestimate and do repeat rinsings rather than to**

overestimate and have some hexane remaining). When a final volume of 10.0 ml is achieved **mix the sample carefully but thoroughly by sucking some extract into the pipett then squirting it back into the volumetric flask repeatedly until the sample takes on a uniform appearance.** If there is a precipitate in the extract then it must be set aside for a few minutes to allow the precipitate to settle from the liquid portion. Transfer about 1 ml of the extract (do not transfer any particulate) with the same pipet into a labeled 2 ml vial labeled with the AEL #, P/P H_2O and the date. If the final extract volume is greater than 10 ml record the final volume on the vial. Pour the remaining sample from the volumetric into a 12 ml amber vial. Cap the vial, attach the tape label to the vial cap and place the extract in the freezer designated for Pesticide/PCB extracts.

8.5.7 If the TurboVap automatically shuts off before the extract has been reduced to <10.0 ml (this can happen repeatedly) the analyst has several options:

1)Reset the TurvoVap cell holding that sample with a few more minutes on the timer, until the sample is cooked down to <10.0 ml. Then go back to 8.5.6 for vialing. This option should be considered in most cases unless the sample is extremely oily or contains particulates.

2)Transfer the sample from the TurboVap tube to an appropriately sized collection tube. Set up nitrogen blow-down apparatus and condense sample to <10.0 ml or until sample volume reduction stops. If sample goes to <10.0 ml, return to section 8.5.6 for vialing. Otherwise, move on to option #3.

3)If sample volume can't be reduced to <10.0 ml using either of these techniques, the sample must be brought to a convenient volume. Make an estimate of the volume of the remaining sample and choose a Class A volumetric of sufficient size to contain the extract plus any hexane used to rinse the TurboVap tube. Use the pipette to transfer a portion of the extract to a clear 2.0 ml vial. Store the remainder of the extract in an appropriate container.

8.5.8 Extracts are stored in the freezer and must be analyzed within 40 days.

8.6 Transfer Sheets and Log Book

8.6.1 A transfer sheet (Figure 2) is generated each day that samples are completed. Deliver a copy to the GC/ECD analyst. In addition, make a copy to be included in the appropriate client folder. The original is filed in the laboratory output folder. Fill out "Final Volume" and "Date Transfered" columns in the sample prep log book. Make note of any unusually large final volumes.

9. Quality Control

9.1 Per sample

9.1.1 Surrogates are added to every sample, matrix spike, laboratory control sample and blank prior to extraction to test the efficiency of the extraction procedure. Percent recoveries are calculated by the GC/ECD analyst. If surrogate recoveries are outside of the pre-determined acceptable ranges, an Extractables Deviation Report explaining the cause is completed (Figure 3). Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. In this case, the GC/ECD analyst will transfer the deviation report to the extraction laboratory. Unusual circumstances during the extraction should have been noted in the sample preparation log notebook. Once the deviation report has been completed, it should be returned to the GC/ECD analyst. A copy is filed in the client's folder and the original is filed in the Extractable Deviation Notebook in the file room. Corrective action may include re-extracting the sample if sufficient sample exists.

9.2 Per sample batch (20 samples or per eight hour shift)

9.2.1 A method blank must be performed with every sample batch (20 samples, or per eight hour shift). A blank consists of 1 Liter of reagent water, that is carried throughout the extraction procedure. Blanks are analyzed prior to the appropriate sample batch to monitor interferences or contaminants that may have occurred during the extraction process. If a blank is found to contain more than the reporting limit of any compound, the sample batch associated with it must be re-extracted. If insufficient sample exists, the contaminant will be qualified with a "B" flag.

9.3 Per 20 Samples

9.3.1 One laboratory control sample and laboratory control sample duplicate is performed per batch of 20 samples or within a one month period (one each for pesticides and PCB's). The laboratory control samples are subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy and precision. The percent recoveries and relative percent difference of the compounds will be calculated by the GC/ECD analyst. If the recoveries or relative percent differences are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. An Extractables Deviation Report (Figure 3) will be completed by the analyst. Extraction personnel may be asked to determine the cause of the deviation. In this case the GC/ECD analyst will transfer the Extractables Deviation Report to the extraction laboratory. Unusual circumstances during the extraction should be recorded in the preparation log. Once the deviation report has been completed by the extraction laboratory, it should be returned to the analyst. A copy is filed in the QC Notebook with the chormatogram and the original is filed in the Extractables Deviation Notebook in the file room.

9.3.2 Matrix spikes are performed when sufficient sample has been provided to analyze both the sample and a spike of the sample. The matrix spike is subjected to the same extraction procedure as associated client samples. The spike recoveries will be calculated by the GC/ECD analyst. If the spike recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. An Extractables Deviation Report (Figure 3) will be completed by the analyst. Extraction personnel may be asked to determine the cause of the deviation. In this case the GC/ECD analyst will transfer the Extractables Deviation Report to the extraction laboratory. Unusual circumstances during the extraction should be recorded in the preparation log. Once the deviation report has been completed by the extraction laboratory, it should be returned to the analyst. A copy is filed in the QC Notebook with the chromatogram and the original is filed in the Extractables Deviation Notebook in the file room.

9.4 All surrogate and spike solutions are tested prior to use (QA-M015).

Table 1

Target Compounds

Compound	CAS #
Single Response Compounds	
α-BHC	319-84-6
β-BHC	319-85-7
γ-BHC (Lindane)	58-89-9
δ-BHC	319-86-8
Heptachlor	76-44-8
Aldrin	309-00-2
Heptachlor Epoxide	1024-57-3
α-Chlordane	5103-71-9
γ-Chlordane	5103-74-2
Endosulfan I	959-98-8
4,4'-DDE	72-55-9
Dieldrin	60-57-1
Endrin	72-20-8
Endosulfan II	33212-65-9
4,4'-DDD	72-54-8
Endrin Aldehyde	7421-93-4
4,4'-DDT	50-29-3
Endosulfan Sulfate	1031-07-8
Endrin Ketone	53494-70-5
4,4'-Methoxychlor	72-43-5
Multi-Response Compounds	
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
Toxaphene	8001-35-2
Chlordane (technical)	12789-03-6

FIGURE 1

QC SUMMARY PEST/PCB WATER SAMPLES

PCB LCS_____
PCB LCS DUP_____
PEST LCS_____
PEST LCS DUP_____
SMPL SPK PCB_____
SMPL SPK PEST_____

Procedure: PEST / PCB Batch ID# _____

	Sample #	Date	Blank	PCB LCS	PCB LCS DUP	PEST LCS	PEST LCS DUP	PCB SMPL	PEST SMPL
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									

REV 1/98

FIGURE 2

Transfer Date: 01/02/98

Initials: MR

EXTRACT TRANSFER SHEET

Organochlorine PST

Sample	Analysis	Extraction Date	Matrix	Initial Volume (mL)	Initial Weight (g)	Final Volume (mL)	% Solids	Extraction Comments
38209-1	PP608	01/02/98	Aqueous	1060		10.0		Emulsion

FIGURE 3

EXTRACTABLES DEVIATION REPORT

AEL#:_____ Client:_____ Matrix:_____

Analysis

ABN PEST PCB HERB TPH

Reason for investigation (list recoveries if possible):

Instrumentation Lab
Investigator:

Date:

Comments:

Extraction Lab
Investigator:

Date:

Comments:

Comment and/or Corrective Action
Investigator:

Date:

Comments:

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Sonication Extraction for the Determination of Pesticide and PCB Compounds in Solid Samples

Approved By:

QA Officer: Melissa A. Delli Date: 11/06/01

Laboratory Director: Steve L. Kadlubowski Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Sonication Extraction for the Determination of Pesticide and PCB Compounds in Solid Samples.

EPA References: Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Method 3550B, Revision 2, December 1996

Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Method 3500B, Revision 2, December 1996

Comprehensive Quality Assurance Plan for Analytics Environmental Laboratory, LLC, January 2001

2. Scope and Application\

This method describes the isolation and concentration of pesticides and PCB's from solid samples in preparation for analysis by GC/ECD. The parameters listed in Table 1 can be measured by this method.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevations in baselines. Method blanks must be routinely analyzed to show and absence of these interferences.

3.2 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

3.3 Phthalates esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

3.4 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Spefically, Aldrin, Heptachlor, and most organo-phosphorus pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-ml K-D flask). These items should be hand rinsed to avoid this problem.

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard.

4.2 Exposure should be minimized by wearing solvent resistant gloves, safety goggles and a laboratory coat when samples or standards are handled, and by working in a fume hood.

4.3 **The following compounds included in this method are classified as known or suspected human carcinogens: the BHC's, the DDT series, and PCBs.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

4.4 The sonicator probe should never be touched when engaged. The owner's manual should be consulted regarding other safety concerns and the maintenance of the sonicator.

5. Method Summary

Approximately 30 g of the solid sample is mixed with sodium sulfate then sonicated three times for three minutes with acetone/hexane. The extract is then dried, concentrated to a volume of 10 ml, and analyzed by GC/ECD.

6. Sample Collection, Handling and Preservation

6.1 All samples should be collected in glass jar with a screw top Teflon lid.

6.2 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling instructions.

6.3 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and log-in procedures.

6.4 Solid samples must be extracted within 14 days of collection.

6.5 Sample extracts must be analyzed within 40 days of extraction.

6.6 Extracts must be stored in a freezer at –10°C to –20°C protected from light.

7. Definitions

7.1 ml = milliliter

7.2 g = gram

7.3 GC/ECD = gas chromatograph/electron capture detector

7.4 P/P= pesticide/PCB

7.5 PCB= polychlorinated biphenyl

7.6 °C = degrees Celsius

7.7 QC = quality control

7.8 AEL # = Analytics Environmental Laboratory sample identifier

7.9 N_2 = nitrogen gas

7.10 psi = pounds per square inch

7.11 CAS = Chemical Abstract Service

7.12 LCS = Laboratory Control Sample

8. Apparatus and Materials

8.1 Sonicator -Heat systems, Inc. Ultrasonic Liquid Processor Model XL2020, equipped with twin ¾-inch titanium disrupter horns or equivalent.

8.2 Super Sonabox w/ring clamp and beaker tray - Heat System, Ultrasonics, Inc. Part Number 432B or equivalent.

8.3 TurboVap II concentration Workstation - Zymark Part Number 46368 or equivalent.

8.4 250 ml glass beakers.

8.5 250 ml Erlenmeyer flask, glass.

8.6 Long stem funnel with a glass wool plug.

8.7 Turbo-Vap Tubes with 1.0 ml stem.

8.8 Disposable pasteur pipettes, 9 inch glass.

8.9 metal spatula

8.10 Class A 10 ml volumetric flask.

8.11 2 ml auto-sampler vials with Teflon™ lined crimp caps

8.12 12 ml amber vials with Teflon™ lined screw caps

8.13 1 ml syringe.

8.14 Kimwipes

8.15 (2)- 50 ml graduated cylinders

816 (2)- 400 ml Teflon™ beakers for rinsing sonicator tips.

8.17 Balance capable of accurately weighing to 0.01 grams.

8.18 Red labeling tape.

8.19 P/P sample preparation logbook

8.20 Nitrogen blow down apparatus.

8.21 Wax pencil

9.0 Standards and Reagents

9.1 Acetone - Pesticide quality or equivalent.

9.2 Hexane - pesticide quality or equivalent

9.3 Sodium sulfate - Granular, anhydrous. Purified by heating at 300 °C for a minimum of four hours in a shallow glass-baking dish and stored in a sealed glass container.

9.4 Silica Sand - reagent grade.

9.5 P/P surrogate - Refer to QA-P001 for Standard preparation.

9.6 Pesticide Matrix Spiking Solution - Refer to QA-P001 for Standard preparation.

9.7 PCB Matrix Spiking Solution - Refer to QA-P001 for Standard preparation.

9.8 Methanol - purge and trap grade or equivalent for syringe rinsing.

9.9 Nitrogen gas - grade 5 from a liquid nitrogen tank

9.10 1:1 acetone/hexane - prepared by adding equal volumes of each solvent

10. Procedure

10.1 Sample Preparation Notebook

All information pertinent to pesticide/PCB sample preparation is recorded in a bound paginated laboratory notebook. Each entry should contain a general heading with the extraction date, P/P SLD and the initials of the analyst(s) that performed the extraction. A column format should then include the following headings: sample number, matrix, weight (g), standard number and amount of surrogate added, standard number and amount of spike added, final volume, the date the concentration was completed and initials of the analyst who concentrated the sample. Observations about specific samples must also be recorded in the notebook.

10.2 Determine Necessary Quality Control

Each client sample extracted is recorded in the QC summary notebook in groups of 20 (Figure 1). QC includes blank, laboratory control samples, matrix spike samples, and matrix spike duplicate samples for both pesticides and PCB's, six total QC samples per 20 client samples.

10.2.1 A Method Blank, consisting of approximately 30 g of sodium sulfate must be extracted with each extraction batch (group of co-extracted samples not to exceed 20 samples). The mixture is spiked with the P/P surrogate spike. Method Blank samples are subject to the same extraction procedure as the associated samples thus indicating extraction accuracy. Blanks are labeled as follows: BMMDDYPS, where;

B designates a blank
MM is a two number designation for the month
DD is two number designation of the day
Y is a single number designation for the year
PS signifies that the extract is a pesticide/PCB sonication.

10.2.2 A laboratory control sample must be performed with each extraction batch (group of co-extracted samples not to exceed 20 samples).A laboratory control duplicate sample must be extracted for every 20 samples or once per month. The laboratory control sample consists of 30 g of silica sand mixed with approximately 30 g of sodium sulfate (enough to cover the silica sand). The mixture is spiked with 1.0 ml of either the pesticide or PCB matrix spiking solution and 1.0 ml P/P surrogate spiking solution. The laboratory control samples are subjected to the same extraction procedure as the associated samples, thus indicating extraction accuracy. Laboratory control samples are labeled as follows: LMMDDYPTS or LMMDDYPBS, where;

L designates a laboratory control sample
MM is a two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the year
PTS indicates pesticide MS was used
PBS indicates that PCB MS was used

10.2.3 One matrix spike and matrix spike duplicate must be extracted for pesticides and one for PCB's every twenty client samples or within a one month period. A matrix spike consists of 30 g of a client sample mixed with approximately 30 g of sodium sulfate. The mixture is spiked with 1.0 ml of the pesticide or PCB matrix spiking solution and 1.0 ml P/P surrogate spiking solution. The matrix spike and matrix spike duplicate are subjected to the same extraction procedure as the associated samples, thus indicating matrix effects. Matrix spikes are labeled with the client sample number and the suffix MST for pesticide or MSB for PCB's and MSDT and MSDB for the duplicates.

10.3 Sample Extraction

10.3.1 Connect sonicator control unit to the sonibox by attaching cable coming from the top of the sonibox to the back of the control unit. Turn the sonicator on by pressing the red switch on the lower left -switch panel will light when on. Set intensity dial to maximum setting-10. Readout will prompt procedure for preparing sonicator for use. (Press "CLEAR"; press "PROG/DATA", press number 1). The number displayed on the upper left of the readout is the total time the sonicator will be in the "PULSAR ON" phase; the number on the lower left is the "PULSAR ON" interval, while the number on the lower right is the "PULSAR OFF" interval.

10.3.2 Program number 1 is set for three minutes total process time with the pulsar "ON" for 0.5 sec and pulsar "OFF" for 0.5 sec. Use program 1 for sonicating samples.

10.3.3 Program number 2 is set for 30 sec total process time and the same ON/OFF intervals as program 1. Use the Teflon™ beakers for rinsing tips since glass beakers tend to shatter. Rinse the tips using Program 2 prior to sonicating any samples.

10.3.4 For each sample, pre-rinse a 250 ml beaker and a 250 ml Erlenmeyer flask with hexane. Use red tape to label the flask with the AEL #, P/P SON and the associated blank number. Also label the beaker with the AEL # using a wax marker.

10.3.5 Calibrate the balance as per QA-M009.

10.3.6 Weigh approximately 30 g of sample into the beaker (decant any free liquid first.). Record the weight to the nearest one hundredth of a gram in the sample preparation logbook.

10.3.7 Add approximately 30 g of sodium sulfate to the sample (more if the sample seems especially damp or wet) and mix thoroughly to uniform consistency. The sample should be free flowing at this point.

10.3.8 Using the 1 ml syringe designated for P/P surrogate, add 1.0 ml of P/P surrogate solution to the sample in the beaker.

10.3.9 If a sample is designated as a matrix spike, matrix spike duplicate, or laboratory control sample, use a 1 ml syringe to add 1.0 ml of the appropriate pesticide or PCB matrix spiking solution to the sample in the beaker. **The syringe used for spiking is not a dedicated syringe so it must be rinsed 3 times before and after use with Purge and Trap grade methanol.**

10.3.10 Rinse sonicator probes by pouring approximately 250 ml hexane into both Teflon beakers. Place the beakers on the sonicator tray and raise the ring so that sonicator tips are immersed in hexane but not touching the beakers. Close sonibox and check to make sure sonicator is set to program 2 and press "START/STOP" button to begin sonication. Sonicator will stop automatically when total process time has been reached.

10.3.11 Using a graduated cylinder, pour 100 ml of 1:1 acetone/hexane into the beaker. Place beaker(s) on tray in sonibox. Two sample beakers may be sonicated at the same time. If two samples are not sonicated, one of the

rinse teflon beakers containing solvent is placed on the tray instead. Raise the ring so that sonicator tip is immersed in the solvent but not touching the sample or the beaker. Close sonibox and check to make sure sonicator is set to program 1 and press "START/STOP" button to begin sonication. Sonicator will stop automatically when total process time has been reached.

10.3.12 Remove beaker(s) and decant the solvent into the labeled 250 ml flask, being cautious that none of the solid goes into the flask.

10.3.13 Repeat steps 8.3.11 and 8.3.12 two more times using 50 ml of 1:1 acetone/hexane. Stopper the flask and set aside.

10.3.14 After each sample has been completely processed, change program to 'rinse' program by pressing the number 2 on the sonicator keypad. If probes are particularly dirty, wipe each sonicator tip with a Kimwipe to remove particulates. Place Teflon™ beakers with about 200 ml hexane on beaker tray and raise to immerse the tips. Press "START/STOP" to engage. One process time is sufficient to clean tips. Change program back to program 1 by pressing the number 1 to prepare for next sample. Change rinse hexane as needed.

10.4 Extract Drying and Concentration

10.4.1 If extracts are not to be concentrated immediately they must be stored in a refrigerator.

10.4.2 Plug a long-stem funnel with glass wool and fill 1/2 full with baked sodium sulfate. Rinse the sodium sulfate with 10-20 ml of hexane. Collect rinseate in a waste container.

10.4.3 Place the funnel into the TurboVap tube. Pour the sample extract through the funnel into the TurboVap tube.

10.4.4 Rinse the flask that contained the sample three times with hexane and pour the rinsings through the funnel. **Transfer the label from the Erlenmeyer flask to the TurboVap tube.**

10.4.5 When the sample has drained completely, rinse the sodium sulfate with an additional 20 ml of hexane and allow to drain.

10.4.6 Set TurboVap concentrator temperature to 35°C for P/P samples. The nitrogen pressure should be set to **10 psi. The select button should be set on timer and the timer initially set to 40 minutes.** Depending on the amount of extract collected a sample will need more or less time in the

turbovap. The extract should be concentrated to a volume of 5-7 ml as marked on the turbo tubes before they are removed from the turbovap.

10.4.7 Rinse a 10 ml volumetric flask three times with hexane. Transfer concentrated sample extract with a clean 9 inch pasteur pipette into the 10 ml volumetric flask. With a clean pasteur pipette add a small amount of hexane to the TurboVap tube to rinse sides. Using the original pipette, rinse the walls of the lower portion of the Turbo tube at least three times then transfer the hexane to the volumetric flask. Repeat as many times as needed to achieve 10.0 ml final volume in the flask. **(It is preferable to underestimate and do repeat rinsings rather than to overestimate and have some hexane remaining). When a final volume of 10.0 ml is achieved, mix the sample carefully and thoroughly with the pipette.** If there is obvious precipitate in the extract then it must be set aside for a few minutes to alow the precipitate to settle from the liquid portion. Transfer a portion of the extract (do not transfer any particulates) with the same pipette into a 2 ml vial labeled with the sample number, P/P SON, and the date. Cap. If the final extract volume is different than 10 ml, note the volume on the vial. Transfer the tape label from the turbo tube to the cap of the auto-sampler vial. Pour the remaining extract from the volumetric into an appropriately12 ml amber vial, cap. Place the extracts in the freezer designated for P/P extracts.

10.4.8 If the Turbo-Vap automatically shuts off before the extract has been reduced to <10.0 ml (this can happen repeatedly) the analyst has several options:

1) Reset the Turvo-Vap cell holding that sample with a few more minutes on the timer, until the sample is cooked down to 5-7 ml. Then go back to 10.4.7 for vialing. This option should be considered in most cases unless the sample is extremely oily or contains particulates.

2) Transfer the sample from the Turbo-Vap tube to an appropriately-sized collection vessel. Set up nitrogen blow-down apparatus and condense sample to <10.0 ml or until sample volume reduction stops. If sample goes to <10.0 ml, return to section 10.4.7 for vialing. Otherwise, move on to option #3.

3) If sample volume can't be reduced to <10.0 ml using either of these techniques, the sample must be brought to a convenient volume. Make an estimate of the volume of the remaining sample and choose a Class A volumetric of sufficient size to contain the extract plus any hexane used to rinse the Turbo-Vap tube. Use pipette to transfer a portion of the extract to a clear 2.0 ml vial. Store the remainder of the extract in an appropriate

container. The final volume of the concentrated extract is recorded in the Sample Log book under the appropriate heading.

10.4.9 Extracts are stored in the freezer designated for P/P extracts and must be analyzed within 40 days.

10.4.10 A transfer sheet (Figure 2) is generated each day that samples are completed. The original is filed in the laboratory output folder. A copy is given to the GC/ECD analyst and a copy is filed in the client's folder. Fill in the "Final Volume" and "Date Transfered" columns of the sample prep log book and note any unusual observations.

11. Quality Control

11.1 Per sample

11.1.1 Surrogates are added to every sample, matrix spike, matrix spike duplicate, laboratory control sample and blank before extraction to test the efficiency of the extraction procedure. The GC/ECD analyst calculates percent recoveries. If surrogate recoveries are outside the laboratory's pre-determined acceptance ranges, a QC Deviation Report (Figure 3) explaining the cause is completed. Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. In this case, the GC/ECD analyst will transfer the deviation report to the extraction laboratory. Unusual circumstances during the extraction should have been noted in the sample preparation log notebook. Once the extraction laboratory has completed the deviation report, it should be returned to the ECD analyst. A copy is filed in the client's folder and the original is filed with the Quality Assurance Officer. Corrective action may include re-extracting the sample if sufficient sample exists.

11.2 Per sample batch

11.2.1 A method blank must be performed with every sample batch (20 samples per 8 hour shift). A blank consists of 30 g of sodium sulfate. The blank is extracted with the associated sample batch and carried throughout the extraction procedure. The blank is analyzed just prior to the associated samples to monitor interferences or contaminants that may have occurred during the extraction process. If a blank is found to contain more than the reporting limit of any compound, the sample batch must be re-extracted. If insufficient sample exists, the contaminant will be qualified with a "B" flag.

11.2.2 A Laboratory Control Sample must be performed with every sample batch (20 samples, or per eight hour shift). The laboratory control sample is

subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy. The GC/ECD analyst will calculate the percent recoveries of the compounds. If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. Extraction personnel may be asked to determine the cause of the deviation. Unusual circumstances during the extraction are recorded in the preparation log.

11.3 Per QC Batch

11.3.2 A matrix spike and matrix spike duplicate must be extracted per 20 samples or within a one month period. A matrix spike consists of 30 g of a client sample mixed with 30 g of sodium sulfate. The mixture is spiked either with 1.0 ml of the Pesticide matrix spiking solution or 1.0 ml of the PCB matrix spiking solution. The matrix spikes are subjected to the same extraction procedure as associated client samples. The GC/ECD analyst will calculate the spike recoveries and relative percent differences. If the spike recoveries or relative percent differences are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. A QC Deviation Form (Figure 3) will be completed.

11.4 The sonicator must be tuned monthly. Refer to Instruction Manual tuning instructions. Document tuning procedure in the miscellaneous analysis book.

11.5 Spiking solutions are tested prior to use according to QA-M015.

12.0 Pollution Prevention and Waste Management

12.1 See SOP W001 for handling of waste and unused portions of sample.

Table 1

Target Compounds

Compound	CAS #
Single Response Compounds	
α-BHC	319-84-6
β-BHC	319-85-7
γ-BHC (Lindane)	58-89-9
δ-BHC	319-86-8
Heptachlor	76-44-8
Aldrin	309-00-2
Heptachlor Epoxide	1024-57-3
α -Chlordane	5103-71-9
γ -Chlordane	5103-74-2
Endosulfan I	959-98-8
4,4'-DDE	72-55-9
Dieldrin	60-57-1
Endrin	72-20-8
Endosulfan II	33212-65-9
4,4'-DDD	72-54-8
Endrin Aldehyde	7421-93-4
4,4'-DDT	50-29-3
Endosulfan Sulfate	1031-07-8
Endrin Ketone	53494-70-5
4,4'-Methoxychlor	72-43-5
PCB Congeners	
Multi-Response Compounds	
Aroclor 1016	12674-11-2
Aroclor 1221	1104-28-2
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
Toxaphene	8001-35-2
Chlordane (technical)	12789-03-6

FIGURE 1

QC SUMMARY PEST/PCB
SOIL SAMPLES

Procedure: PEST / PCB SLD Batch ID# _____

	Sample #	Date	Blank	PEST LCSD	PEST LCS	PEST MS	PEST MSD
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

FIGURE 1A

QC SUMMARY PCB SOIL SAMPLES

Procedure: PCB SLD Batch ID# _____

	Sample #	Date	Blank	PCB LCSD	PCB LCS	PCB MS	PCB MSD
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

FIGURE 2

Transfer Date: 02/05/98

EXTRACT TRANSFER SHEET

Initials: MR

Organchlorine PST:

Sample	Analysis	Extraction Date	Matrix	Initial Volume (mL)	Initial Weight (g)	Final Volume (mL)	% Solids	Extraction Comments
39287-1	8081/82S	02/04/98	Solid		25.16	10.0	39.75	
39290-1	8081/82S	02/04/98	Sludge		19.25	10.0	15.94	

FIGURE 3

Quality Assurance Deviation and Documentation

Analysis Code:
QC Batch ID:
(attach copy or list samples affected by this memo)

Out of Control Event with Brief Explanation

☐ Method Blank

☐ Continuing Calibration or Initial Calibration Issue

☐ Laboratory Control Spike

☐ Matrix Spike/MSD

☐ Surrogate Recovery

☐ Other

SOP Corrective Action Followed:

☐ Re-extract in Hold time

☐ Re-inject to confirm

☐ Other

☐ Document SOP corrective action or "See QA Memo" on raw data file

QA Officer Review: _____ _____
Initials Date

Comments:

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Acid Cleanup for Polychlorinated Biphenyls

Approved By:

QA Officer: Melissa A. Pelli Date: 11/7/01

Laboratory Director: Steven C. Knollyn Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Acid Cleanup for Polychlorinated Biphenyls

EPA References: Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Method 3600C, Revision 3, December 1996

Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Method 3665A, Revision 1, December 1996

EPA-600/4-81-045 Test Method The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils, September 1982

Comprehensive Quality Assurance Plan for Analytics Environmental Laboratory, LLC, January 2001

2. Scope and Application

This method is used for the cleanup of polychlorinated biphenyl (PCB) compounds that are partitioned into an organic solvent (hexane) and are amenable to gas chromatography. **This method cannot be used to clean up extracts for other target analytes, as it destroys most organic chemicals**. It is important that all extracts be in hexane prior to initiating this procedure.

The purpose of applying a cleanup method is to remove interferences and high boiling material that may result in: errors in quantitation,, false positive results, false negative results, column degredation and injection port problems.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevations in baselines. Method blanks must be routinely analyzed to show and absence of these interferences.

3.2 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

3.3 Phthalates esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

3.4 This method will not destroy chlorinated benzenes, chlorinated naphthalenes (Halowaxes) and several chlorinated pesticides.

3.5 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Spefically, Aldrin, Heptachlor, and most organo-phosphorus pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-ml K-D flask). These items should be hand rinsed to avoid this problem.

3.6 The presence of water in the extract can cause an exothermic reaction that may compromise the results.

3.7 Residual acid in the extract may destroy the chromatographic column. Extreme care must be taken to remove only the hexane layer of the extract.

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard.

4.2 Exposure should be minimized by wearing solvent resistant gloves, safety goggles and a laboratory coat when samples or standards are handled, and by working in a fume hood.

4.3 **PCBs are classified as known or suspected human carcinogens.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

5. Method Summary

The sample extract (hexane only) is shaken with sulfuric acid for the removal of non-target compounds. The organic phase is allowed to separate and the organic phase is transferred off the sulfuric acid for analysis.

6. Sample Collection, Handling and Preservation

6.1 Sample extracts must be analyzed within 40 days of extraction.
6.2 Extracts must be stored in a freezer at –10°C to –20°C protected from light.

7 Definitions

7.1 ml= milliliter

7.2 GC= gas chromatograph

7.3 PCB=polychlorinated biphenyls

7.4 AEL=Analytics Environmental Laboratory

7.5 P/P = pesticide and PCB

7.6 H_2SO_4 = Sulfuric Acid

8. Apparatus and Materials

8.1 12 ml vial with teflon lined cap

8.2 Disposable Pasteur Pipettes, glass

8.3 2 ml Crimp Top Vials with teflon lined caps

8.4 Syringe - 1.0 ml

8.5 Volumetric flask - 1 ml Class A

8.6 pH paper wide range (1-14)

9. Standards and Reagents

9.1 H_2SO_4 -Reagent Grade

9.2 Reagent water - distilled then de-ionized with a Sybron Barnstead 3-canister filtering system, or equivalent

10. Procedure

All QC samples are subjected to the same clean-up procedures as the samples.

10.1 Refer to the following Standard Operation Procedures for sample preparation.

QA-P002: Separatory Funnel Liquid/Liquid Extraction for the Determination of Pesticides and PCBs in Aqueous Samples.

QA-P004: Waste Dilution Method for the Determination of Pesticides and PCBs in Non- Aqueous Liquid Samples.

QA-P006: Sonication Extraction for the Determination of Pesticides and PCBs in Solid Samples.

10.2 Transfer exactly 1.0 ml of sample extract using a 1.0 ml syringe into a 12 ml vial. **Slowly** add 5 ml of sulfuric acid and shake vigorously for 2 minutes. Allow layers to separate for at least 1 minute. Examine the hexane (top) layer; it should not be highly colored nor should it have a visible emulsion.

10.3 If a clean phase separation is achieved, proceed to section 10.6

10.4 If the hexane layer is colored or the emulsion persists for several minutes, remove the hexane layer from the vial and transfer to a clean 12 ml vial, it is OK if some of the acid or emulsion transfers over. Add another 5 ml of clean sulfuric acid solution.

10.5 Shake the sample for two minute and allow the phases to separate.

10.6 Transfer the hexane layer to a clean 2 ml auto-sampler vial. **Be careful not to include any of the acid layer as it can cause damage to the analytical equipment.**

10.6 If it is questioned whether some acid is present. check the extract pH. The pH must be neutral. If the pH is not neutral allow additional time for the extract to settle. Perhaps overnight

10.7 Cap vial and label vial with AEL identifer number and "AC" to denote acid cleanup was performed. Extract is ready for analysis by GC/ECD.

10.8 An acid cleaned method blank is prepared by treating the method blank to the same procedure as the sample(s).

10.9 Record that a cleanup was performed in the P/P Sample Prep logbook on the same page that has the extraction data.

11. Quality Control

11.1 Method blank(s) are subjected to the same cleanup as the associated samples.

11.2 Laboratory control samples and sample spikes are subjected to the same cleanup as the associated samples.

12.0 Pollution Prevention and Waste Management

12.1 See SOP W001 for handling of waste and unused portions of sample.

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PCB Wipe Test Preparation

Approved By:

QA Officer: _____ Date: 03/29/01

Laboratory Director: _____ Date: 3/29/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

PCB Wipe Test Preparation

EPA References: Test Methods For Evaluating Solid Waste Physical/Chemical Methods-Method 8000B, Revision 2, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods-Method 3500B, Revision 2, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods-Method 3550B, Revision 2, December 1996

Comprehensive Quality Assurance Plan for Analytics Environmental Laboratory, LLC. January 2001

2. Scope and Application

This method is used for the preparation and extraction of wipes for PCB analysis. The target compounds are partitioned into an organic solvent (hexane) and are amenable to gas chromatography.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevations in baselines. Method blanks must routinely be analyzed to show an absence of these interferences.

3.2 Matrix inferences may include contaminants that are co-extracted from the sample. These may include but are not limited to phthalate esters, petroleum hydrocarbons, elemental sulfur and other organic acids.

4. Safety

The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard. **PCBs are known carcinogens.** Exposure should be minimized by wearing a laboratory coat, safety goggles and solvent resistant gloves when samples or standards are handled, and by working in a fume hood. Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method

5. Method Summary

A 2 inch x 2 inch cotton square of material is placed in a jar with hexane. This jar is taken to the site and a measured area is wiped with the material and then returned to the jar. When the jar is returned to the laboratory, surrogate is added and the kit is sonicated for a total of 2 minutes. The sample extract is then quantitatively transferred to a class A 25mL volumetric flask. If the extract is highly colored or has sediment present, the extract may be put through a florisil column and/or acid cleaned. The extract is then analyzed by GC/ECD (QA-P010). See Figure 1 for a method summary.

6. Sample Collection, Handling and Preservation

6.1 Wipe Preparation for Field Testing

6.1.1 Lint-free cotton or other wipe material is cut into approximately 2in. X 2in. squares. Each square is added to a 2oz. Glass jar with a Teflon lid.

6.1.2 To each 2oz. Jar add approximately 20 mL of hexane and cap tightly. Jars are ready to be delivered to the client.

6.2 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-Loo1 for proper cooler handling instructions.

6.3 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and log-in procedures.

6.4 Wipe samples must be extracted within 14 days of collection

6.5 Sample extracts must be analyzed within 40 days of extraction.

6.6 Extracts must be stored in a freezer at -10°C to -20°C protected from light.

7. Definitions

7.1 in. = inch

7.2 oz = ounce

7.3 ml = milliliter

7.4 µg = microgram

7.5 GC/ECD = gas chromatography/electron capture detector

7.6 AEL = Analytics Environmental Laboratory, LLC.

7.7 PCB = Polychlorinated Biphenyl

7.8 LCS/LCSD = Laboratory Control Sample/Laboratory Control Sample Duplicate

7.9 QC = Quality Control

8. Apparatus and Materials

8.1 Lint-free cotton (Hewlett Packard part number 9310-4828 or equivalent)

8.2 2 oz screw top jars with Teflon™ lined caps.

8.3 10 ml re-pipettor, graduated to 0.1, capable of delivering 10 ml.

8.4 1.0 ml syringe.

8.5 Class A 25 ml volumetric flask.

8.6 2 ml Autosampler Vials with Teflon™ lined caps.

8.6 12 ml screw top vials with Teflon™ lined caps.

8.8 Disposable pipettes, glass.

8.9 Nitrogen blowdown apparatus.

8.10 Sonicator- Heat Systems,Inc. Ultrasonic Liquid Processor Model XL2020, equipped with twin 3/4 inch titanium disruptor horns or equivalent.

8.11 Super Sonabox w/ring clamp and beaker tray- Heat Systems, Ultrasonics, Inc. Part Number 432B or equivalent.

8.11 Pesticide/PCB sample preparation notebook

9. Standards and Reagents- equivalents may be used

9.1 Standard Solutions: Pesticide/PCB Surrogate Spiking Solution
PCB Matrix Spiking Solution
Refer to Standard Operation Procedure QA-P001 for standard preparation.

9.2 Hexane- Pesticide quality or equivalent.

9.3 Nitrogen gas - Grade 5, for concentrating sample extract.

10. Procedure

10.1 Sample Preparation Notebook. All information pertinent to extraction is recorded in a bound paginated laboratory notebook. Each entry should contain a general heading with the preparation date and the initials of the analyst. The following information is recorded in a column format: AEL sample identifier, matrix, volume and standard number of the surrogate and spike used and the final volume of hexane. Anything unusual observed during the extraction should also be noted.

10.2 Determine Necessary Quality Control. Fill out the QC Summary (Figure 2) to determine if it is necessary to perform an LCS/LCSD. Two laboratory control samples (LCS and LCSD) must be performed for every twenty samples or within a one-month period.

10.2.1 A method blank is prepared with every sample batch (20 samples, or one eight hour shift). The method blank consists of 20.0 ml of hexane and 1.0 ml of pesticide/PCB surrogate. The method blank is taken through the procedure described in Section 10.3. The method blank is labeled: BMMDDYPW, where;

B denotes that the sample is a blank
MM is the two number designation for the month

DD is the two number designation for the day
Y is the one number for the year
PW indicates that a PCB wipe was performed

10.2.2 A laboratory control sample and laboratory control sample duplicate (LCS/LCSD) are performed per batch of 20 samples or within a one month period. The LCS/LCSD consist of 20.0 ml of hexane, 1.0 ml of Pesticide/PCB surrogate and 1.0 ml of the PCB Matrix Spiking solution. The LCS and LCSD are taken through the procedure described in Section 10.3. The LCS and LCSD are labeled: LMMDDYPW and LDMMDDYPW where;

L denotes that the sample is a LCS
LD denotes that the sample is a LCSD
MM is the two number designation for the month
DD is the two number designation for the day
Y is the one number for the year
PW indicates that a PCB wipe was performed

10.3 Sample Preparation

10.3.1 All glassware must be cleaned as described in QA-M001, then rinsed before use with hexane. Syringes should be rinsed 3 times with hexane between uses.

10.3.2 Samples are in jars and should be saturated with hexane. If sample is dry, make a note in preparation logbook. Also record in logbook if the matrix is other than a material prepared by Analytics (i. e. cotton gauze, filter paper, etc.).

10.3.3 Add 1.0 ml of Pesticide/PCB surrogate to the sample in the jar using a dedicated 1.0 ml syringe.

10.3.4 Connect sonicator control unit to the sonibox by attaching the cable coming from the top of the sonibox to the back of the control unit. Turn the sonicator on by pressing the red switch on the lower left --switch panel will light when on. Set intensity dial to maximum setting--10. Readout will prompt procedure for preparing sonicator for use. (Press "CLEAR"; press "PROG./DATA", press number 2). The number displayed on the upper left of the readout is the total time the sonicator will be in the "PULSAR ON" phase; the number on the lower left is the "PULSAR ON" interval, while the number on the lower right is the "PULSAR OFF" interval.

10.3.5 Program number 2 is set for four minutes total process time with the pulsar "ON" for 0.5 sec and pulsar "OFF" for 0.5 sec. Use program 2 for sonicating wipe samples.

10.3.6 Program number 1 is set for 30 sec total process time and the same ON/OFF intervals as program 2. Use the Teflon™ beakers for rinsing tips since glass beakers tend to shatter. Rinse the tips using Program 1 prior to sonicating any samples.

10.3.7 Transfer sample extract to a 25 ml Class A volumetric. Rinse the jar and wipe three times with 1 - 2 ml of hexane to complete the quantitative transfer.

10.3.8 Bring to volume with hexane.

10.3.9 Aliquot approximately 1 ml of the sample extract into a labeled 2 ml autosampler vial and transfer some extract to a labeled 12 ml vial. The remaining extract may be disposed of according to QA-W001. The extract is stored in the freezer designated for pesticides/PCB's for up to 40 days. The extract is ready for PCB analysis by gas chromatography.

10.3.10 If sample extract is highly colored or has sediment, the extract may be florisil cleaned as described in QA-P007 or may be acid cleaned as described in QA-P009.

10.3.11 A transfer sheet is completed (Figure 3). The original is filed in the clients folder, and a copy is given to the GC/ECD analyst .

11. Quality Control

11.1 Per sample

11.1.1 Surrogates are added to every sample, sample matrix spike, sample matrix spike duplicate, laboratory control sample and blank before extraction to test the efficiency of the extraction procedure. The GC/ECD analyst calculates percent recoveries for surrogates using SOP QA-P012. If surrogate recoveries are outside the laboratory's pre-determined acceptance ranges, the following steps should be taken:

1.) Re-inject the extract to verify the surrogate result.
2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

11.2 Per sample batch

11.2.1 A method blank must be performed with every sample batch (20 samples or daily). A blank consists of 20mL hexane that is carried throughout the extraction procedure. The blank is analyzed prior to the associated samples to monitor interferences or contaminants that may have occurred during the extraction process. If a blank is found to contain more than the reporting limit of any compound, the sample batch must be re-extracted. If insufficient sample exists, the contaminant will be qualified with a "B" flag.

11.2.2 A laboratory control sample (LCS) must be extracted with every sample batch (20 samples or daily). A laboratory control sample consists of 20mL Hexane. The mixture is spiked with 1.0 ml of the PCB matrix spiking solution. The laboratory control samples are subjected to the same

extraction procedure as the client samples, thus indicating extraction accuracy. The GC/ECD analyst will calculate the percent recoveries in accordance with SOP QA-P012. If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. A LCS with recoveries above the laboratory acceptance criteria is considered acceptable for samples with no analytes detected. The following steps should be taken for recoveries below the acceptance limits or samples that have analytes detected:

1.) Re-inject the extract to verify the result.
2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

11.3 Per 20 samples

11.3.1 Matrix spike sample and matrix spike duplicate must be extracted per 20 samples or within a one month period. A matrix spike and matrix spike duplicate consists of a client sample that is collected in triplicate. The replicate samples are spiked with 1.0 ml of the Pest/PCB surrogate spiking solution and 1.0 ml PCB spiking solution. The matrix spike and matrix spike duplicate is subjected to the same extraction procedure as associated client samples. The GC/ECD analyst will calculate the percent recoveries and relative percent difference (RPD) in accordance with SOP QA-P012. If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. The following steps should be taken for recoveries

1) Re-inject the extract to verify the result.
2) Re-extract the affected sample if sample volume and holding time permits.
3) Evaluate the LCS if recovery is acceptable , matrix affect should be noted
4) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

12.0 Pollution Prevention and Waste Management

12.1 See SOP W001 for handling of waste and unused portions of sample.

Table 1

Target Compounds

Compound	CAS #
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

FIGURE 1

WIPE TEST FLOWCHART

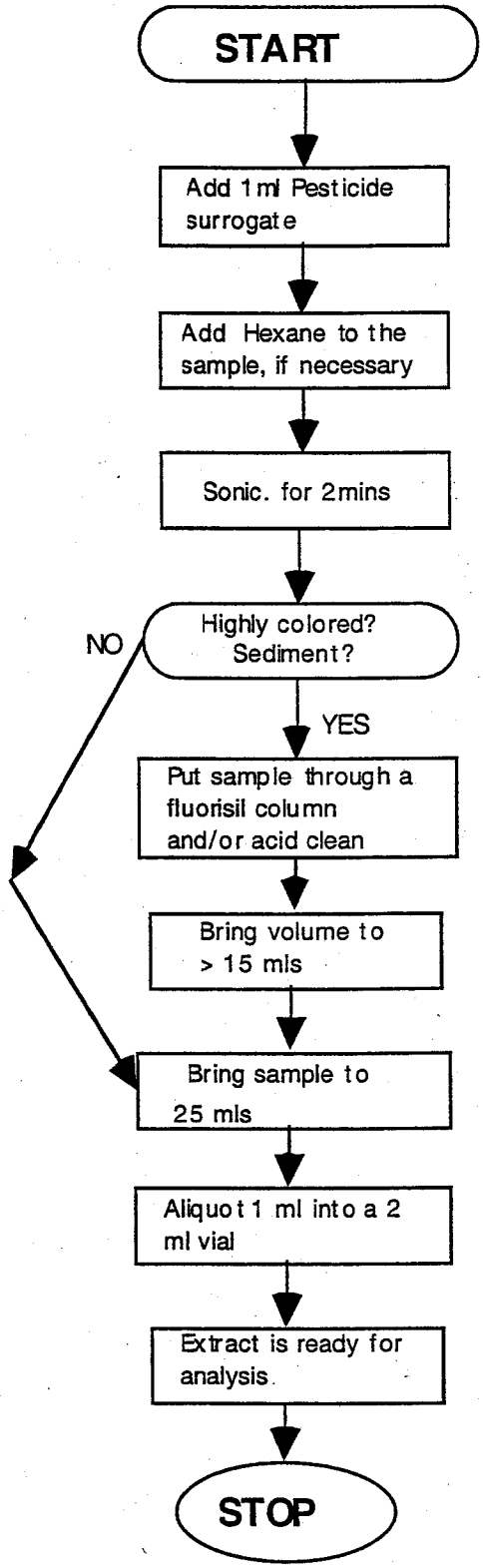

FIGURE 2

QC SUMMARY FOR PCB WIPE SAMPLES

PCB_____
LCS_____
LCSDUP _____

Procedure: _____ Batch ID# _____

	SAMPLE #	DATE	BLANK	LCS	LCS DUP
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

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FIGURE 3

EXTRACT TRANSFER SHEET

DATE:

Instrument: Pesticides/PCB's
608/8081/8082

Sample #	Matrix	Extraction Date	Initial Vol (mL or g)	Surr. #	mLs surr added	Spike #	mLs spike added	Final Vol (mL)	Percent Solid	Initials

Figure 4

Quality Assurance Deviation

Analysis Code:
QC Batch ID:
(attach copy or list samples affected by this memo)

Out of Control Event with Brief Explanation

☐ Method Blank

☐ Continuing Calibration or Initial Calibration Issue

☐ Laboratory Control Spike

☐ Matrix Spike/MSD

☐ Surrogate Recovery

☐ Other

SOP Corrective Action Followed:

☐ Re-extract in Hold time

☐ Re-inject to confirm

☐ Other

☐ Document SOP corrective action or "See QA Memo" on raw data file

QA Officer Review: _____ _____
Initials Date

Comments:

analytics environmental laboratory ~~inc.~~ LLC
mg 5-10-01
195 Commerce Way
Portsmouth, New Hampshire 03801
603-436-5111/800-929-9906
Fax 603-430-2151
email: analytics@analyticslab.com

Laboratory Method for the Determination of Polychlorinated Biphenyl (PCB) Compounds in All Matrices

COPY

Approved By:

QA Officer: Melissa A. Sulli Date: 11/2/99

Laboratory Director: Steven L. Kroll Date: 11/2/99

Revision History

Revision	Description of Change	Approval	Date
3/30/01	Run parameters section 6	mg	3-30-01

1. Title

Laboratory Method for the Determination of Polychlorinated Biphenyl (PCB) Compounds in All Aqueous and Waste Matrices by Capillary Column Technique.

EPA References: Federal Register, Method 608
SW-846, Method 8000B and 8082

2. Scope and Application

This method is used for the determination of polychlorinated biphenyl (PCB) compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The parameters listed (Table 1) can be detected by this method. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of chromatographic data.

3. Safety

The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard. Exposure should be minimized by wearing solvent resistant gloves, safety goggles and a lab coat when samples or standards are handled, and by working in a fume hood. **PCBs are classified as known human carcinogens.** Material Safety Data Sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

4. Method Summary

A 3 µl volume of the sample extract is injected into the gas chromatograph (GC) using a Hewlet Packard autosampler. Analytes are detected with an electron capture detector (ECD). Quantitation of target compounds is performed by the external standard technique using Hewlet Packard Enviroquant software. Target compounds are confirmed by additional analysis on a dissimilar capillary column. Refer to Figure 1 for a method summary.

5. Definitions

5.1 µl = microliter
5.2 ml = milliliter
5.3 L = liter
5.4 µg = microgram
5.5 g = gram
5.6 kg = kilogram
5.7 min = minute
5.8 GC = gas chromatograph
5.9 TCX = Tetrachloro-m-xylene
5.10 DCB = Decachlorobiphenyl
5.11 ^{o}C = degrees Celsius
5.12 QC = quality control
5.13 PCB = polychlorinated biphenyl
5.14 ECD = electron capture detector
5.15 psi = pounds per square inch
5.16 ID = internal diameter
5.17 AEL = Analytics Environmental Laboratory
5.18 CAS = Chemical Abstract Service

5.19 wt = weight
5.20 vol = volume
5.21 conc = concentration
5.22 Cf = calibration factor
5.23 LCS = Laboratory control sample
5.24 ALS = automatic liquid sampler
5.25 CV = calibration verification

6. Apparatus and Materials

6.1 GC System
6.1.1 Gas Chromatograph: Hewlett Packard 5890 Series II or equivalent.
6.1.2 Autosampler Sequence:

Hewlett Packard 7673A autosampler or equivalent
ALS Conditions from HP Chemstation:

Method:	~~PEST~~. M PCB.M
Sample washes	2 MJ 3-30-01
Sample pumps	3
Injection volume	3.0 µL
Syringe size	10.0 µL
On column	Off
Nanoliter adapter	Off
PostInj Solvent A washes	3
PostInj Solvent B washes	3
Viscosity delay	1 second
Plunger Speed	Fast

6.2 GC Columns - equivalents may be used.
6.2.1 Primary column: Rtx-CLPesticides 30 meter, 0.53 mm ID, 0.5 µm film thickness
6.2.2 Confirmation column: Rtx-CLPesticides II, 30 meter, 0.53 mm ID, 0.42 µm film thickness.

6.2.3 Oven temperature program

Inlet A	~~150°~~ 200°C MJ 3-30-01
Inlet B	Off ~~off 250°C~~ MJ 3-30-01
Detector A	320°C
Detector B	320°C
Oven equib time	0 minutes
Initial Temperature	150° ~~120°C~~ MJ 3-30-01
Initial time	0.00 minutes

Level	Rate (°C/minute)	Final Temp (°C)	Final Time (min)
1	~~9.0~~ 18°C	~~300~~ 300	~~3.00~~ 0.17 MJ 3-30-01

Next run time 8.50 ~~23.00~~ minutes MJ 3-30-01

6.3 Gases
6.3.1 Helium Grade 5 for carrier gas
Argon Methane (95:5) Grade 5 for make-up gas
Column Flow: 6 ml/min
Column + Make-up Flow: 50 ml/min
Purge vent flow rate: 8 ml/min
Split vent flow rates: 75 ml/min

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.50	0.00	Yes
B	Off	0.50	0.00	Yes

6.4 Electronic Pressure Program
Constant Flow: ~~off~~ ON Constant flow Pressure 15.0 psi Constant flow temp 120°C
Initial Pressure: ~~13.0~~ psi 13.0 psi
~~5.0~~ 38300

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (min)
1	13.00	13.0	13.00
2 (A)	99.00	10.0	8.00
3 (B)	99.00	30.0	1.77

Total program time 23.00

6.5 Data System: Hewlett Packard Environmental Chemstation
6.6 Instrument Run Logbook.
6.7 Sample Preparation Logbook.
6.8 Instrument Maintenance Logbook.

7. Standards and Reagents

7.1 Standard Solutions: Aroclor Curves - 5 levels. Refer to Standard Operation Procedure QA-P001 for standard preparation.
7.2 Hexane - Pesticide quality or equivalent for dilutions, autosampler rinseate #1 and column washing.
7.3 Acetone - Pesticide quality or equivalent for column washing
7.4 Methylene Chloride - Pesticide quality or equivalent for column washing.
7.5 Toluene - Pesticide quality or equivalent, autosampler rinseate #2.

8. Procedure

8.1 Refer to the following Standard Operation Procedures for sample preparation.
QA-P002: Separatory Funnel Liquid/Liquid Extraction For The Determination Of Pesticides And PCBs In Aqueous Samples
QA-P004: Waste Dilution Method For The Determination Of Pesticides And PCBs In Non-Aqueous Liquid Samples.
QA-P006: Sonication Extraction For The Determination Of Pesticides And PCBs In Solid Samples.
QA-P011: PCB Wipe Sample Preparation.

8.2 Instrument Run Logbook. A bound paginated laboratory notebook is assigned to each instrument for use as a sample run log. The date is noted above each day's analyses along with the analysts initials. Each day starts a new page. Each run is recorded with the following information: the run number, the autosampler vial number, the AEL sample # or standard #, the dilution, the method file, and relevant comments (i. e. rerun for low surrogate, standard passes, dilution necessary, etc.). Each instrument is identified with a unique letter. Data files (run numbers) are assigned using this letter followed by the next consecutive number. The letter designation for this instrument is "F". Also on the run log is a "loading check" and "unloading check". Prior to loading the vials, check to ensure that the tape label agrees with the label on the vial. When unloading the samples, check to ensure that the order in the autosampler agrees with that in the run log. Record both checks in the run log. See Figure 2 for an example of a run log page.

8.3 Initial Calibrations. Before any sample analysis can begin, the instrument must be calibrated for each parameter. Calibration standards are prepared as described in QA-P001 Laboratory Method for the Preparation of Pesticide and PCB Standards. The lowest level standard must be at or below the reporting limit for each parameter. The remaining levels are chosen based on the linear working range of the GC/ECD for each parameter with the highest level not exceeding the concentration limits of the instrument. Initial calibration requires five levels of calibration for aroclors. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Therefore, a multipoint initial calibration employing a mixture of Aroclors 1016 and 1260 is sufficient to demonstrate the linearity of the detector response without performing initial calibrations for each Aroclor. In addition, this standard can be used to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. Single standards of each of the other Aroclors are required to aid the analyst in pattern recognition. These single standards are used to determine the calibration factor for each Aroclor.

8.3.1 Determination of Linearity
Quantitation of aroclors is based on the area of 3 - 6 peaks. Choose peaks which are at least 25% of the height of the largest Aroclor peak if possible. Quantitation of PCBs is based on the area or height of the analyte peak at the known retention time. Linearity is determined from the calibration factor (Cf) of the standards. The calibration factor is the peak area (or height) of the compound divided by the concentration of the compound injected. When the relative standard deviation of the calibration factors is ≤20% the standard curve is considered to be linear and the average calibration factor can be used for quantitation. Alternatively, if the mean of the RSD values for <u>all</u> analytes in the calibration is less than or equal to 20%, the average calibration factor can be used for all analytes. If neither of these conditions are met, a calibration curve must be used. If a linear calibration curve is used the correlation coefficient (r) must be 0.995 or greater to meet acceptance criteria. For a quadratic fit, the coefficient of determination must be 0.995 or greater to meet acceptance criteria. **Quadratic fits require 6 calibration standards. It is never acceptable to force a calibration curve through the origin. Nor is it acceptable to include the origin (0,0) as a sixth calibration point.**

8.3.2 Each time the five point calibration is analyzed a PCB standard prepared from an independent source is analyzed to verify the calibration. The measured value must agree within 20% of the true value for the calibration to be considered valid.

8.3.3 Calibration Verification (CV)
Calibration verifications are conducted on a daily basis, prior to sample analysis, every tenth analysis, and at the end of each sequence to validate the calibration of the system for each parameter. PCB Level 3 standard is used for this purpose. If the response (or calculated concentration) is within ±15% of the initial calibration, then the initial calibration is considered valid. Alternatively, if the average of the responses of <u>all</u> analytes is within 15% the initial calibration is considered valid. If the average of <u>all</u> response (or calculated concentration) is >15%, the initial calibration fails and corrective action must be taken. It may be necessary to analyze a new calibration.

8.3.4 Corrective Action
If the calibration verification or initial calibration is unacceptable, the analyst must consider that the analytical system is out of control and corrective action is needed. Once the problem is determined, correct it and re-analyze the initial calibration if necessary. Corrective action must be documented in the instrument's maintenance logbook. Each log entry should contain the date of the action, the initials of the analyst, a description of the problem and the corrective action taken. Hard copies of the unacceptable initial calibration

and/or calibration verification standard should be stored in the QC notebook with an explanation of the problem and how it was corrected.

8.4 Analyzing Samples

8.4.1 Inject 3 µl of the sample or blank extract onto the GC column using the Hewlett Packard 7673A autosampler. Analyze the sample using the GC method conditions in the method PEST.M. Record the sample analyses in the instrument's run logbook (Section 8.2).

8.5 Quantitation of Sample Analyte.

8.5.1 Target compounds are identified qualitatively based on the retention time and pattern comparison with the calibration standard. The final interpretation of pattern identification is based on the descretion of an experienced GC analyst. Each identification must be confirmed using a second dissimilar GC column.

8.5.2 Target compounds are quantitated using the method file in the Enviroquant software. Enviroquant quantitates the sample extract result by calculating the corresponding analyte concentration found based on the calibration curve or the average calibration factor stored. The calibration files are named for the standard and the date analyzed, i.e. PB01308.M for PCB's aquired on January 30, 1998.

8.5.3 Dilutions. If the concentration of any target compound exceeds the upper level of the calibration, the extract must be diluted in hexane to achieve an on-column concentration that will be in the upper half of the calibration. Analyze the diluted extract and recalculate the sample result. Dilutions are documented in the Pesticide/PCB sample preparation notebook.

8.5.4 The sample result is calculated, correcting for sample weight and percent solids or volume, using Equation 3.

Equation 3

$$\text{Sample result } (\mu g/L \text{ or } \mu g/kg) = \frac{\text{Sample extract conc } (\mu g/ml) * \text{ actual vol of extract } (ml) * \text{Dilution}}{\text{sample wt or vol } (kg \text{ or } L) * \% \text{ solids}/100}$$

where:
1) Sample extract conc (µg/ml) is found on the raw data report from HP Enviroquant
2) Vol of extract (ml) and sample wt or vol is found on the extraction transfer sheet (Figure 3)
3) % solids is found on the extraction transfer sheet (Figure 3)
4) Dilution represents a dilution performed by the analyst

8.5.5 If target compounds are detected on the primary analysis, a confirmatory analysis is performed simultaneously to verify the presence of the target compound. This confirmation analysis is performed on a different column type, i. e. Rtx -CLPesticides II. The result from the confirmatory analysis should be within 40% of the result from the primary analysis. If one result is >40% and no anomalies are noted, report the higher result. The data user should be advised of the disparity between results.

8.5.6 Weathering of PCBs in the environment and changes resulting from waste treatment may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems. The quantitation of the Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be described to the data user and the procedures employed should be thoroughly documented.

9. Quality Control

9.1 The initial calibration is verified by an independent source calibration check standard.

9.2 The initial calibration is verified at the beginning of each day, every 10th analysis, and at the end of each sequence with a calibration verification. If the calibration verification is unacceptable, the problem must be corrected and documented. It may be necessary to analyze a new initial calibration. Each sample analysis must be bracketted with an acceptable initial or calibration verification standard. However, if the samples are "ND" and the ending calibration verification fails high, the preceeding runs are considered acceptable.

9.3 Surrogates.

9.3.1 Surrogates are added to each sample, blank, lab control sample and matrix spike during the extraction process. Percent recoveries are calculated by dividing the amount found by the amount added. The recovery acceptance ranges are listed in instrument methods. If either or both surrogates fall outside the accepatance range, an investigation of the extraction and analytical procedure is conducted. An Extractables Deviation Report (Figure 4) is completed. The original is filed in the Extractables Deviation Notebook in the file room and a copy is filed in the client's folder. When both surrogates fail and an explanation cannot be determined, the sample is re-extracted, if sample volume permits. Otherwise, the result is qualified.

9.3.2 Surrogate recoveries are charted by matrix in batches of 20. The analyst uses this information to obeserve trends, which can indicate a problem with the instrument, or extraction procedure. The following conditions shall be considered out-of-control events for all control charts:

- Any one point outside control limits
- Any three consecutive points outside warning limits
- Any six consecutive points such that each point is larger (or smaller) than its immediate predecessor
- Any eight consecutive points on the same side of the centerline

9.4 Method blanks are analyzed prior to their associated samples. Method blanks should be free of all target compounds and contain only the surrogates. If there are target compounds present which exceed the reporting limit, an investigation is performed to determine the problem. An Extractables Deviation Report (Figure 4) is completed and filed in the Extractables Deviation Notebook in the file room and a copy is filed with the data in the Pesticide QC Notebook. If sufficient sample exists, all samples associated with a contaminated blank will be re-extracted. If insufficient sample exists, the contaminants found in the blamk will be qualified with a "B" flag.

9.5 Spike recoveries are calculated. If the spike recoveries or relative percent difference are outside of the laboratory's acceptance limits listed in the instument methods, an investigation is performed to determine if there is an instrument or extraction error. An Extractables Deviation Report (Figure 4) is completed. The original is filed in the Extractables Deviation Notebook in the file room and a copy is filed with the data in the Pesticide/PCB QC Notebook.

9.6 Minimum detection limit (MDL) studies are performed annually. A series of spiked blanks (a minimum of seven) are extracted and analyzed for each parameter on all matrices. MDL's are calculated using the following formula:

$$MDL = t\sigma$$

where t = Students' t value at the 99% confidence level

σ = standard deviation

9.7 Run log sequences are confirmed when loading and unloading sample extracts.

10. Routine Instrument Maintenance All instrument maintence is recorded in the maintence logbook.

10.1 Inlet Maintenance
A diagram of the GC inlet can be found in the Hewlett Packard 5890A manual on page 18-14 (Figure 18-13). All inlet maintenance is recorded in the maintenance logbook for the instrument.

10.1.1 The inlet septum is replaced after 20-40 injections. An 11 mm septum is used. (Supelco Thermogreen™ LB-2 Septa catalog #2-0654 or equivalent).

10.1.2 A clean deactivated/silanized splitless single taper liner should be installed each day, or as needed. Install the liner in the GC with a new O-ring (Hewlett Packard catalog #5180-4182 or equivalent), being careful not to over-tighten the nut. Refer to QA-M011 for cleaning and silanization of inlet liners.

10.1.3 The injection port inlet seal and washer must be replaced periodically. This should be checked bi-monthly. If the gold seal is black or pitted a new seal and washer is required. Refer to Hewlett Packard 5890A manual on page 18-14 (Figure 18-13) for proper seal/washer placement.

10.2 Column Maintenance
All column maintenance is recorded in the maintenance logbook for the instrument.

10.2.1 The front end of the columns and the guard column are inspected when instrument performance is poor. Compound and particle deposits will degrade the chromatography by the broadening of peaks and the reduction of sensitivity. One loop (about 12 inches) of the front end can be clipped from each of the columns and/or the guard column to remove these contaminants.

10.2.2 Periodic baking of the column at its upper temperature limit will clean the column of contaminants which have remained on the column during the day's normal operating temperatures. This should be performed for a maximum of 24 hours.

10.2.3 A new column is required when the previous two steps or 10.8 below do not lead to improvement of the chromatography. Generally, a column will last 6 - 10 months under normally operating conditions. When installing a new column a description of the column which includes manufacturer, column length, internal diameter, film thickness, catalog and lot numbers and maximum temperature will be documented in the maintence logbook.

10.4 The molecular sieve in-line trap for the helium gas is changed annually.

10.5 Charcoal trap for ECD vent is changed annually.

10.6 ECD Leak tests are performed every six months. Testing materials are purchased from National Leak Test or a similar company. Procedures vary so it is necessary to read the instructions received with each kit.

10.7 Gas tanks are replaced when tank pressure falls below 500 psi.

10.8 Periodic tightening of the detector's make-up gas fitting is necessary. The oven temperature programming loosens the fitting. Check weekly.

10.9 Solvent Washing the Column
Washing the column may extend its lifetime by removing deposited contaminents.

10.9.1 Remove the column from the gas chromatograph. Carefully clamp it to a ring stand in a hood.

10.9.2 Place the detector end of the column through the septum of a VOA vial and the injector end of the column in a collection vessel, i.e. beaker, flask, etc.

10.9.3 Attach to nitrogen gas tank a regulator capable of 40 psi. Attach to the regulator 1/8" tubing with a fitting to reducing the tubing to 1/16" stainless steel tubing. Place the free end of the stainless steel tubing through the septum of the VOA vial.

10.9.4 Fill the VOA vial with 25 mL of hexane. The column should be placed with the end in the solvent and the tubing should not be more than half way into the vial.

10.9.5 Turn on nitrogen gas to 40 psi. Allow all solvent to be forced into the column.

10.9.6 Repeat steps 10.8.4 and 10.8.5 with 25 mL acetone.

10.9.7 Repeat steps 10.8.4 and 10.8.5 with 80 mL methylene chloride.

10.9.8 Repeat steps 10.8.4 and 10.8.5 with 25 mL acetone.

10.9.9 Repeat steps 10.8.4 and 10.8.5 with 25 mL hexane.

10.9.10 Allow nitrogen to flow through column for 30 minutes.

10.9.11 Remove column from setup and cut 18" from the front end of the column.

10.9.12 Install the front end of the column in the gas chromatograph, leaving the detector end unconnected. Turn on the carrier gas and heat the column to its upper limit. Hold for 30 minutes.

10.9.13 Install the column in the detector end and heat at the column's upper limit overnight.

Table 1

Target Compounds

Compound	CAS #
Multi-Response Compounds	
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

Table 2

Calibration Levels

Level	PCB Concentration (µg/ml)
1	0.02
2	0.05
3	0.1
4	0.5
5	1.0

FIGURE 1

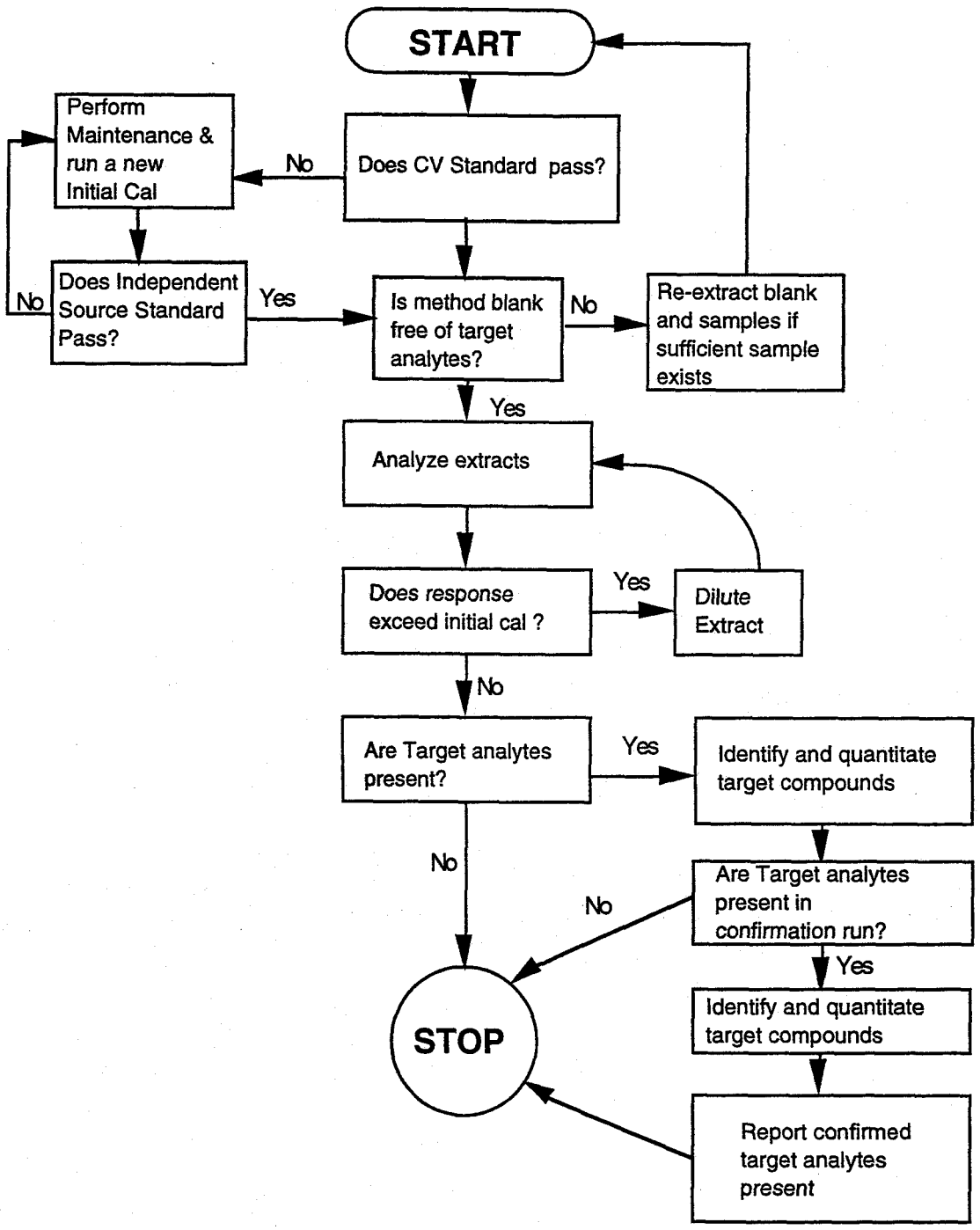
START
Does CV Standard pass?
No
Perform Maintenance & run a new Initial Cal
Does Independent Source Standard Pass?
No
Yes
Is method blank free of target analytes?
No
Re-extract blank and samples if sufficient sample exists
Yes
Analyze extracts
Does response exceed initial cal ?
Yes
Dilute Extract
No
Are Target analytes present?
Yes
Identify and quantitate target compounds
Are Target analytes present in confirmation run?
No
Yes
Identify and quantitate target compounds
No
STOP
Report confirmed target analytes present

FIGURE 2

Pesticide/PCB/Herbicide Logbook

Page:____

Instrument F

Loading Check:_____
Unloading Check:_____

Run	Vial	Sample	Dil	Meth/Cal	Comments

Rev 12/96

FIGURE 3

Transfer Date: 11/21/97

Initials: MR

EXTRACT TRANSFER SHEET

Organochlorine PCB

Sample	Analysis	Extraction Date	Matrix	Initial Volume (mL)	Initial Weight (g)	Final Volume (mL)	% Solids	Extraction Comments
39059-1	615	11/20/97	Aqueous	1060		10.0		
38209-1	T8151W	11/20/97	Sludge	200		10.0		

FIGURE 4

EXTRACTABLES DEVIATION REPORT

AEL#:_____ Client: _____ Matrix:_____

Analysis

ABN PEST PCB HERB TPH

Reason for investigation (list recoveries if possible):

Instrumentation Lab
Investigator:

Date:

Comments:

Extraction Lab
Investigator:

Date:

Comments:

Corrective Action
Investigator:

Date:

Comments:

Rev2, 6/93

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Separatory Funnel Liquid/Liquid Extraction for the Determination of Semi-Volatile Compounds in Aqueous Samples

COPY

Approved By:

QA Officer: Melissa A Sulli Date: 11/1/99

Laboratory Director: Steph L. Knollman Date: 11/1/99

Revision History

Revision	Description of Change	Approval	Date

1. Title

Separatory funnel liquid/liquid extraction for the determination of semi-volatile compounds in aqueous samples.

EPA References: Federal Register, Method 625
SW-846, Method 3510C

2. Scope and Application

This method describes the isolation and concentration of semi-volatiles from aqueous samples in preparation for analysis by GC/MS. The parameters listed in Table 1 can be measured by this method.

3. Safety

The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard. Exposure should be minimized by wearing solvent-resistant gloves, a lab coat, and safety goggles when samples or standards are handled, and by working in a fume hood. **The following compounds included in this method are classified as known or suspected human carcinogens: benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, methylene chloride and N-nitrosodimethylamine.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

4. Method Summary

Approximately 1 Liter of an aqueous sample is serially extracted with methylene chloride at a pH greater than 11 and again at a pH less than 2 using a separatory funnel extraction. The methylene chloride extract is then dried, concentrated to a volume of 1.0 ml, and analyzed by GC/MS.

5. Definitions

5.1 µl= microliter
5.2 ml= milliliter
5.3 L= liter
5.4 GC/MS= gas chromatograph/mass spectrometer
5.5 BN=Base Neutral
5.6 PAH= Polynucleararomatic Hydrocarbon
5.7 °C = Degrees Celsius
5.8 $MeCl_2$ = Methylene Chloride
5.9 ABN = Acid/Base Neutral
5.10 QC = Quality Control
5.11 CAS = Chemical Abstact Service
5.12 PSI = Pounds per square inch
5.13 NaOH = Sodium Hydroxide
5.14 N_2 = Nitrogen gas
5.15 AEL = Analytics Environmental Laboratory, Inc.

6. Apparatus and Materials

6.1 Automatic Shaker Table-Glas-Col 3-D Shaker
6.2 Separatory funnel - 2 liter glass, with Teflon stopcock and glass or Teflon cap.
6.3 Erlenmeyer flasks - 500ml glass.
6.4 Long-stem glass funnel with glass wool plug.
6.5 TurboVap II Concentration Workstation - Zymark Part Number 46368 or equivalent.
6.6 TurboVap Tubes - 1.0 ml stem.

6.7 Vials - 2ml amber glass autosampler with Teflon lined crimp caps.
6.8 pH indicator paper - Wide range, capable of measuring 0-14 pH units.
6.9 Graduated cylinder - 1 liter.
6.10 Graduated cylinder - 100ml.
6.11 Syringes - 1ml and 0.5ml.
6.12 Ring stands.
6.13 Class A volumetric glassware as required for final volume determination - rinsed 3 times with methylene chloride before and after use.
6.14 ABN sample prep log.
6.15 Disposable Pasteur pipettes, 9 inch glass.
6.16 Nitrogen blow down apparatus.
6.17 125ml glass Separatory funnel, with Teflon stopcock and glass or Teflon cap.
6.18 Purple labeling tape
6.19 Wax pencil

7. Standards and Reagents

7.1 Methylene chloride - Pesticide quality or equivalent.
7.2 Reagent water - Distilled then deionized with a Sybron Barnstead 3-canister filtering system, or equivalent.
7.3 10N NaOH - Purchased as a solution.
7.4 1:1 Sulfuric Acid - Prepared by slowly adding concentrated acid to an equal volume of reagent water.
7.5 Sodium sulfate - Granular, anhydrous; suitable for environmental residue analysis. Purify by heating at 200°C for a minimum of four hours in a shallow glass baking dish and store in a closed glass container.
7.6 ABN Surrogate Solution - Refer to QA-S004.
ABN Spike Solutions - Refer to QA-S004.
7.7 Nitrogen gas-grade 5, from a liquid nitrogen tank
7.8 Methanol - purge and trap grade or equivalent for syringe rinsing.

8. Procedure

8.1 Sample Preparation Notebook
All information pertinent to semi-volatile sample preparation is recorded in a bound paginated laboratory notebook. Each entry should contain a general heading with the extraction date and the initials of the analyst(s) that performed the extraction. A column format should then include the following headings: sample number, initial volume (mls), surrogate standard number and amount added, spike standard number and amount added, volume extraction solvent recovered (BN and Acid fractions), final extract volume (after cookdown), and the date the concentration was completed as well as the initials of the individual(s) performing the concentration. Observations about specific samples will also be recorded in the notebook. For example, it is important to note when emulsions or precipitates form.
8.2 Determine Necessary Quality Control
8.2.1 One method blank must be performed for each analytical batch (twenty samples, or one eight hour shift.) The method blank consists of one liter of reagent water with **500 µl** of surrogate that is extracted in the same manner as the samples. The blank is labeled as follows: BMMDDYAW, where;

B indicates a blank
MM is the two number designation for the month
DD is a two number designation for the day
Y is a single number designation for the year

A designates ABN
W designates an aqueous matrix

8.2.2 One laboratory control sample must be performed for each analytical batch (twenty samples, or one eight hour shift.) Laboratory control samples are prepared by spiking 1 liter of reagent water with 1.0 ml of spiking solution and **500 µl** of surrogate. The laboratory control sample is labeled as follows: LMMDDYAW where;

L designates laboratory control sample
MM is the two number designation for the month
DD is a two number designation for the day
Y is a single number designation for the year
A designates ABN
W designates an aqueous matrix.

8.2.3 One laboratory control sample duplicate must be performed for every twenty samples or within a one month period. Fill out the QC Summary (Figure 1) to determine if it is necessary to perform a laboratory control sample duplicate. Laboratory control sample duplicates are prepared by spiking 1 liter of reagent water with 1.0 ml of spiking solution and **500 µl** of surrogate. The laboratory control duplicate sample is labeled as follows: LDMMDDYAW where;

L designates laboratory control sample
MM is the two number designation for the month
DD is a two number designation for the day
Y is a single number designation for the year
A designates ABN
W designates an aqueous matrix.

8.2.3 A matrix spike and matrix spike duplictae of an actual sample will be performed on an as-assigned basis when sufficient sample is provided. Matrix spikes will be labeled with the sample identifier followed by MS or MSD. Matrix spikes and matrix spike duplicates are prepared by spiking 1 liter of sample with 1.0 ml of spiking solution and **500 µl** surrogate.

8.3 Glassware Preparation

8.3.1 For each sample, set up a ring stand with a ring, a 2-liter separatory funnel, and a 500ml Erlenmeyer flask. Pre-rinse all of the glassware, including the caps, with methylene chloride.

8.3.2 Place each separatory funnel in a ring and label it, using a wax pencil, with the sample number it will contain. Label (with purple tape) each of the 500ml flasks with the sample number, ABN-H_2O, and the blank # that the sample batch is associated with. It is imperative that the labels be clear. The labels will be transferred throughout the extraction process.

8.3.3 Place the sample container on the ring stand supporting the appropriate funnel. The funnel label and sample are to be checked by another individual for agreement. Note that the samples were checked in the sample preparation notebook by putting a check over the initials of the individual performing the check.

8.3.4 Using a wax pencil, mark the meniscus of the sample on the outside of the Sample container. Pour the sample into the separatory funnel being careful not to transfer any sediment.

8.3.5 Using a 1 ml syringe, designated for ABN surrogate, add **500µl** of the ABN surrogate to every sample. Mark each separatory funnel with the wax pencil after adding surrogate.

8.3.6 If a laboratory control sample and laboratory control sample duplicate are being extracted, use a 1 ml syringe to add 1.0 ml of spiking solution to a liter of reagent water. **The syringe used for spiking is not a dedicated syringe so it must be rinsed 3**

times before and after use with Purge and Trap grade methanol. Mark each separatory funnel with the wax pencil after adding spike.

8.3.7 If a matrix spike and matrix spike duplicate are being extracted, use the 1 ml syringe in 8.3.6 above to add 1.0ml of spiking solution to the samples which are being spiked. Mark each separatory funnel with the wax pencil after adding spike.

8.4 Extraction of the Base/Neutral Fraction

8.4.1 Adjust the pH of the sample in the separatory funnel to >11 with 10N NaOH. Mix the sample in the separatory funnel after adding NaOH. Use the wide range pH paper to check the pH. Normally, two- 2ml dispo-pipette volumes are required to achieve the correct pH. **Decomposition of some analytes has been demonstrated under basic extraction conditions. This decomposition increases with increasing pH. An attempt should be made to have the pH of the extract as close to 11 as possible. The basic extraction should be performed as quickly as possible to minimize decomposition.** If the sample requires more than 2 volumes of NaOH, note the approximate amount in the sample preparation notebook.

8.4.2 Measure 60ml methylene chloride in the 100ml graduated cylinder. Pour this aliquot of methylene chloride into the sample container, rinse. Add the methylene chloride to the separatory funnel.

8.4.3 Seal the separatory funnel and place it in the automatic shaker holder. Invert the shaker holder and open the stopcock. Set the motor speed to 66 and program the timer for 2 minutes. Turn on the shaker table. **Methylene chloride creates excessive pressure rapidly and should be initially vented immediately after the funnel has been sealed and shaken once.**

8.4.4 Allow the methylene chloride to completely separate from the aqueous phase (about 10minutes). If an emulsion occurs, mechanical techniques must be employed to try to complete phase separation. Gentle rocking or stirring may help. A 125ml separatory funnel may be useful in dispersing emulsions. Slowly drain the emulsion from the 2 liter separatory funnel into a pre-rinsed 125ml separatory funnel. This action alone may be sufficient to disperse the emulsion. If it does not, return the emulsion to the separatory funnel and employ a different technique. Be sure to rinse the 125ml separatory funnel with methylene chloride to remove any residuals left behind. The emulsion may be filtered into the appropriate flask through sodium sulfate, **rinsed with methylene chloride**, in a long stem funnel plugged with a small amount of glass wool. Slowly drain the methylene chloride extract into the corresponding numbered flask by carefully opening the stopcock. Close the stopcock when all of the methylene chloride has been collected in the flask. Record all information on emulsions in the ABN preparation logbook.

8.4.5 Add a second 60ml aliquot of methylene chloride to the sample container. Repeat steps 8.4.3 and 8.4.4, combining the extracts in the flask.

8.4.6 Repeat steps 8.4.3 and 8.4.4 with a third aliquot of methylene chloride. Again, combining the extracts in the flask. The Base Neutral extraction is now complete.

8.4.7 Note the volume of extraction solvent recovered. **The total volume recovered must be at least 140 mL or additional extractions must be performed.**

8.5 Extraction of the Acid Fraction

8.5.1 Adjust the pH of the sample in the separatory funnel to <2 with 1:1 sulfuric acid. Two-2ml dispo-pipette volumes should be sufficient. If more than 2 volumes of acid are required, estimate the amount used and record it in the sample preparation notebook. Check the pH with wide-range pH indicator paper.

8.5.2 Measure a 60 ml aliquot of methylene chloride in a 100 ml graduated cylinder and add it to the separatory funnel.

8.5.3 Repeat section 8.4.3

8.5.4 Allow the methylene chloride phase to completely separate from the aqueous phase. If emulsions form, follow procedures described in 8.4.4 .

8.5.5 Collect the methylene chloride extract in the 500ml flask.

8.5.6 Repeat steps 8.5.2 through 8.5.5 two more times with fresh portions of methylene chloride. Combine all solvent extracts from BN and Acid fractions in the 500ml flask.

8.5.7 **Final volume of collected solvent extract should be at least 285ml. If this is not the case, additional extractions must be performed.** Record the final volume in the ABN sample preparation logbook.

8.5.8 Determine the initial volume of the sample. If necessary bring the volume in the separatory funnel up to the original volume line with tap water. Pour the entire sample into a 1000ml graduated cylinder. Record the volume to the nearest 5mls in the sample preparation log notebook. Dispose of spent sample in the liquid waste stream.

8.6 Extract Drying and Concentration

8.6.1 If the extract is not concentrated immediately, store in the refrigerator.

8.6.2 Plug a long-stem glass funnel with a small wad of glass wool and fill funnel about 3/4 full with baked sodium sulfate. Rinse the sodium sulfate thoroughly with methylene chloride. Collect methylene chloride in a container designated as 'methylene chloride waste' and dispose of properly.

8.6.3 The TurboVap temperature should be set at 35°C. The select button should be set on the sensor. This will allow the TurboVap to concentrate the sample extract down to <1.0 ml without completely evaporating. The nitrogen pressure should be set to 10 psi.

8.6.4 ABN water extractions yield twice the volume of all other extractables, the extract will need to be concentrated in two stages. Label the TurboVap tube with the sample identifier with a wax pencil and fill the tube to the fill line (200ml) by filtering the sample through the funnel prepared in 8.6.2 above. Remove the sodium sulfate funnel from the TurboVap tube and place it in the appropriate sample flask. Store the funnel and the flask in the refrigerator. Once the first half of the extract has been reduced to 1-5 ml place the funnel back in the TurboVap tube and pour the remaining extract into the tube. After the sample has passed through the sodium sulfate, rinse the flask three times with methylene chloride and pour over the sodium sulfate. Transfer the purple tape label from the flask to the TurboVap tube. Pour about 20 ml of methylene chloride over the sodium sulfate to rinse out any of the remaining extract. Concentrate the sample to 1.0 ml.

8.6.5 Transfer concentrated sample extract with a clean 9 inch pasteur pipette into a 1.0 ml volumetric flask. With a clean pasteur pipette add a small amount of methylene chloride to the TurboVap tube to rinse sides. Using the original pipette, rinse the walls of the lower portion of the Turbo tube at least three times then transfer the methylene chloride to the volumetric flask. Repeat as many times as needed to achieve 1.0 ml volume in the flask. **(It is preferable to underestimate and do repeat rinsings rather than to overestimate and have some methylene chloride remaining).** When a final volume of 1.0 ml is achieved, transfer the extract with the same pipette into a 2 ml vial labeled with the AEL #, ABNH_2O and the date. **If the final volume is greater than 1.0 ml, record the final volume on the vial.** Cap the vial, attach the tape label to the vial cap and place the extract in the freezer designated for the ABN extracts.

8.6.6 If the TurboVap automatically shuts off before the extract has been reduced to <1.0 ml (this can happen repeatedly) the analyst has several options:

1) Reset the TurvoVap cell holding that sample, until the sample is cooked down to <1.0 ml. Then go back to 8.6.5 for vialing. This option should be considered in most cases unless the sample is extremely oily or contains particulates.
2) Transfer the sample from the TurboVap tube to an appropriately sized collection tube. Set up nitrogen blow-down apparatus and condense sample to <1.0 ml or until sample volume reduction stops. If sample goes to <1.0 ml, return to section 8.6.5 for vialing. Otherwise, move on to option #3.
3) If sample volume can't be reduced to <1.0 ml using either of these techniques, the sample must be brought to a convenient volume. Make an estimate of the volume of the remaining sample and choose a Class A volumetric of sufficient size to contain the extract plus any methylene chloride used to rinse the TurboVap tube. Using a 1.0 ml

syringe, place **exactly** 1.0 ml of the extract in a clear 2.0 ml vial. Store the remainder of the extract in an appropriate container.

8.6.7 Extracts are stored in the freezer designated for ABN extracts and must be analyzed within 40 days.

8.7 Transfer Sheets and Log Book

8.7.1 A transfer sheet (Figure 2) is generated each day that samples are completed. Deliver a copy to the GC/MS analyst. In addition make a copy to be included in the appropriate client folder. The original is filed in the laboratory output folder. Fill out "Final Volume" and "Date Transfered" columns in the sample prep log book. Make note of any unusually large final volumes.

9. Quality Control

9.1 Per sample

9.1.1 Surrogates are added to every sample matrix spike, laboratory control sample, and blank prior to extraction to test the efficiency of the extraction procedure. Percent recoveries are calculated by the GC/MS analyst. If surrogate recoveries are outside of the pre-determined acceptable ranges, an Extractables Deviation Report explaining the cause is completed (Figure 3). Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. In this case, the GC/MS analyst will transfer the deviation report to the extraction laboratory. Unusual circumstances during the extraction should have been noted in the sample preparation log notebook. Once the deviation report has been completed, it should be returned to the GC/MS analyst. A copy is filed in the client's folder and the original is filed in the Extractable Deviation Notebook in the file room.

9.2 Per sample batch (20 samples or per eight hour shift)

9.2.1 A method blank must be performed with every sample batch (20 samples, or per eight hour shift). A blank consists of 1 Liter of reagent water, that is carried throughout the extraction procedure. Blanks are analyzed prior to the appropriate sample batch to monitor interferences or contaminants that may have occurred during the extraction process. If a blank is found to contain more than the reporting limit of any compound, the sample batch associated with it must be re-extracted. If insufficient sample exists, the contaminant will be qualified with a "B" flag.

9.3 Per 20 Samples

9.3.1 One laboratory control sample and laboratory control sample duplicate is performed per batch of 20 samples or within a one month period. The laboratory control samples must be subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy and precision. The percent recoveries and relative percent difference of the compounds will be calculated by the GC/MS analyst. If the recoveries or relative percent differences are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. Extraction personnel may be asked to determine the cause of the deviation. In this case the GC/MS analyst will transfer the Extractables Deviation Report to the extraction laboratory. Unusual circumstances during the extraction should be recorded in the preparation log. Once the deviation report has been completed by the extraction laboratory, it should be returned to the analyst. A copy is filed in the QC Notebook with the chormatogram and the original is filed in the Extractables Deviation Notebook in the file room.

9.3.2 Matrix spikes are performed when sufficient sample has been provided to analyze both the sample and a spike of the sample. The matrix spike is subjected to the same extraction procedure as associated client samples. The spike recoveries will be calculated by the GC/MS analyst. If the spike recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. An Extractables Deviation Report (Figure 3) will be completed by the analyst. Extraction personnel may be asked to determine the cause of the deviation. In this case the GC/MS analyst will transfer

the Extractables Deviation Report to the extraction laboratory. Unusual circumstances during the extraction should be recorded in the preparation log. Once the deviation report has been completed by the extraction laboratory, it should be returned to the analyst. A copy is filed in the QC Notebook with the chromatogram and the original is filed in the Extractables Deviation Notebook in the file room.

9.4 All surrogate and spike solutions are tested prior to use (QA-M015).

9.5 The preparation notebook is reviewed by the laboratory director or designate on a biweekly basis.

Table 1

Target Compounds

Compound	CAS #
Pyridine	110-86-1
N-Nitrosodimethylamine	62-75-9
Aniline	62-53-3
Phenol	108-95-2
2-Chlorophenol	95-57-8
Bis(2-Chloroethyl) Ether	111-44-4
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1
Benzyl alcohol	100-51-6
2-Methylphenol (o-Cresol)	95-48-7
Bis (2-chloroisopropyl) ether	108-60-1
3-Methylphenol	108-39-4
4-Methylphenol (m,p-Cresols)	106-44-5
Hexachloroethane	67-72-1
N-Nitroso-di-n-propylamine	621-64-7
Nitrobenzene	98-95-3
Isophorone	78-59-1
2-Nitrophenol	88-75-5
2,4-Dimethylphenol	105-67-9
Bis (2-chloroethoxy) methane	111-91-1
2,4 Dichlorophenol	120-83-2
Benzoic Acid	65-85-0
1,2,4-Trichlorobenzene	120-82-1
Naphthalene	91-20-3
2,6-Dichlorophenol	87-65-0
4 -Chloroaniline	106-47-8
Hexachlorobutadiene	87-68-3
4-Chloro-3-methylphenol	59-50-7
2-Methylnaphthalene	91-57-6
Hexachlorocylopentadiene	77-47-4
2,4,6-Trichlorophenol	88-06-2
2,4,5-Trichlorophenol	95-95-4
2-Chloronaphthalene	91-58-7
2-Nitroaniline	88-74-4
2,6-Dinitrotoluene	606-20-2
Dimethyl phthalate	131-11-3
Acenaphthylene	208-96-8

Table 1 (continued)

Target compounds

3-Nitroaniline	99-09-2
Acenaphthene	83-32-9
2,4-Dinitrophenol	51-28-5
Dibenzofuran	132-64-9
4 Nitrophenol	100-02-7
2,4-Dinitrotoluene	121-14-2
2,3,4,6-Tetrachlorophenol	58-90-2
Fluorene	86-73-7
Diethyl phthalate	84-66-2
4-Chlorophenyl phenyl ether	7005-72-3
4-Nitroaniline	100-01-6
4,6-Dinitro-2-methylphenol	534-52-1
N-Nitrosodiphenylamine	86-30-6
Azobenzene	103-33-3
4-Bromophenyl phenyl ether	101-55-3
Hexachlorobenzene	118-74-1
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Anthracene	120-12-7
Carbazole	86-74-8
Di-n-butyl phthalate	84-74-2
Fluoranthene	206-44-0
Benzidine	92-87-5
Pyrene	129-00-0
Butyl benzyl phthalate	85-68-7
Benz (a) anthracene	56-55-3
Chrysene	218-01-9
3-3'-Dichlorobenzidine	91-94-1
Bis (2-ethylhexyl) phthalate	117-81-7
Di-n-octyl phthalate	117-84-0
Benzo (b) fluoranthene	205-99-2
Benzo (k) fluoranthene	207-08-9
Benzo [a] pyrene	50-32-8
Indeno [1,2,3-cd] pyrene	193-39-5
Dibenz [a,h] anthracene	53-70-3
Benzo [ghi] perylene	191-24-2

FIGURE 1

QC SUMMARY WATER SAMPLES

LCS_____
LCS DUP _____
MS _____
MSD_____

Procedure: _____ Batch ID# _____

	SAMPLE #	DATE	BLANK	LCS	LCS DUP	MS	MSD
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

FIGURE 2

Transfer Date: 12/10/97

EXTRACT TRANSFER SHEET

Initials: MR

Semi-Volatile Compounds:

Sample	Analysis	Extraction Date	Matrix	Initial Volume (mL)	Initial Weight (g)	Final Volume (mL)	% Solids	Extraction Comments
39130-1	PP625	12/10/97	Aqueous	1030		1.0		

FIGURE 3

EXTRACTABLES DEVIATION REPORT

AEL#:_____ Client:_____ Matrix:_____

Analysis

ABN PEST PCB HERB TPH

Reason for investigation (list recoveries if possible):

Instrumentation Lab
Investigator:

Date:

Comments:

Extraction Lab
Investigator:

Date:

Comments:

Comment and/or Corrective Action
Investigator:

Date:

Comments:

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COPY

Laboratory Method for the Determination of Base/Neutral and Acid Compounds in All Matrices

Approved By:

QA Officer: _____ Date: 11/7/01

Laboratory Director: _____ Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Laboratory Method for the Determination of Base/Neutral and Acid Compounds in All Matrices.

EPA References: EPA 600 Series-40 CFR, PART 136, Revised as of July 1, 1995 Appendix A to Part 136-Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. Method 625 –Semi-Volatile Organic Compounds

Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Methods 8270C Revision, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods-Method 8000B, Revision, December 1996

Comprehensive Quality Assurance Plan for ANALYTICS Environmental Laboratory, LLC. January 2001

2. Scope and Application

This method is used for the determination of semi-volatile compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The parameters listed in Table 1 can be measured by this method. This method is restricted to use by or under the supervision of analysts experienced in the use of GC/MS, and in the interpretation of mass spectra.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevation in baselines. Method blanks must be routinely analyzed to show the absences of these interferences.

3.2 Interferences co-extracted from the sample will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample extract may be necessary.

3.3 Phthalates esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because of phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time id consistent quality control is not practiced.

3.4 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of phenolic analytes. This problem is especially pronounced with glassware that may be difficult to rinse. These items should be hand rinsed to avoid this problem.

3.5 Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. In order to reduce carryover, the sample syringe must be rinsed with solvent between sample injections and whenever an unusually concentrated sample is analyzed, it should be followed by a solvent blank to check for contamination.

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard.

4.2 Exposure should be minimized by wearing solvent resistant gloves, safety goggles and a laboratory coat when samples or standards are handled, and by working in a fume hood.

4.3 **The following compounds included in this method are classified as known or suspected human carcinogens: benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, methylene chloride and N-nitrosodimethylamine.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

5. Method Summary

Approximately 1 liter of an aqueous sample is serially extracted with methylene chloride at a pH greater than 11 and again at a pH less than 2 using a separatory funnel extraction. For solid matrices, 30g of the sample is extracted by sonication with methylene chloride. The methylene chloride extract is then dried, concentrated to a volume of 1.0ml, and analyzed by GC/MS. A combination of retention time and relative abundance of characteristic masses (m/z) is used to make qualitative identification of target compounds. Quantitative analysis is performed using internal standard techniques. For a method summary refer to Figure 1.

6. Sample Collection, Handling and Preservation

6.1 All samples should be collected in glass jar with a screw top Teflon lid.

6.2 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling instructions.

6.3 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and log-in procedures.

6.4 Solid samples must be extracted within 14 days of collection.

6.5 Aqueous samples must be extracted within 7 days of collection

6.7 Sample extracts must be analyzed within 40 days of extraction.

6.8 Extracts must be stored in a freezer at –10°C to –20°C protected from

7. Definitions

7.1 ABN= Acid and Base Neutral

7.2 A/D= analog to digital

7.3 ALS= automated liquid sampler

7.4 AMU= atomic mass unit

7.5 BN= base/neutral

7.6 °C= degrees Celcius

7.7 DFTPP= decafluorotriphenyl phosphine

7.8 EPA= Environmental Protection Agency

7.9 eV= electron volt

7.10 g= gram

7.11 GC/MS= gas chromatograph/mass spectrometer

7.12 PAH= polynuclear aromatic hydrocarbons

7.13 psi= pound per square inch

7.14 L= liter

7.15 m= meter

7.16 ml= milliliter

7.17 mg= milligram

7.18 m/z= mass to charge ratio

7.19 ng= nanogram

7.20 QC= quality control

7.21 RSD= Relative Standard Deviation

7.22 SIM= Selected Ion Monitoring

7.23 SOP= standard operating procedure

7.24 μg= microgram

7.25 μl= microliter

8. Apparatus and Materials

8.1 GC/MS System Components

8.1.1 Gas Chromatograph and components

Gas Chromatograph	Hewlett Packard 5890E Series II *Plus* or equivalent
GC Column	H.P.-5MS Hewlett Packard cat. #190915-433 or Rtx-5MS Restek cat. #12623, Crossbonded® 95% dimethyl - 5% diphenyl polysiloxane 30 meter, 0.25mm ID, 0.25μm film thickness or equivalent
Septa	Thermogreen™ LB-2, 11mm Supelco catalog#2-0654 or equivalent
Splitless Sleeves	4mm borosilicate Restek catalog#20773 or equivalent (packed with 2cm deactivated glass wool Restek catalog#20790)
O-rings	viton (fluorocarbon) Restek catalog#20377 or equivalent
Inlet Seals	Gold-plated, 1.2mm Restek catalog#21306 or equivalent
Ferrules	injector end, HP P/N 5181-3323, 90%Vespel, 10% Graphite dectector end, HP P/N 5062-3508 , 85%Vespel, 15%Graphite

8.1.2 Auto-Sampler

Auto-sampler	Hewlett Packard ALS 7673A with Controller or equivalent
Auto-sampler syringes	10μl, 23s gauge fixed needle Restek catalog#21203 or equivalent

8.1.3 Mass Spectrometer Hewlett Packard Model 5972A, or equivalent, capable of scanning from 35-500 AMV every one second or less using 70eV. Replacement parts and maintenance thereof is found in the manual entitled *HP 5972A MSD Hardware Manual* located in the instrument lab.

8.2 DOS Chemstation Data System Components

Data acquisition methods are accessed by opening the "ENVTop #1" Icon in the Program Manager.

8.2.1 Scan Aquisition

GC methods for scan aquisition use Hewlett Packard Environmental Chemstation Software. Scan is an MS aquisition mode that scans a selected mass range linearly and repeatedly over the course of the analysis. The scan acquistion method used in the analysis of tunemix is TUNEMIX.M. The scan acquisition method used in the analysis of ABNs is the same as the method used for data analysis and is named SMMDDY.M. A summary of the instrument parameters used in methods TUNEMIX.M and SMMDDY.M are listed in Tables 2 and 3 respectively.

8.2.2 SIM Aquisition

GC methods for SIM aquisition use Hewlett Packard MS Chem Station Software. SIM is an MS aquisition mode that monitors specified masses repeatedly over the course of an analysis. SIM aquisition is only used when low level detection is required. The SIM aquisition method used in the analysis of PAHs is the same as the method used for data aquisition and is named SIMMMDDY.M. A summary of the instrument parameters used in SIMMMDDY.M are listed in Table 4.

8.2.3 Enviroquant Software

The enviroquant software package is used for data analysis and reporting. This houses the methods necessary for the quantitation of data including compound lists, retention times and calibration levels. Data quantitation methods are accessed by opening the "ENVDA #1" Icon in the Program Manager. The quantitaion reports are generated either interactively or automatically using enviroquant. There are two active method files used in the analysis of ABNs. They are labeled according to the day of the initial calibration used in the method.
For example the active quantitation database for all semi-volatiles is SMMDDY, the PAH SIM database is SIMMMDDY, where;

the first S or SIM would indicate the compound list,
MM is a two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the year

8.3 Instrument run logbook

8.4 Sample preparation logbook

8.5 Maintenance logbook

9. Standards and Reagents

9.1 Methylene Chloride - Pesticide quality or equivalent

9.2 Toluene- Pesticide quality or equivalent

9.3 Refer to Standard Operating Procedure QA-S004 for a list of standards.

9.4 Internal Standards Mixture - 4000 μg/l in methylene chloride, Ultra cat. #US-108N-4 or equivalent.

10. Procedure

Refer to the following Standard Operating Procedures for sample preparation.

QA-S001: Separatory funnel liquid/liquid extraction for the determination of semi-volatiles in aqueous samples.

QA-S007: Sonication extraction for the determination of semi-volatiles in solid samples.

QA-S003: Waste dilution method for the determination of semi-volatiles in non aqueous wastes.

10.1 Instrument Run Logbook

A bound paginated laboratory notebook is assigned to each instrument for use as a sample run log. Each day's runs are recorded on a separate page, with the following information noted at the top: the date, the analyst's initials, the multiplier voltage, and the tune file name. Refer to Figure 2 for an example of a sample run log page. In addition, the following information must be included in a column format. For each run include: auto-sampler position (vial), method file, sample number and/or standard information, dilution, data file name, and comments relevant to the run. Each GC/MS system is identified with a unique letter. Data files are assigned by using that letter, followed by the next consecutive number. The 5972A letter designation is "D" therefore, the first data file for instrument "D" would be D0001, with subsequent files following in numerical order. Also on the run log is a "loading check" and "unloading check". Prior to loading the vials, check to ensure that the tape label agrees with the label on the vial. When unloading the samples, check to ensure that the order in the auto-sampler agrees with that in the run log. Record initials in the loading check boxes. Also, It is necessary to note the window batch identifier at the top of the page.
(ex. W- Inst. letter ID -MMDDYY-1(window #)-analyst's initials)

10.2 DFTPP Tuning

Before any sample analysis can begin, the instrument must be hardware tuned so that DFTPP will meet the criteria listed in Table 5.

10.2.1 Prepare a 50ng/μl DFTPP tuning mix standard (which also contains 50 ng/μl each of 4,4-DDT, pentachlorophenol, and benzidine) as described in QA-S004. Using the scan aquisition method tunemix.M, inject 1μl of the working standard.

10.2.2 Obtain a mass spectrum of the DFTPP using the enviroquant software. This can be a single scan or can include scans added under the peak or scans subtracted outside of the peak. **If subtraction is performed, it must be**

done using a scan no more than 20 scans prior to the elution of DFTPP. Evaluate the spectrum against the criteria listed in Table 5 by using the DFTPP pull-downs in the "Tuner" menu. The analyst must re-inject and/or take corrective action until a passing DFTPP spectrum is achieved. The success of the tune depends on the current condition of the ion source and the GC system. Refer to *HP 5972A MSD Hardware Manual* for troubleshooting techniques.

10.2.3 Print a hard copy of the passing tune using the "Evaluate DFTPP to Printer" pull-down in the "Tuner" menu. Notate on the hard copy report and in the comments section of the run log the scan number/s of the passing spectrum and any necessary manipulations (i.e. additions or subtractions of scans). Also initial and date the tune printout.

10.2.4 In order to assess GC column performance and injection port inertness, evaluate DDT breakdown and the tailing factor of benzidine and pentachlorophenol. The tailing factor for benzidine must be less than 3 and the tailing factor for PCP must be less than 5. (40CFR). This calculation is performed using the Enviroquant software under the dropdown menu "Quant".

10.2.5 File a hard copy of the passing tune in the Semi-Volatiles QC notebook.

10.2.6 **A tune mix standard must be analyzed and pass every 12 hours.**

10.3 Initial Calibration – A Five Point Calibration is required.

10.3.1 Prepare the ABN five point standards as described in SOP QA-S004. A separate 5 point is used for the full semi-volatile scan versus the PAH by SIM.

10.3.2 Analyze 1μl injections of the 5 standards.

10.3.3 Quantitate each standard using the SMMDDY or SIMMMDDY method in the enviroquant software. Response factors are calculated using Equation 1.

Equation 1 $$RF = \frac{AsCis}{AisCs}$$

where:
As =Area or height of the characteristic m/z for the parameter to be measured.
Ais=Area of the characteristic m/z for the internal standard.
Cs =Concentration of the parameter to be measured (μg/ml).
Cis=Concentration of the internal standard (μg/ml).

10.3.4 Update each of the calibration levels in SMMDDY or SIMMMDDY. Do this by loading each datafile and using the "InitCal" menu pull-down "Update Levels". It is important to save the method after updating. When updating the mid-point standard be sure to check the boxes for update retention times, and update reference spectra.

10.3.5 Print a copy of the 5-point using the "InitCal" menu pull-down "Response Factors to Printer". This will produce a hardcopy including average response factors and percent relative standard deviation. The percent relative standard deviation should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) must be less than 30%. Furthermore, the average response factors for the system performance check compounds (SPCCs), must be greater than 0.05. The CCC and SPCC's are listed in Table 6. If the %RSD of any compound is 15% or less the average response factor can be used for quantitation. If the %RSD is greater than 15%, a calibration curve may be used to quantitate. Alternatively, if the mean of the RSD values for all analytes in the calibration is less than or equal to 15%, the average response factor can be used for all analytes. If neither of these conditions are met, a new calibration curve must be analyzed. If a linear calibration is used, the correlation coefficient (r) must be 0.995 or greater to meet acceptance criteria. For a quadratic fit, the coefficient of determination (r^2) be 0.995 or greater to meet acceptance criteria and **six** calibration levels are required. **It is never acceptable to force a calibration curve through the origin nor to include the origin (0,0) as a sixth calibration point.**

%RSD is calculated using Equation 2.

Equation 2 $$\% \text{ RSD} = \frac{\text{SD}}{\text{RF}} \times 100$$

where:
RSD = Relative Standard Deviation
SD = Standard Deviation of the RFs for a compound from the initial calibration
RF = mean of 5 initial Response Factors for a compound from the initial calibration

10.3.6 If the calibration is acceptable, file a copy of each standard, along with the calibration report in the Semi-Volatiles QC notebook which has been initialed and dated. **If manual integration was performed the report must be reprinted before filing. A hardcopy of the manual integration must be printed and filed with the curve. Refer to the manual integration SOP for proper protocol.**

10.3.7 If the calibration is not acceptable, corrective action must be taken. Determine the cause of the problem, correct it, and re-analyze the five point. Corrective action is documented in the instrument maintenance log-book. The log entry should contain the date, the initials of those involved with the maintenance, a description of the problem, and what the corrective action was.

10.3.8 Before a multipoint calibration is accepted, a second source standard at the midpoint concentration is analyzed to verify the calibration. The analytes of interest must fall within ± 20% of the expected concentrations for the calibration to be valid for that analyte. Some of the more problematic compounds may be accepted with wider limits upon review by the QC Officer or the Laboratory Director.. Any out of control event shall be documented on a QC Deviation Form and given to the QC Officer for

review and filing. Prepare the calibration check standard as described in SOP QA-S004.

10.4 Calibration Verification

Once an acceptable five point calibration has been generated, it must be verified every twelve hours with a mid point calibration standard.

10.4.1 Prepare the calibration verification standard as described in SOP QA-S004.

10.4.2 Analyze 1 μl injections of the standard.

10.4.3 Quantitate the standard using the SMMDDY.M or the SIMMMDDY.M method in the enviroquant software.

10.4.4 Check the calibration to ensure that the system performance check compounds are met (Table 6). CCCs listed in Table 6 are also used to verify the calibration. The percent difference is calculated by the HP software using the following equations;

$$\%Difference = (Rf_V - Rf) / Rf * 100$$

Rf_V = Response factor of verification standard
Rf = Mean response factor from initial calibration

$$\%Drift = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theoretical Concentration}} * 100$$

Use percent difference when performing average Rf calibration; use percent drift when calibrating with a curve. If the percent difference or percent drift for each CCC is less than 20%, the initial calibration is assumed to be valid. If any criterion is not met for any one CCC or SPCC, the system must be evaluated, and corrective actions must be taken before sample analysis begins. It may be necessary to analyze a new 5-point calibration. If any compounds are set to a curve fit the measured concentration of the compound must be ±15% of expected concentration.

10.4.5 The internal standard responses and retention times in the calibration check standard or calibration verification standard must be evaluated after data acquisition. If the retention time for any internal standard changes by more than 0.5 minutes from that in the mid-point standard level of the current initial calibration, the system must be inspected for malfunctions and corrections must be made. The minimum internal standard area must be at least 50% to a maximum area of 200% from that in the mid-point standard level of the current initial calibration.

10.4.6 If the calibration verification is acceptable, file a copy of the standard quantitation report, along with the Continuing Calibration Report (which has been initialed and dated) in the Semi-Volatiles QC notebook. **If manual integration was performed the report must be reprinted before filing.**

A hardcopy of the manual integration must be printed and filed with the curve. Refer to the manual integration SOP for proper protocol

10.4.7 If the calibration verification is not acceptable, determine the source of the problem and run another calibration verification or a new five point. Possible sources of problems include a dirty liner, a dirty seal, or a contaminated column.

10.4.8 **A calibration verification standard must be analyzed and pass every 12 hours.**

10.5 Analyzing samples
Sample batches are sequenced by selecting the "Instrument D MS Top" icon in the program manager and using the "Edit Sample Log Table" pul-ldown under the "Sequence" menu. Sample batches are sequenced, saved and run using the activated dialog box in this window.

10.5.1 Before sample analysis can begin, a blank that has been through the extraction process (and has had 10 µL of internal standard added) must be analyzed. This is necessary to determine extraction efficiency via surrogate standards and also to determine if any interferences are present. Acceptable blanks will not have any contaminants which exceed the reporting limit. If a blank is unacceptable, the accompanying samples must be re-extracted. In addition, an QC Deviation Report (Figure 3) must be completed. If insufficient sample exists for re-extraction, contaminants found in both the blank and the sample must be flagged with a "B" on the hard copy report. The source of contamination should be determined and the necessary precautions to prevent future interferences will become a part of the appropriate Standard Operating Procedure(s).

10.5.2 Add 10µL of internal standard to each 1.0ml sample extract just prior to analysis. Inject 1µl of sample and run using the appropriate GC conditions described in Table 3 and Table 4. Record the sample runs in the instrument run log book as described in section 10.1. Include any dilutions and comments that are relevant to the run.

10.5.3 Quantitate the samples using the enviroquant software. Samples will be quantitated using one of the methods described in Section 10.3. When MS response is linear (%RSD <15%) and the average response factor is selected, calculate the concentration of each identified analyte in the sample using Equation 4. If the software does not correctly integrate any of the analytes, a manual integration is performed and a graphics copy is printed out along with a new datafile quant report.

Equation 4 $$\text{Concentration} = \frac{(As)(Is)(Vt)}{(Ais)(Rf)(Vo)(D)}$$

where:
As=Area or height of characteristic m/z for the parameter to be measured.
Ais=Area or height of the characteristic m/z for the internal standard.

D= (100 - per cent moisture/100)for solid samples to be reported on a dry weight basis. Use 1 for water samples or solid samples that are reported on a wet weight basis.
Is= Amount of Internal Standard added to each extract (μg/L).
Vo=Volume of water extracted(L), or weight extracted (kg).
Vt= Dilution.
Rf= Response factor of initial calibration

10.5.4 **If the RSD is not <15%, a calibration curve may be used.**

10.5.5 In calculating sample concentrations by the internal standard method, the regression equation is rearranged to solve for the concentration of the target analyte (C_S), as shown below.

$$C_S = \left[(A_S C_{IS}) / A_{IS} - b\right] / a$$

A_S = Area or height of the peak for the target analyte in the sample
A_{IS} = Area or height of the peak for the internal standard
C_S = Concentration of the target analyte in the calibration standard
C_{IS} = Concentration of the internal standard
a = Slope of the line (also called the coefficient of C_S)
b = The intercept

10.5.6 Non-linear calibration
In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen should have no more than four parameters, i.e., if the model is polynomial, it may be no more than third order, as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

10.5.7 If any target compounds are beyond the calibration range (i.e. >200 μg/mL for acids or >100 μg/ml for base neutrals) the extract must be diluted in methylene chloride (so that the concentration of the largest target compound is in the upper half of the calibration range), have internal standard added to obtain a final concentration of 40μg/ml, and re-analyzed. Dilutions are noted in the ABN sample run-log notebook.

10.5.8 Target compounds are identified qualitatively based on retention time and relative m/z abundances. The relative retention times of the sample compounds must be within 0.06 relative retention time units of the standard compounds. Furthermore, relative abundances of the compound's key ions must agree with 30 percent of those of the standard's. Sample chromatogram and peak spectra can be analyzed and edited using the "Qedit" option in the "Quant" menu. "False positive" peaks are Q-deleted.

10.5.9 Non-target compounds may be identified using the NIST library (nbs75k.l). The non-target peaks can be searched individually or a Library Search Compound (LSC) report can be generated. The LSC report is used to search all peaks in the chromatogram that were not identified by the quantitation database search algorithm. Library search strategies are set by the analyst as well as integration parameters. They may be quantitated using the nearest internal standard as in Equation 5.

Equation 5 $$\text{Concentration} = \frac{(As)(Cis)(Vt)}{(Ais)(Vo)(D)}$$

where:
As= Area of the characteristic m/z for the parameter to be measured.
Ais=Area of the characteristic m/z for the internal standard.
D=(100 - per cent moisture/100)for solid samples to be reported on a dry weight basis. Use 1 for water samples or solid samples that are reported on a wet weight basis.
Vt= Dilution.
Cis=Amount of Internal Standard added to each extract (μg/L).
Vo= Volume of water extracted (L), or weight extracted (kg).

Alternatively, the compound may be reported as "present but not calibrated for".

10.5.10 Prior to removal from the auto-sampler, re-confirm that the sequence in the auto sampler agrees with that in the run log. Note this in the run log (Figure 2) under "Unloading check".

11. Quality Control

11.1 Auto-tuning should be done as the system degrades and tuning becomes difficult. Auto-tuning is also good for diagnostic purposes when troubleshooting the system. The software has the capability of target tuning to DFTPP specifications. This is done by selecting "Target Tune" from the "TuneMS" window of the "ENVDA #1". The target tune is routinely saved as ABN.U. All aquisition methods will use the ABN.U tune file. Keep a hardcopy of the Target Tune report in the diagnostic section of the Semi-Volatiles QC notebook for use in monitoring system performance.

11.2 The initial calibration is verified by an independent source calibration check standard.

11.3 Calibration check- criteria

11.3.1 Every twelve hours

11.3.2 A 50ng DFTPP tune mix must pass the criteria listed in Table 5, as well as tailing criteria for Benzidine and Pentachlorophenol (see 9.2.4)

11.3.3 The calibration is verified with the midpoint calibration standard. Refer to section 10.4 for specific criteria

11.4 Per sample

11.4.1 Surrogates are added to each sample, blank, lab control sample and matrix spike during the extraction process. Percent recoveries are calculated by dividing the amount found by the amount added. The recovery acceptance ranges are listed in the analytical method on the instruments. Whenever a surrogate is outside of the acceptable range, an investigation of the extraction and analytical procedures is conducted. The extract may be re-analyzed to determine whether instrumental error occurred. A QA Deviation Form (Figure 3) is completed explaining the cause and corrective action taken. This form is filed with the chromatogram in the client's folder and a copy is filed with the QA officer. If an explanation cannot be determined, the sample is re-extracted, if original sample permits. Otherwise, the result is qualified.

11.4.2 Surrogate recoveries are charted in accordance with SOP QA-M023 in batches of 20 according to matrix. The analyst uses this information to look for trends, which can indicate a problem with the instrument, extraction procedure or spiking solution. The following conditions shall be considered out-of-control events for all control charts:

- One point outside control limits
- Three consecutive points outside warning limits
- Six consecutive points such that each point is larger (or smaller) than its immediate predecessor
- Eight consecutive points on the same side of the centerline

11.4.3 Internal standards are added to every extract. The areas of the internal standards in the samples must be -50% to +100% of the areas of the internal standards of the midpoint level of the initial calibration. In addition, the IS retention time cannot shift by more than 30 seconds from the retention time of the midpoint of the initial calibration. If the internal standards are out of range, the sample must be rerun. If they are still out of range, corrective action must be taken. A QA Deviation Form (Figure 3) is completed explaining the cause and corrective action taken. This form is filed with the chromatogram in the client's folder and a copy is filed with the QA officer. Non-target compounds directly affect responses of the internal standards therefore dilutions often solve the problem, howeverthe QC officer or Laboratory Director should be consulted before large dilutions are made due to matrix affect of non-target analytes Note any maintenance in the instrument maintenance log. In addition note in the comments section of the sample run log why the run was not acceptable.

11.5 Per sample batch (20 samples or per eight hour shift)

11.5.1 An extraction blank must be performed with every sample batch (20 or less co-extracted samples). A blank consists of 1-Liter of Reagent water, or 30 g sodium sulfate that is carried throughout the extraction procedure. Blanks are analyzed just prior to the appropriate sample batch to monitor interferences or contaminants that may have occurred during the extraction process. Samples associated with failing blanks must be re-extracted if sufficient sample exists. Otherwise, all results are qualified with a "B" flag and a QA Deviation Form (figure 5) is filled out and given to the QA officer The source of contamination should be determined and the necessary

precautions to prevent future interferences will become a part of the appropriate Standard Operating Procedure(s).

11.5.2 Laboratory control samples are extracted daily and/or every 20 samples. They are prepared in accordance with the appropriate extraction procedure (10.1).

11.5.3 All LCS recoveries should be calculated using the following formula:

$$\text{Recovery}=\%R=\frac{Cs-Cu}{Cn}X100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the un-spiked sample aliquot(use 0 for non-detects)
Cn = Nominal (true) concentration

If the spike recoveries do not fall within the established laboratory ranges the analyst must determine the cause, i.e. matrix effect or analytical/extraction error. A QA Deviation Form(Figure 3) is completed and given to the QA officer.

11.5.4 All LCS recoveries should be plotted on control charts as described in SOP QA-M023

11.6 Per QC Batch (20 samples or 1 month period)

11.6.1 Laboratory control duplicate samples are extracted monthly and/or every 20 samples.
They are prepared in accordance with the appropriate extraction procedure (10.1). Recoveries are calculated as stated in section 10.5.3
Additionally the relative percent difference (RPD) of the LCS and LCSD is calculated. The limits for the RPDs are listed in Methods on athe analytical instrument.

The RPD is calculated by using the formula: $\frac{(D1-D2)}{(D1+D2)/2}$ X 100

where,
D1 = Measured concentration of the LCS
D2 = Measured concentration of the LCS duplicate

11.6 One matrix spike and matrix spike duplicate is performed per batch of 20 soil samples. Table 8 lists laboratory acceptance limits for the spiking compounds If the spike recoveries do not fall within these ranges the analyst must determine the cause, i.e. matrix effect or analytical/extraction error. A QA Deviation Form (Figure 3) is completed and given to the QA officer.

11.7 Periodically

11.7.1 Minimum detection limit studies are performed whenever a major analytical change or instrumentation change takes place.. A series of at least 7

standards are extracted and analyzed. MDL's are calculated using the following formula:

MDL=(t)(S)

where MDL= Minimum Detection Limit
t = Students' t value at the 99% confidence level
S = standard deviation

11.8 A validation study is performed by each analyst for initial demonstration of capability of measuring accuracy and precision before any analyst is considered trained.

11.9 Run log sequences are confirmed when loading and unloading samples.

12. Routine Instrument Maintenance

12.1 Any maintenance performed on the instrument shall be noted in the maintenance logbook. The log entry should include a brief description of the maintenance, the problem that precipitated the maintenance and any instrument part numbers where applicable. Each entry must be dated and initialed.

12.2 The GC inlet must be cleaned ocassionally using a methanol. Refer to the *HP 5890 Series II Manual* for cleaning instructions. Benzoic Acid and Benzidine compounds are good indicators for inlet maintenance.

12.2.1 The inlet septum must be replaced daily, or after every 50-100 injections. An 11mm (7/16") septum is used as specified in Section 6. This is necessary to prevent leaks and septum coring.

12.2.2 A new deactivated splitless liner should be installed daily. Refer to SOP QA-M011 for cleaning and preparing liners. Hexachlorocyclopentadiene recovery is a good indicator of a dirty liner. Also pentachlorophenol and 2,4- Dinitrophenol are affected by dirty liners.

12.2.3 The inlet seal at the base of the GC injection port comes into contact with the sample and must be changed approximately once per month to prevent adsorption of active compounds. In addition, septum fragments and sample residue accumulate on the disk surface requiring replacement. The gold plated seals described in Section 6 are used due to their ability to deform easily making a leak-tight seal and also because of their superior inertness. Acid recoveries are a good indicator of a contaminated seal.

12.2.4 The column must be trimmed periodically. Typically the last three late eluting compounds are good indicators of when the column should be trimmed because they are the heaviest and have poor recoveries when trimming is necessary. Also chromatographic peak shape of these compounds is an early indicator of column residues.

12.2.5 The inlet port should be lightly scrubbed with 600 grit aluminum oxide paper approximately once per month to prevent adsorption of compounds onto active sites.

12.3 The capillary column specified in Section 6 is typically replaced every three to four months.

Pentachlorophenol is a good indicator of column degradation. Pentachlorophenol's response factor will decrease over time and when the first level of the five point calibration standard shows a faint response for pentachlorophenol, column replacement may be necessary.

12.4 Source Maintenance-Refer to the *HP 5972A MSD Hardware Manual* for detailed instructions on filament replacement and source cleaning.

12.5 Column replacement is noted in the instrument maintenance logbook. Each entry must have the date the column was installed, a description of the column, (to include manufacturer, column length, internal diameter, film thickness, catalogue number, and maximum temperature limit).

13. Pollution prevention and waste management.

13.1 See SOP W001 for handling of waste and unused portions of sample.

14.0 ABN Troubleshooting

Symptom	Cure
• d14-p-Terphenyl surrogate is high in ABN extracts	• Test the surrogate standard for concentration of d14-p-Terphenyl
• C36 is gone on PT1 Aliphatic standard (EPH)	• Scrub the injection port with green sandpaper
• The responses for the phenols and benzoic acid in daily are low	• Swab injection port with MeOH Clean gold seal with $MeCl_2$ or replace gold seal **Cut 1 loop off of the column (trimming)**
• Ion 441 is missing in the DFTPP tune	• Target tune **(after target tune set voltage back to current voltage)** If this doesn't solve the problem, clean the source.
• Ion 127 is below tune criteria	• Increase voltage and rerun DFTPP
• Hexachlorocyclopentadiene is low in second source check	• The BN stock mix is getting old, replace with a new vial
• Rf for 2,4-Dinitrophenol and 4-Nitrophenol in PT1 is less than 0.1 **after trimming**.	• Need new column
• Have to use linear fit in 5PT for 10 or more compounds **after trimming**. (Only if average % RSD > 15%)	• Need new column
• Linear fit for phenols just barely passes 0.99 criteria (ex. 0.990) **after trimming**.	• Need new column

Table 1

QL= Quantitation Limit

Target Compounds

Compound	CAS #	Aqueous QL ug/l	Solid QL ug/kg
Pyridine	110-86-1	2	250
N-Nitrosodimethylamine	62-75-9	2	250
Aniline	62-53-3	2	250
Phenol	108-95-2	5	250
2-Chlorophenol	95-57-8	5	250
Bis(2-Chloroethyl) Ether	111-44-4	2	250
1,3-Dichlorobenzene	541-73-1	2	250
1,4-Dichlorobenzene	106-46-7	2	250
1,2-Dichlorobenzene	95-50-1	2	250
Benzyl alcohol	100-51-6	5	250
2-Methylphenol (o-Cresol)	95-48-7	5	250
Bis (2-chloroisopropyl) ether	108-60-1	2	250
3-Methylphenol	108-39-4	5	250
and 4-Methylphenol (m,p-Cresols)	106-44-5	5	250
Hexachloroethane	67-72-1	2	250
N-Nitroso-di-n-propylamine	621-64-7	2	250
Nitrobenzene	98-95-3	2	250
Isophorone	78-59-1	2	250
2-Nitrophenol	88-75-5	5	250
2,4-Dimethylphenol	105-67-9	5	250
Bis (2-chloroethoxy) methane	111-91-1	2	250
2,4 Dichlorophenol	120-83-2	5	250
Benzoic Acid	65-85-0	10	250
1,2,4-Trichlorobenzene	120-82-1	2	250
Naphthalene	91-20-3	2	250
2,6-Dichlorophenol	87-65-0	5	250
4 -Chloroaniline	106-47-8	2	250
Hexachlorobutadiene	87-68-3	2	250
4-Chloro-3-methylphenol	59-50-7	10	250
2-Methylnaphthalene	91-57-6	2	250
Hexachlorocylopentadiene	77-47-4	2	250
2,4,6-Trichlorophenol	88-06-2	5	250
2,4,5-Trichlorophenol	95-95-4	5	250
2-Chloronaphthalene	91-58-7	2	250
2-Nitroaniline	88-74-4	2	250
2,6-Dinitrotoluene	606-20-2	2	250
Dimethyl phthalate	131-11-3	2	250
Acenaphthylene	208-96-8	2	25

Table 1 (continued)

QL= Quantitation Limit

Target compounds

Compound	CAS #	Aqueous QL ug/l	Solid QL ug/kg
3-Nitroaniline	99-09-2	2	250
Acenaphthene	83-32-9	2	250
2,4-Dinitrophenol	51-28-5	5	250
Dibenzofuran	132-64-9	2	250
4-Nitrophenol	100-02-7	5	250
2,4-Dinitrotoluene	121-14-2	2	250
2,3,4,6-Tetrachlorophenol	58-90-2	5	250
Fluorene	86-73-7	2	250
Diethyl phthalate	84-66-2	2	250
4-Chlorophenyl phenyl ether	7005-72-3	2	250
4-Nitroaniline	100-01-6	2	250
4,6-Dinitro-2-methylphenol	534-52-1	5	250
N-Nitrosodiphenylamine	86-30-6	2	250
Azobenzene	103-33-3	2	250
4-Bromophenyl phenyl ether	101-55-3	2	250
Hexachlorobenzene	118-74-1	2	250
Pentachlorophenol	87-86-5	10	250
Phenanthrene	85-01-8	2	250
Anthracene	120-12-7	2	250
Carbazole	86-74-8	2	250
Di-n-butyl phthalate	84-74-2	2	250
Fluoranthene	206-44-0	2	250
Benzidine	92-87-5	20	250
Pyrene	129-00-0	2	250
Butyl benzyl phthalate	85-68-7	2	250
Benz (a) anthracene	56-55-3	2	250
Chrysene	218-01-9	2	250
3-3'-Dichlorobenzidine	91-94-1	20	250
Bis (2-ethylhexyl) phthalate	117-81-7	2	250
Di-n-octyl phthalate	117-84-0	2	250
Benzo (b) fluoranthene	205-99-2	2	250
Benzo (k) fluoranthene	207-08-9	2	250
Benzo [a] pyrene	50-32-8	2	250
Indeno [1,2,3-cd] pyrene	193-39-5	2	250
Dibenz [a,h] anthracene	53-70-3	2	250
Benzo [ghi] perylene	191-24-2	2	250

Table 2
Scan Acquisition Method Tunemix.M Parameters

[Method Name: C:\HPCHEM\1\METHODS\tunemix.M]

Aquisition Mode	Scan
Tune File	ABN.U

[Injector Information]	
Injection Source	Auto
Injection Location	Rear
Sample Washes	3
Sample Pumps	5
Injection Volume	1.0 μL
Solvent A	Methylene Chlorine
Solvent B	Toluene
Post Injection Solvent A Washes	3
Post Injection Solvent B Washes	3
Viscosity Delay	0
Plunger Speed	Fast

GC Temperature Information

[GC Zone Temperatures]	
Inlet A	Off
Inlet B	250°C
Detector A	Off
Detector B	280°C
[Oven Program]	
Equilibration	0.00 minutes
Oven Max	325°C
Oven State	On
Initial Temperature	70°C
Initial Time	0 min
Rate	20°C/min
Final Temperature	320°C
Final Time	0 min
Next Run Time	12.5 min

Table 2 (continued)

Scan Acquisition Method Tunemix.M Parameters

GC Inlet Information

[Inlet B Pressure Program]		
Constant Flow	On 7.7 psi	
[Inlet B Flow settings]		
Column Length	30m	
Column Diameter	0.25 mm	
Gas	Helium	
Vacuum Compensation	On	
Pressure	7.7 psi	
Flow	1.0 ml/min	
Inlet Purge B	Initial Value: off	On Time: 0.50

MS Information

Solvent Delay:	7.42 minutes	
EM Voltage	Relative, 0 ABN.U	
[Scan Parameters]		
Low Mass	35	
High Mass	500	
Threshold	variable	
Sampling #	2	A/D Samples 4

Table 3

Scan Acquisition Method SMMDDY.M Parameters

[Method Name: C:\HPCHEM\1\METHODS\SMMDDY.M]

Aquisition Mode	Scan
Tune File	ABN.U
[Injector Information]	
Injection Source	Auto
Injection Location	Rear
Sample Washes	3
Sample Pumps	5
Injection Volume	1.0 μL
Solvent A	Methylene Chlorine
Solvent B	Toluene
Post Injection Solvent A Washes	6
Post Injection Solvent B Washes	0
Viscosity Delay	0 sec
Plunger Speed	Fast
GC Temperature Information	
[GC Zone Temperatures]	
Inlet A	Off
Inlet B	250°C
Detector A	Off
Detector B	280°C
[Oven Program]	
Equilibration	0.50 Minutes
Oven Max	325°C
Oven State	On
Initial Temperature	40°C
Initial Time	2 min
Rate	10°C/min
Final Temperature	280°C
Rate	1°C/min
Final Temperature	285°C
Final Time	2 min
Next Run Time	33 min

Table 3 (continued)

Scan Acquisition Method SMMDDY.M Parameters

GC Inlet Information

[Inlet B Pressure Program]			
Initial Pressure	7.7 psi		
Initial Time		0.0 min	
Level 1 Rate		99 psi/min	
Level 1 Final Pressure		40 psi	
Level 1 Final Time		0.30 min	
Level 2 Rate		99 psi/min	
Level 2 Final Pressure		7.7 psi	
Total Program Time		0.95 min	
[Inlet B Flow settings]			
Column Length		30m	
Column Diameter		0.25 mm	
Gas		Helium	
Vacuum Compensation		On	
Pressure		7.7 psi	
Flow		1.0 ml/min	
Inlet Purge B		Initial Value: Off	On Time: 0.50

MS Information

Solvent Delay:	1.1 minutes	
EM Voltage	Relative, 0 ABN.U	
[Scan Parameters]		
Low Mass	35	
High Mass	500	
Threshold	variable	
Sampling #	2	A/D Samples 4

Table 4

SIM Acquisition Method SIMMMDDY.M Parameters

[Method Name: C:\HPCHEM\1\METHODS\SIMMMDDY.M]

Aquisition Mode	Sim
Tune File	ABN.U
[Injector Information]	
Injection Source	Auto
Injection Location	Rear
Sample Washes	3
Sample Pumps	5
Injection Volume	1.0 μL
Solvent A	Methylene Chlorine
Solvent B	Toluene
Post Injection Solvent A Washes	6
Post Injection Solvent B Washes	0
Viscosity Delay	0 sec
Plunger Speed	Fast
GC Temperature Information	
[GC Zone Temperatures]	
Inlet A	Off
Inlet B	250°C
Detector A	Off
Detector B	280°C
[Oven Program]	
Equilibration	0.50 Minutes
Oven Max	325°C
Oven State	On
Initial Temperature	40°C
Initial Time	2 min
Rate	10°C/min
Final Temperature	275°C
Final Time	8.5 min
Next Run Time	34 min

Table 4 (continued)

SIM Acquisition Method SIMMMDDY.M Parameters

GC Inlet Information

[Inlet B Pressure Program]		
Initial Pressure	7.7 psi	
Initial Time	0.0 min	
Level 1 Rate	99 psi/min	
Level 1 Final Pressure	40 psi	
Level 1 Final Time	0.30 min	
Level 2 Rate	99 psi/min	
Level 2 Final Pressure	7.7 psi	
Total Program Time	0.95 min	
[Inlet B Flow settings]		
Column Length	30m	
Column Diameter	0.25 mm	
Gas	Helium	
Vacuum Compensation	On	
Pressure	7.7 psi	
Flow	1.0 ml/min	
Inlet Purge B	Initial Value: Off	On Time: 0.50

MS Information

Solvent Delay:	7.00 minutes
EM Voltage	Relative, 0 ABN.U
[Sim Parameters]	
Group 1	Group 1
Dwell Per Ion	100 msec
Group Start Time	7.00 min (prior to elution of D5-nitrobenzene)
Ions in Group	82,102,115,128,136,141,142
Group 2	
Group ID:	Group 2
Dwell Per Ion	100 msec
Group Start Time	13.00 min (prior to elution of 2-fluorobiphenyl))
Ions in Group	76,127,152,154,162,164,166,172

Table 4 (continued)

SIM Acquisition Method SIMMMDDY.M Parameters

Group 3

Group ID:	Group 3
Dwell Per Ion	100 msec
Group Start Time	16.50 min (prior to elution of phenanthrene)
Ions in Group	76,101,178,188,202

MS Information (continued)

Group 4

Group ID:	Group 4
Dwell Per Ion	100 msec
Group Start Time	21.40 min (prior to elution of pyrene)
Ions in Group	101,114,202,228,240,244

Group 5

Group ID:	Group 5
Dwell Per Ion	100 msec
Group Start Time	26.00 min (prior to elution of benzo(b)fluoranthene)
Ions in Group	113,126,132,138,252,264,276,278

Table 5

DFTPP Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Degradation of DDT to DDD and DDE should not exceed 20%. Calculate breakdown as follows;

$$\% \text{ breakdown DDT} = \frac{\text{sum of degradation peak areas of DDD + DDE}}{\text{sum of peak areas of DDT + DDD + DDE}} \times 100$$

Benzidine and pentachlorophenol should be present at their normal responses, and the peak tailing factor should be less than 3 and 5 respectively. (See 9.2.4) If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may be necessary to remove 6-12 inches from the front of the column.

Table 6

Calibration Check Compounds (CCC)
Percent Relative Standard Deviation Must be <30% for Initial Calibration, Relative Percent Difference must be <20% for Continuing Calibration

Base-Neutral Compounds	Acid Compounds
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

System Performance Check Compounds (SPCC's)
Minimum Acceptable Average Response Factor is 0.050

Base-Neutral Compounds	Acid Compounds
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

Table 7

Surrogate Recovery Limits for Soil and Water Samples

Surrogate Compound	Water	Soil
d5-Nitrobenzene	40-119	25-99
2-Fluorobiphenyl	40-109	33-96
d14-p-Terphenyl	26-130	19-121
d5-Phenol	11-79	35-98
2-Fluorophenol	29-82	33-90
2,4,6-Tribromophenol	54-116	44-89

Table 8

Matrix Spike Recovery Limits for Soil and Water Samples

Matrix Spike Compound	Acceptance Limit	
	Water	Soil
Pyrene	44-99	42-99
Acenaphthene	42-90	42-81
N-Nitroso-di-n-propylamine	41-95	36-83
1,4-Dichlorobenzene	32-74	31-76
2,4-Dinitrotoluene	50-95	49-84
1,2,4-Trichlorobenzene	35-78	34-84
Phenol	20-43	43-82
2-Chlorophenol	48-85	41-82
4-Nitrophenol	12-46	39-95
Pentachlorophenol	41-114	29-115
4-Chloro-3-methylphenol	57-92	48-92

Table 9

QC Limits for Relative Percent Difference

Method	Analytes	Limit
EPA 625 Acid	Phenol	42
	2-Chlorophenol	50
	4-Nitrophenol	50
	Pentachlorophenol	50
	4-Chloro-3-methyl phenol	42
SW-846 8270 Acid	Phenol	35
	2-Chlorophenol	50
	4-Nitrophenol	50
	Pentachlorophenol	47
	4-Chloro-3-methylphenol	33
	Other Compounds	50
EPA 625 Base / Neutral		
	Pyrene	31
	Acenaphthene	31
	N-Nitroso-n-propylamine	38
	1,4-Dichlorobenzene	28
	2,4-Dinitrotoluene	38
	1,2,4 Trichlorobenzene	28
SW-846 8270 Base / Neutral		
	Pyrene	36
	Acenaphthene	19
	N-Nitroso-n-propylamine	38
	1,4-Dichlorobenzene	27
	2,4-Dinitrotoluene	47
	1,2,4-Trichlorobenzene	23
	Other Compounds	50

Figure 1

GC/MS Semi-volatile Organics: EPA Method 625/8270

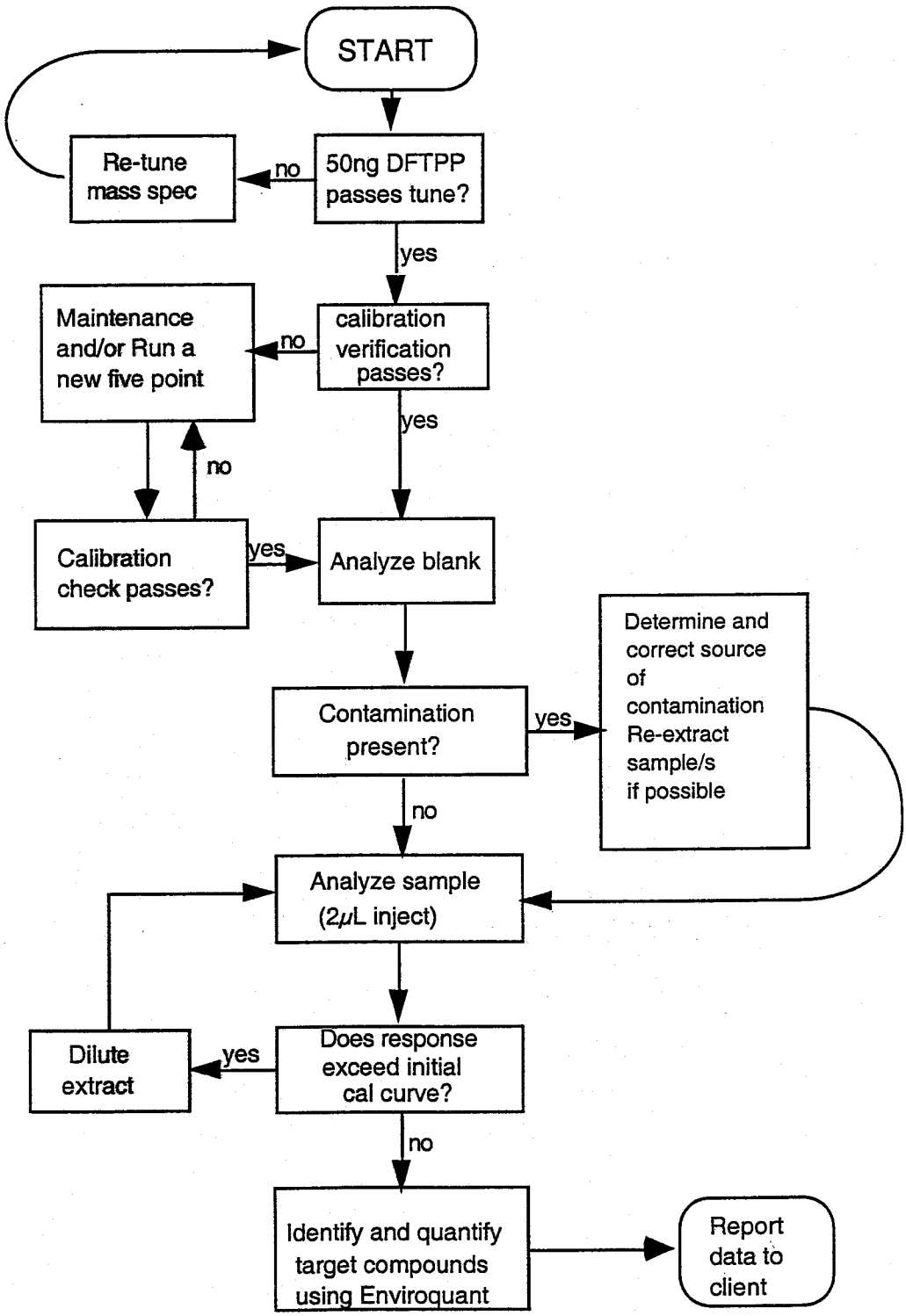

Figure 2

INSTRUMENT D PAGE:

Tune File:_____ Date:_____ Loading Check:_____

Voltage:_____ Analyst:_____ Unloading Check:_____

Tube	Method	Sample Information	Dil	Data File	Comments

REV 2/98

Figure 3

Quality Assurance Deviation

Analysis Code:
QC Batch ID:
(attach copy or list samples affected by this memo)

Out of Control Event with Brief Explanation

☐ Method Blank

☐ Continuing Calibration or Initial Calibration Issue

☐ Laboratory Control Spike

☐ Matrix Spike/MSD

☐ Surrogate Recovery

☐ Other

SOP Corrective Action Followed:

☐ Re-extract in Hold time

☐ Re-inject to confirm

☐ Other

☐ Document SOP corrective action or "See QA Memo" on raw data file

QA Officer Review: _____ _____
Initials Date

Comments:

analytics environmental laboratory LLC

195 Commerce Way Suite E
Portsmouth, New Hampshire 03801
603-436-5111 Fax 603-430-2151
800-929-9906
analytics@analyticslab.com

COPY

Sonication Extraction for the Determination of Semi-Volatile Compounds in Solid Samples

Approved By:

QA Officer: Melissa A Milli Date: 11/06/01

Laboratory Director: Steven L. Kroll Date: 11/7/01

Revision History

Revision	Description of Change	Approval	Date

1. Title

Sonication Extraction For The Determination Of Semi-Volatile Compounds In Solid Samples.

EPA References: Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Method 3550B, Revision 2, December 1996

Test Methods for Evaluating Solid Waste Physical/Chemical Methods-Method 3500B, Revision 2, December 1996

Comprehensive Quality Assurance Plan for Analytics Environmental Laboratory, LLC, January 2001

2. Scope and Application

This method describes the isolation and concentration of semi-volatiles from solid samples in preparation for analysis by GC/MS. The parameters listed in Table 1 can be measured by this method.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevation in baselines. Method blanks must be routinely analyzed to show the absences of these interferences.

3.2 Interferences co-extracted from the sample will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample extract may be necessary.

3.3 Phthalates esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because of phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time id consistent quality control is not practiced.

3.4 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of phenolic analytes. This problem is especially pronounced with glassware that may be difficult to rinse. These items should be hand rinsed to avoid this problem.

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard.

4.2 Exposure should be minimized by wearing solvent resistant gloves, safety goggles and a laboratory coat when samples or standards are handled, and by working in a fume hood.

4.3 **The following compounds included in this method are classified as known or suspected human carcinogens: benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, methylene chloride and N-nitrosodimethylamine.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

4.4 The sonicator probe should never be touched when engaged. The owner's maual should be consulted regarding other safety concerns and the maintenance of the sonicator.

5. Method Summary

Approximately 30 g of the solid sample is mixed with sodium sulfate then sonicated three times for three minutes with methylene chloride. The methylene chloride extract is then dried, concentrated to a volume of 1.0 ml, and analyzed by GC/MS.

6. Sample Collection, Handling and Preservation

6.1 All samples should be collected in glass jar with a screw top Teflon lid.

6.2 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling instructions.

6.3 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and log-in procedures.

6.4 Solid samples must be extracted within 14 days of collection.

6.5 Sample extracts must be analyzed within 40 days of extraction.

6.6 Extracts must be stored in a freezer at –10°C to –20°C protected from light.

7. Definitions

7.1 ml = milliliter

7.2 g =gram

7.3 GC/MS = gas chromatograph/mass spectrometer

7.4 ABN = Acid/Base Neutral

7.5 °C = degrees Celsius

7.6 QC = quality control

7.7 AEL # = Analytics Environmental Laboratory sample identifier

7.8 N_2 = nitrogen gas\

7.9 psi = pounds per square inch

7.10 CAS = Chemical Abstract Service

7.11 LCS = Laboratory Control Sample

8. Apparatus and Materials

8.1 Sonicator - Heat systems, Inc. Ultrasonic Liquid Processor Model XL2020, equipped with twin ¾-inch titanium disrupter horns or equivalent.

8.2 Super Sonabox w/ring clamp and beaker tray - Heat System, Ultrasonics, Inc. Part Number 432B or equivalent.\

8.3 Turbo-Vap II concentration Workstation - Zymark Part Number 46368 or equivalent.

8.4 250 ml glass beakers

8.5 250 ml Erlenmeyer flask, glass.

8.6 Long stem funnel with a glass wool plug.

8.7 Turbo-Vap Tubes with 1.0 ml stem.

8.8 Disposable pasteur pipettes, 9 inch glass.

8.9 metal spatula

8.10 Class A volumetric glassware as required for final volume determination - rinsed 3 times with methylene chloride prior to use.

8.11 2 ml amber auto-sampler vials with Teflon™ lined crimp cap

8.12 12 ml amber vials with Teflon™ lined screw caps

8.13 0.5 and 1 ml syringe.

8.14 Kimwipes

8.15 (2)- 50 ml graduated cylinders

8.16 (2)- 400 ml Teflon™ beakers for rinsing sonicator tips.

8.17 Balance capable of accurately weighing to 0.01 grams.

8.18 Purple labeling tape.

8.19 ABN sample preparation logbook.

8.20 Nitrogen blow down apparatus.

8.21 Wax pencil

9. Standards and Reagents - equivalent may be used

9.1 Methylene chloride - Pesticide quality or equivalent.

9.2 Sodium sulfate - Granular, anhydrous. Purified by heating at 200 °C for a minimum of four hours in a shallow glass baking dish and stored in a sealed glass container.

9.3 Silica Sand - reagent grade.

9.4 ABN surrogate - Refer to QA-S001 for Standard preparation.

9.5 ABN Matrix Spiking Solution - Refer to QA-S001 for Standard preparation.

9.6 Nitrogen Gas-grade 5, from a liquid nitrogen tank.

9.7 Methanol - purge and trap grade or equivalent for syringe rinsing.

9.8 Nitrogen gas - grade 5 from a liquid nitrogen tank.

10. Procedure

10.1 Sample Preparation Notebook

All information pertinent to semi-volatile sample preparation is recorded in a bound paginated laboratory notebook. Each entry should contain a general heading with the extraction date, ABN-SON and the initials of the analyst(s) that performed the extraction. A column format should then include the following headings: sample number, matrix, weight (g), standard number and amount of surrogate added, standard number and amount of spike added, final volume, the date the concentration was completed and initials of the analyst who concentrated the sample. Observations about specific samples must also be recorded in the notebook.

10.2 Determine Necessary Quality Control

Each client sample extracted is recorded in the QC summary notebook in groups of 20 (Figure 1). QC includes blank, laboratory control sample, matrix spike sample, and matrix spike duplicate sample.

10.2.1 One method blank, consisting of approximately 30 g of sodium sulfate must be extracted with each analytical batch (group of co-extracted samples not to exceed 20 samples). Blanks are labeled as follows: BMMDDYAS, where;

B designates a blank
MM is a two number designation for the month
DD is a two number designation of the day
Y is a single number designation for the year
AS signifies that the extract is an Acid/Base Neutral sonication

10.2.2 A laboratory control sample must be extracted with each analytical batch (group of co-extracted samples not to exceed 20 samples). The laboratory control sample consists of 30 g of silica sand mixed with approximately 30 g of sodium sulfate (enough to cover the silica sand). The mixture is spiked with 1.0 ml of the ABN matrix spiking solution and 0.5 ml ABN surrogate spiking solution. The laboratory control sample is subjected to the same extraction procedure as the associated

samples, thus indicating extraction accuracy. Laboratory control samples are labeled as follows: LMMDDYAS where;

L designates a laboratory control sample
MM is a two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the year
AS signifies that the extract is an Acid/Base Neutral Sonication

10.2.3 One matrix spike and the matrix spike duplicate must be extracted for every twenty samples or within a one month period. A matrix spike consists of 30 g of a client sample mixed with approximately 30 g of sodium sulfate. The mixture is spiked with 1.0 ml of the ABN matrix spiking solution and 0.5 ml ABN surrogate spiking solution. The matrix spikes must be subjected to the same extraction procedure as the associated samples, thus indicating matrix effects. Matrix spikes are labeled with the client sample number and the suffix MS and MSD.

10.3 Sample Extraction

10.3.1 Connect sonicator control unit to the sonibox by attaching cable coming from the top of the sonibox to the back of the control unit. Turn the sonicator on by pressing the red switch on the lower left --switch panel will light when on. Set intensity dial to maximum setting, 10. Readout will prompt procedure for preparing sonicator for use. (Press "CLEAR"; press "PROG./DATA", press number 1). The number displayed on the upper left of the readout is the total time the sonicator will be in the "PULSAR ON" phase; the number on the lower left is the "PULSAR ON" interval, while the number on the lower right is the "PULSAR OFF" interval.

10.3.2 Program number 0 is set for three minutes total process time with the pulsar "ON" for 0.5 sec and pulsar "OFF" for 0.5 sec. Use program 0 for sonicating samples.

10.3.3 Program number 1 is set for 30 sec total process time and the same ON/OFF intervals as program 0. Use program 1 for rinsing sonicator tips in ~250 ml methylene chloride between samples. Use the Teflon™ beakers for rinsing tips since glass beakers tend to shatter. Rinse the tips using Program 1 prior to sonicating any samples.

10.3.4 For each sample, pre-rinse a 250 ml beaker and a 250 ml Erlenmeyer flask with methylene chloride. Use purple tape to label the flask with the AEL #, ABN SON and the associated blank number. Also label the beaker with the AEL # using a wax marker.

10.3.5 Calibrate the balance as per QA-M009.

10.3.6 Weigh approximately 30 g of sample into the beaker (decant any free liquid first.). Record the weight to the nearest one hundredth of a gram in the sample preparation logbook.

10.3.7 Add approximately 30 g of sodium sulfate to the sample (more if the sample seems especially damp or wet) and mix thoroughly to uniform consistency. The sample should be free flowing at this point.

10.3.8 Using a 0.5 ml syringe, designated for ABN surrogate, add 0.5 ml of ABN surrogate solution to the sample in the beaker.

10.3.9 If a sample is designated as a matrix spike, matrix spike duplicate, or a laboratory control sample use a 1 ml syringe to add 1.0 ml of the ABN matrix spiking solution to the sample in the beaker. **The syringe used for spiking is not a dedicated syringe so it must be rinsed 3 times before and after use with Purge and Trap grade methanol.**

10.3.10 Rinse sonicator probes by pouring approximately 250 ml methylene chloride into both Teflon beakers. Place the beakers on the sonicator tray and raise the ring so that sonicator tips are immersed in methylene chloride but not touching the beakers. Close sonibox and check to make sure sonicator is set to program 1 and press "START/STOP" button to begin sonication. Sonicator will stop automatically when total process time has been reached.

10.3.11 Using a graduated cylinder, pour 100 ml of methylene chloride into the beaker. Place beaker(s) on tray in sonibox. Two sample beakers may be sonicated at the same time. If two samples are not sonicated, one of the rinse Teflon™ beakers containing solvent is placed on the tray instead. Raise the ring so that sonicator tip is immersed in methylene chloride but not touching the sample or the beaker. Close sonibox and check to make sure sonicator is set to program 0 and press "START/STOP" button to begin sonication. Sonicator will stop automatically when total process time has been reached.

10.3.12 Remove beaker(s) and decant methylene chloride into the labeled 250 ml flask, being cautious that none of the solid goes into the flask.

10.3.13 Repeat steps 10.3.11 and 10.3.12 two more times using 50 mls of methylene chloride. Stopper the flask and set aside.

10.3.14 After each sample has been completely processed, change program to 'rinse' program by pressing the number 1 on the sonicator keypad. If probes are particularly dirty, wipe each sonicator tip with a Kim wipe to remove particulates. Place Teflon™ beakers with about 200 ml methylene chloride on beaker tray and raise to immerse the tips. Press "START/STOP" to engage. One process time is sufficient to clean tips. Change program back to program 0 by pressing the number 0 to prepare for next sample. Change rinse methylene chloride as needed.

10.4 Extract Drying and Concentration

10.4.1 If extracts are not to be concentrated immediately they must be stored in a refrigerator.

10.4.2 Plug a long-stem funnel with glass wool and fill 1/2 full with baked sodium sulfate. Rinse the sodium sulfate with 10-20 ml of methylene chloride. Collect rinseate in a waste container.

10.4.3 Place the funnel into the Turbo-Vap tube. Pour the sample extract through the funnel into the Turbo-Vap tube.

10.4.4 Rinse the flask that contained the sample three times with methylene chloride and pour the rinsings through the funnel. **Transfer the label from the Erlenmeyer flask to the Turbo-Vap tube.**

10.4.5 When the sample has drained completely, rinse the sodium sulfate with an additional 20 ml of methylene chloride and allow to drain.

10.4.6 Set Turbo-Vap concentrator temperature to 32°C,with sensor selected as endpoint. Sensor selection will beep when sample reaches a volume of ≈0.75 ml. Set N_2 pressure to 20 psi.

10.4.7 Remove the funnel from the TurboVap tube. Place the tube into the Turbo-Vap chamber, close lid and press "on" buttons for the cell positions being used. Up to 6 samples may be concentrated at the same time.

10.4.8 Rinse a 1.0 ml volumetric flask three times with methylene chloride. Transfer concentrated sample extract with a clean 9 inch pasteur pipette into the 1 ml volumetric flask. With a clean pasteur pipette add a small amount of methylene chloride to the Turbo-Vap tube to rinse sides. Rinse the lower part of the Turbo tube at least three times with sample pipette. Using the original pipette transfer the methylene chloride to the volumetric flask. Repeat as many times as needed to achieve 1.0 ml volume in the flask. **(It is preferable to underestimate and do repeat rinsings rather**

than to overestimate and have some rinse methylene chloride remaining.)

10.4.9 Transfer the extract into a 2 ml amber auto-sampler vial labeled with the AEL #, ABN-SON, and the date. If the final extract volume is other than 1.0 ml, record this volume of the vial. Cap. Transfer the tape label from the Turbo tube to the cap of the auto-sampler vial and store the vial in the freezer designated for ABN extracts.

10.4.10 If the Turbo-Vap automatically shuts off before the extract has been reduced to <1.ml (this can happen repeatedly) the analyst has several options:

1) Reset the Turvo-Vap cell holding that sample, until the sample is cooked down to <1.0 ml. Then go back to 10.4.8 for vialing. This option should be considered in most cases unless the sample is extremely oily or contains particulates.
2) Transfer the sample from the Turbo-Vap tube to an appropriately sized collection tube. Set up nitrogen blow-down apparatus and condense sample to <1.0 ml or until sample volume reduction stops. If sample goes to <1.0 ml, return to section 10.4.8 for vialing
3) If sample volume can't be reduced to <1.0 ml using either of these techniques, the sample must be brought to the smallest convenient volume. Make an estimate of the volume of the remaining sample and choose a Class A volumetric of sufficient size to contain the extract plus any methylene chloride used to rinse the Turbo-Vap tube. Using a 1.0 ml syringe place **exactly** 1.0 ml of the extract in an amber 2.0 ml vial. Store the remainder of the extract in an appropriate container. Note the reason for the exceptionally large final volumes in the sample prep log book.

10.4.11 Extracts are stored in the freezer designated for ABN extracts and must be analyzed within 40 days.

10.4.12 A transfer sheet (Figure 2) is generated each day that samples are completed. The original is filed in the client's output folder,and a copy is given to the GS/MS analyst. Fill in "Final Volume" and "Date Transfered" columns of the sample prep log book and note any unusual observations.

11. Quality Control

11.1 Per sample

11.1.1 Surrogates are added to every sample, matrix spike, laboratory control sample and blank before extraction to test the efficiency of the extraction

procedure. The GC/MS analyst calculates percent recoveries. . If surrogate recoveries are outside the laboratory's pre-determined acceptance ranges, a QC Deviation Report (Figure 3) explaining the cause is completed. Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. In this case, the GC/MS analyst will transfer the deviation report to the extraction laboratory. Unusual circumstances during the extraction should have been noted in the sample preparation log notebook. Once the extraction laboratory has completed the deviation report, it should be returned to the GC/MS analyst. A copy is filed in the client's folder and the original is filed with the Quality Assurance Officer. Corrective action may include re-extracting the sample if sufficient sample exists.

11.2 Per sample batch

11.2.1 A method blank must be performed with every sample batch (20 samples per 8 hour shift). A blank consists of 30 g of sodium sulfate. The blank is extracted with the associated sample batch and carried throughout the extraction procedure. The blank is analyzed just prior to the associated samples to monitor interferences or contaminants that may have occurred during the extraction process. If a blank is found to contain more than the reporting limit of any compound, the sample batch must be re-extracted. If insufficient sample exists, the contaminant will be qualified with a "B" flag.

11.2.2 A Laboratory Control Sample must be performed with every sample batch (20 samples, or per eight hour shift). The laboratory control sample is subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy. The GC/ECD analyst will calculate the percent recoveries of the compounds. If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. Extraction personnel may be asked to determine the cause of the deviation. Unusual circumstances during the extraction are recorded in the preparation log.

11.3 Per QC Batch

11.3.2 A matrix spike and matrix spike duplicate must be extracted per 20 samples or within a one month period. A matrix spike consists of 30 g of a client sample mixed with 30 g of sodium sulfate. The mixture is spiked with 1.0 ml of the abn matrix spiking. The matrix spikes are subjected to the same extraction procedure as associated client samples. The GC/MS analyst will calculate the spike recoveries and relative percent differences. If the spike recoveries or relative percent differences are not within the laboratory's

acceptance limits, the cause must be determined and appropriate corrective action taken. A QC Deviation Form (Figure 3) will be completed.

11.4 The sonicator must be tuned monthly. Refer to Instruction Manual tuning instructions. Document tuning procedure in the miscellaneous analysis book.

11.5 Spiking solutions are tested prior to use according to QA-M015.

12.0 Pollution Prevention and Waste Management

12.1 See SOP W001 for handling of waste and unused portions of sample.

Table 1

Target Compounds

Compound	CAS #
Pyridine	110-86-1
N-Nitrosodimethylamine	62-75-9
Aniline	62-53-3
Phenol	108-95-2
2-Chlorophenol	95-57-8
Bis(2-Chloroethyl) Ether	111-44-4
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1
Benzyl alcohol	100-51-6
2-Methylphenol (o-Cresol)	95-48-7
Bis (2-chloroisopropyl) ether	108-60-1
3-Methylphenol	108-39-4
and 4-Methylphenol (m,p-Cresols)	106-44-5

Hexachloroethane	67-72-1
N-Nitroso-di-n-propylamine	621-64-7
Nitrobenzene	98-95-3
Isophorone	78-59-1
2-Nitrophenol	88-75-5
2,4-Dimethylphenol	105-67-9
Bis (2-chloroethoxy) methane	111-91-1
2,4 Dichlorophenol	120-83-2
Benzoic Acid	65-85-0
1,2,4-Trichlorobenzene	120-82-1
Naphthalene	91-20-3
2,6-Dichlorophenol	87-65-0
4 -Chloroaniline	106-47-8
Hexachlorobutadiene	87-68-3
4-Chloro-3-methylphenol	59-50-7
2-Methylnaphthalene	91-57-6
Hexachlorocylopentadiene	77-47-4
2,4,6-Trichlorophenol	88-06-2
2,4,5-Trichlorophenol	95-95-4
2-Chloronaphthalene	91-58-7
2-Nitroaniline	88-74-4
2,6-Dinitrotoluene	606-20-2

Dimethyl phthalate	131-11-3
Acenaphthylene	208-96-8

Table 1 (continued)

Target compounds

3-Nitroaniline	99-09-2
Acenaphthene	83-32-9
2,4-Dinitrophenol	51-28-5
Dibenzofuran	132-64-9
4 Nitrophenol	100-02-7
2,4-Dinitrotoluene	121-14-2
2,3,4,6-Tetrachlorophenol	58-90-2
Fluorene	86-73-7
Diethyl phthalate	84-66-2
4-Chlorophenyl phenyl ether	7005-72-3
4-Nitroaniline	100-01-6
4,6-Dinitro-2-methylphenol	534-52-1
N-Nitrosodiphenylamine	86-30-6
Azobenzene	103-33-3
4-Bromophenyl phenyl ether	101-55-3

Hexachlorobenzene	118-74-1
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Anthracene	120-12-7
Carbazole	86-74-8
Di-n-butyl phthalate	84-74-2
Fluoranthene	206-44-0
Benzidine	92-87-5
Pyrene	129-00-0
Butyl benzyl phthalate	85-68-7
Benz (a) anthracene	56-55-3
Chrysene	218-01-9
3-3'-Dichlorobenzidine	91-94-1
Bis (2-ethylhexyl) phthalate	117-81-7
Di-n-octyl phthalate	117-84-0
Benzo (b) fluoranthene	205-99-2
Benzo (k) fluoranthene	207-08-9
Benzo [a] pyrene	50-32-8
Indeno [1,2,3-cd] pyrene	193-39-5
Dibenz [a,h] anthracene	53-7-3
Benzo [ghi] perylene	191-24-2

FIGURE 1

QC SUMMARY ABN SOIL SAMPLES

Procedure: ABN SLD Batch ID# _____

	SAMPLE #	DATE	BLANK	LCS	MS	MSD
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

REV 1/98

FIGURE 2

Transfer Date: 02/03/98

EXTRACT TRANSFER SHEET

Initials: DD

Semi-Volatile Compounds:

Sample	Analysis	Extraction Date	Matrix	Initial Volume (mL)	Initial Weight (g)	Final Volume (mL)	% Solids	Extraction Comments
39287-1	8270S	02/03/98	Solid		25.59	1.0	39.75	
39290-1	8270BNS	02/03/98	Sludge		25.46	1.0	15.94	
39291-1	8270PAHS	02/03/98	Solid		25.13	1.0	95.08	

FIGURE 3

Quality Assurance Deviation and Documentation

Analysis Code:
QC Batch ID:
(attach copy or list samples affected by this memo)

Out of Control Event with Brief Explanation

☐ Method Blank

☐ Continuing Calibration or Initial Calibration Issue

☐ Laboratory Control Spike

☐ Matrix Spike/MSD

☐ Surrogate Recovery

☐ Other

SOP Corrective Action Followed:

☐ Re-extract in Hold time

☐ Re-inject to confirm

☐ Other

☐ Document SOP corrective action or "See QA Memo" on raw data file

QA Officer Review: _____ _____
Initials Date

Comments:

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COPY

Preparation of Solid Samples (High Level) Volatile Organic Analysis

Approved By:

QA Officer: Melissa A Stulli Date: 05/22/01

Laboratory Director: Steve L. Knollys Date: 5/22/2001

Revision History

Revision	Description of Change	Approval	Date

Title

Preparation of Solid Samples for Volatile Organic Analysis.

EPA references: Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 8000B, Revision 2, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 5035, Revision 0, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 8260B, Revision 2, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 8015, Revision 2, December 1996

Comprehensive Quality Assurance Plan for ANALYTICS Environmental Laboratory, LLC. January 2001

1. Scope and Application

2.1 This method is used to prepare solid samples for volatile organic analysis and Gasoline Range Organics(GRO) (e.g.,soils, sediments, and solid waste) This method is designed for use on samples containing various concentrations of VOCs or GRO and oily materials. Sample collection and preparation is performed using the procedures described here, and sample analysis is performed using the aqueous purge and trap procedures. Refer to SOP V005, or T008. The parameters listed in Table 1 can be extracted using this method.

2.2 Procedures are included for preparing high concentration samples, those containing VOC level>100 ug/kg and also oily wastes.

2.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

3. Interferences

3.1 Impurities in reagents and on glassware account for the majority of contamination problems. Reagents and materials must be demonstrated to be free from contamination by the analysis of method blanks.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum of the vial during shipment. Therefore a trip blank should be prepared and shipped with all coolers.

3.3 Contamination can also occur from carryover after highly contaminated samples. When practical samples suspected of being highly contaminated should be followed by a cleaning blank to eliminate this possibility.

Whenever carryover is suspected the analyst must rerun all samples in question to verify any reportable results.

4. Safety

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard. Exposure should be minimized by wearing solvent-resistant gloves, a lab coat and safety goggles when handling standards and samples and by working in a fume hood. **The following compounds have been tentatively classified as know or suspected human carcinogens: Benzene, Carbon tetrachloride, Chloroform, 1,4-Dichlorobenzene, 1,2-Dichloroethane, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, 1,1,2-Trichlorethane and Vinyl Chloride.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

5. Method Summary

5.1 This method is suitable for the analysis of samples suspected to contain high concentration of VOCs >100 ug/kg, or those that arrive in the laboratory field preserved with methanol.

5.2 When a bulk sample is collected that is suspected to contain high concentrations of VOCs, a portion of that sample should be transferred to methanol as soon as possible to minimize VOC loss. An aliquot of this extract is then analyzed following SOP V005. Because this procedure involves opening the container and removing a portion of the soil some volatile constituents may be lost.

5.3 It is preferred to have a sample preserved with methanol upon collection in the field. Approximately 10 gms of sample is collected into a pre-weighed vial with a septum-sealed screw-cap that contains 10mL of purge and trap grade methanol. At the time of analysis, surrogates are added to the vial, then an aliquot is removed and analyzed following the appropriate SOP.

5.4 Samples that are composed of oily or solvent wastes may be diluted using this method if they are miscible with methanol.

6. Sample Collection , Preservation, and Handling

6.1 Preparation of vials

6.1.1 Bulk samples should be collected in clean 2oz septa jars

6.1.2 Vials for field preservation should be prepared as follows, all information should be recorded in the Methanol Preservation Logbook:

6.1.2.1 Calibrate the balance according to SOP M009. This needs to be done only once per day.

6.1.2.2 Record the Manufacturer and lot# for the methanol in the Methanol Preservation Logbook (Figure 1).

6.1.2.3 Print labels with the appropriate vial ID. The vial ID + Date prepared +vial # (i.e. 021500-1). Note each vial should have a unique ID. **Note: It is important to affix all labels prior to weighing the vials.**

6.1.2.4 Weigh the vial with the cap on and record the weight.

6.1.2.5 Using the automatic dispenser carefully add 10mL of methanol. (Unless another volume is required)

6.1.2.6 Record the volume of Methanol in Column (M) of the preservation logbook.

6.1.2.7 Reweigh the vial with the cap and record the weight in column (A) of the logbook.

6.2 Sample Collection.

6.2.1 Samples should be collected in accordance with the appropriate sampling plan. As always when collecting samples for Volatile analysis care must be taken to minimize loss of volatile components. Refer to Attachment A for guidelines in collecting solid samples

6.2.2 After collection samples should be stored on ice at 4 °C until they are returned to the lab.

6.2.3 Upon receipt at the laboratory the sample should be logged in in accordance with SOP L001.

6.2.4 After the vials have received a laboratory number and have had the labels verified to be correct the samples must be reweighed.

6.2.5 Locate the appropriate vial ID and record the AEL lab ID in the Methanol Preservation Logbook.

6.2.6 Reweigh the container with the cap on and record the weight of the container and sample in column (B) of the methanol preservation logbook.

6.2.7 Calculate the weight of the sample:Column(B-A) and record the weight in Column (C).

6.2.8 Calculate the ratio of soil/MEOH: Column(C/M). If the ratio of soil/methanol is <0.75 or >1.25 notify the QC officer or the Laboratory Director.

6.2.9 Record the weight of the sample on the vial label for analyst use.

6.3 Sample Handling and Shipment

All samples for analysis should be cooled to approximately 4 °C, packed in appropriate containers, and shipped to the laboratory as described in the sampling plan.

6.4 Sample Storage

6.4.1 All samples should be received cold 0°-6°C; the temperature should be recorded upon receipt. Refer to SOP QA-L001 for proper cooler handling instructions.

6.4.2 Samples should be refrigerated at (4°C+/- 2°C) as soon as possible after they are received at the lab. Refer to SOP QA-L001 for laboratory receipt and log-in procedures.

6.4.3 All samples must be analyzed within 14 days of collection.

7. Definitions

7.1 I.S.= Internal standard

7.2 ml= milliliter

7.3 g= gram

7.4 CAS= Chemical Abstract Service

7.5 VOA= Volatile Organic Analysis

7.6 °C= degrees Celsius

7.7 µl= microliter

7.8 SOP= Standard Operating Procedure

7.9 AEL= Analytics Environmental Laboratory

7.10 LCS= Laboratory Control Sample

8. Apparatus and Materials

8.1 40 ml VOA vials with Teflon™ lined septa and screw caps purchased pre-cleaned.

8.2 Balance capable of accurately weighing 0.01 g. Calibrated according to QA-M009.

8.3 Metal spatulas.

8.4 Syringe - 1.0 ml, gastight.

8.5 Volatiles Soil Prep. Logbook, or Gasoline/GRO Soil Prep logbook.

8.6 Volatiles Standard Prep logbook, or TPH Standards Prep logbook.

8.7 Repipetter - Baxter Top Slider Dispenser or equivalent capable of delivering incremental milliliter to 10 ml.

9. Standards and Reagents

9.1 Methanol - Purge and Trap grade or equivalent

9.2 Surrogate solution - Refer to QA-V001 or-T002 for standards preparation

9.3 Matrix spike solution - Refer to QA-V001 or QA-T002 for standards preparation

10. Procedure

10.1 Samples that are methanol extracted at the laboratory

10.1.1 Information pertinent to volatile organic solid/oil sample preparation is recorded in a bound paginated laboratory notebook. Each entry contains the following information: date, analyst initials, AEL's sample #, weight of sample (g), volume of surrogate (ml), surrogate #, volume of matrix spike (ml), matrix spike #, volume of methanol (ml) used with manufacturer and lot number and any comments relevant to the sample (*i.e.* matrix is other than soil, strong odor, etc.).

10.1.2 Determine necessary Quality Control.

10.1.2.1 A method blank must be performed for each analytical batch (twenty samples or daily). The method blank consists of 10.0 ml of methanol that is extracted and analyzed in the same manner as the samples. The blank is labeled as follows: MBMMDDY, where;

MB indicates a method blank for medium soils
MM is the two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the year

In addition method blanks for total gasoline end with a GAS, gasoline range organics with a GRO, and 8260 blanks with an instrument identifier.

10.1.2.2 One matrix spike and matrix spike duplicate must be performed for every twenty samples or within one month period. Fill out the QC Summary (Figure 2) to determine if it is necessary to perform a matrix spike and matrix spike duplicate. Matrix spike and matrix spike duplicates are prepared by spiking an aliquot of a sample with 10uL of spiking solution. When analyzing full list spikes 10 uL of standard solutions are added to the sample syringe after the methanol extract has been added. The spiked samples are then prepared as all other samples. The matrix spike is labeled with the sample number followed by MS and the matrix spike duplicate uses the suffix MSD. Refer to the appropriate standard SOP for spiking solution.

10.1.2.3 One LCS is extracted with every preparation batch. The LCS is prepared by spiking 10 g clean sand with 10.0 ml of methanol with 10uL of spiking solution, and 10uL of surrogate solution. Refer to the appropriate standard SOP for spiking and surrogate solutions. The LCS is labeled LSMMDDY where;

LS indicates a solid laboratory control sample
MM is the two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the year

10.2 Sample Extraction - modified method

10.2.1 **All work involving Volatile Organics must be performed in the hood in the instrument laboratory to prevent contamination of the sample.**

10.2.2 Label a separate 40 ml VOA vial with the sample identifier for each blank, sample, matrix spike, matrix spike duplicate, and LCS being prepared.

10.2.3 Record the date, your initials and the sample numbers including blank, matrix spike, matrix spike duplicate, and LCS, as needed, in the appropriate soil prep logbook.

10.2.4 Calibrate the balance as per QA-M009.

10.2.5 Tare the uncapped vial for the first sample to be prepared. Quickly weigh 10 g of sample into the tared vial. Recap the vial. Record this weight to the nearest 0.01 g. The sample consists of the entire contents of the container. **Do not decant any liquids.** Mix the

contents prior to weighing the sample aliquot. If the sample is a sludge use 5 g. If the sample is an oily soil or non-aqueous liquid waste, use 1 g.

10.2.6 Add 10.0 mL of methanol to each blank and sample using the repipetter. Add 10.0 mL of methanol to each matrix spike, matrix spike duplicate, and LCS.

10.2.7 Add 10uL of the appropriate surrogate spiking solution to each vial through the septum.

10.2.8 Add 10uL of the appropriate matrix spiking solution to each vial designated as a matrix spike, matrix spike duplicate, or LCS through the septum. When analyzing full list spikes 10 uL of standard solutions are added to the sample syringe after the methanol extract has been added.

10.2.10 Shake the vials for two minutes. Allow samples to settle for at least one hour

10.2.11 Samples are now ready for analysis. Refer to the appropriate SOP for determinative analytical procedures.

10.2.12 No more than 100μl aliquot of the methanol extract is added to 5 mL of reagent water and analyzed. Lesser aliquots may be analyzed for more highly contaminated samples.

10.2.13 Extracts may be stored in the Volatiles Soil refrigerator or the Instrument Lab refrigerator at 4°C ±2°C for up to 14 days from the time the sample was collected.

10.3 Preparation of field preserved samples

10.3.1 Verify that sample weight has been determined. See section 6.2.

10.3.2 To all sample and spikes add 10uL of the appropriate surrogate spiking solution to each vial through the septum.

10.3.3 Shake the vials for two minutes. Allow samples to settle for at least one hour

10.3.4 Samples are now ready for analysis. Refer to the appropriate SOP for determinative analytical procedures.

10.3.5 No more than a 100μl aliquot of the methanol extract is added to 5 mL of reagent water and analyzed. Lesser aliquots may be analyzed for more highly contaminated samples.

10.3.6 Extracts may be stored in the Volatiles Soil refrigerator or the Instrument Lab refrigerator at 4°C ±2°C for up to 14 days from the time the sample was collected

11. Quality Control

11.1 Per Sample

11.1.1 Surrogates are added to every sample to test the efficiency of the extraction procedures. Percent recoveries are calculated by the analyst. (Refer to the appropriate determinative method SOP for acceptance ranges.) If recoveries are outside the pre-determined acceptance ranges, the extract must be reanalyzed or the sample must be re-extracted.

11.2 Per Sample Batch (20 samples or daily)

11.2.1 A method blank must be prepared with every sample batch. A blank consists of 10.0 ml of methanol and 10uL of surrogate that is carried throughout the extraction procedure. Blanks are analyzed prior to the appropriate sample batch to monitor interferences or contaminants. If the blank is found to contain more than the reporting limit of any compound, the sample batch associated with it must be re-extracted. If insufficient sample exists, the contaminant will be flagged with a "B" qualifier.

11.2.2 A matrix spike and matrix spike duplicate are performed per batch of 20 samples or within a one month period. If spike recoveries or the relative percent difference are outside of the laboratory acceptance limits the matrix spike or matrix spike duplicate must be reanalyzed or re-extracted to determine the cause. Recoveries and relative percent difference limits are listed in the appropriate determinative SOP.

11.2.3 A laboratory control sample (LCS) is performed per batch of 20 samples or daily. If spike recoveries are outside of the laboratory acceptance limits the LCS must be reanalyzed or re-extracted to determine the cause. If re-extraction is performed, all associated samples and blanks must be re-extracted as well.

12 Pollution Prevention and Waste Management.

12.1 See SOP W001 for handling of waste and unused portions of samples.

TABLE 1

ANALYTES	CAS NO.
Acetone	67-64-1
Acrolein	107-02-8
Acrylonitrile	107-13-1
Allylchloride	107-05-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochlorobenzene (Surrogate)	
Bromochloromethane (I.S. or target)	74-97-5
Bromodichloromethane	75-27-4
4-Bromofluorobenzene (Surrogate)	460-00-4
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone	78-93-3
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon Disulfide	75-15-0
Carbon Tetrachloride	56-23-5
Chloroacetonitrile	107-14-2
Chlorobenzene	108-90-7
Chlorobenzene-d5 (I.S.)	
1-Chlorobutane	109-69-3
Chlorodibromomethane	124-48-1
Chloroethane	75-00-3
2-Chloroethyl Vinyl Ether	110-75-8
Chloroform	67-66-3
Chloromethane	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
trans-1,4-Dichloro-2-butene	110-57-6
Dichlorodifluoromethane	74-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,2-Dichloroethane-d4 (Surrogate)	
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
1,1-Dichloropropanone	513-88-2
cis-1,3-Dichloropropene	10061-01-5

trans-1,3-Dichloropropene	10061-02-6
2,2-Dichloropropane	590-20-7
1,1-Dichloropropene	563-58-6
Diethyl ether	60-29-7
1,4-Difluorobenzene (I.S.)	540-36-3
Ethylbenzene	100-41-4
Ethyl methacrylate	97-63-2
Hexachlorobutadiene	87-68-3
Hexachloroethane	67-72-1
2-Hexanone	591-78-6
Isopropylbenzene	98-82-8
p-Isopropyltoluene	99-87-6
Methylacrylate	96-33-3
Methylacrylonitrile	126-98-7
Methyl t-Butyl Ether	1634-04-4
Methylene Chloride	75-09-2
Methyl Iodide	74-88-4
Methyl methacrylate	80-62-6
Nitrobenzene	98-95-3
4-Methyl-2-Pentanone	108-10-1
Naphthalene	91-20-3
2-Nitropropanone	79-46-9
n-Propylbenzene	103-65-1
Propionitrile	107-12-0
Pentachloroethane	76-01-7
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
Toluene-d8 (Surrogate)	2037-26-5
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl Acetate	108-05-4
Vinyl Chloride	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3
Gasoline Range Organics	

Figure 1
Methanol Preservation Logbook #1
(VOCs,VPH,GRO,8015GS)

Manuf.+Lot # of MEOH: B+J PFT BY114

NOTE: All weights include the cap of the vial .

Date Prepared	Vial #	Wt. Vial (g)	(M) Volume of MEOH (mL)	(A) Wt. Vial. + MeOH (g)	(B) Wt. Vial. +MeOH+Soil (g)	(C) (B-A) Wt. Soil(g)	Ratio of soil/MEOH (C/M) *	AEL Lab ID
092000	01	25.14	10mL	32.58				
	02	25.12		33.27				
	03	24.85		33.02				
	04	25.20		33.43	43.65	10.22	1.02	44338-1A
	05	25.15		33.31	44.83	11.52		44295-2-A
	06	25.04		33.40	44.52	11.12		44295-4-A
	07	25.05		33.31				
	08	25.25		33.48				
	09	25.03		33.19				
	10	24.98		33.19				
	11	24.94		33.07				
	12	25.21		33.45				
	13	25.07		33.35	44.98	11.63		44295-3-A
	14	25.21		33.56				
	15	24.97		33.24				
	16	25.04		33.29	33.15	-.14	-.01	44338-15
	17	25.06		33.31				
	18	25.11		33.31	42.89	9.58	.96	44337-3A
	19	25.15		33.35	43.57	10.22	1.02	44338-3A
	20	25.14		33.41	43.23	9.82	.98	44338-2A
	21	25.06		33.29				
	22	25.30		33.52				
	23	25.13		33.28				
	24	25.05		33.31	42.95	9.64	.96	44338-13A

* If ratio of soil/MEOH <0.75 or >1.25 Notify the Laboratory Supervisor

Figure 1 Cont.

PROCEDURE

Preparation of sample containers:

1: Calibrate the balance according to SOP M009. This needs to be done only once per day.

2: Record the Maufacturer and lot # for the Methanol

3: Print labels with the appropriate Vial ID. The Vial ID = Date Prepared+vial #
IE: 021500-1. Note: each vial should have a unique ID.

4: Weigh the vial with cap on and record the weight.

5: Using the automatic dispenser add 10mL MEOH. (Unless another volume of MEOH is required.)

6: Record the volume of MEOH in column (M).

7 Reweigh the vial with cap on and record the weight in column (A).

Upon return to the laboratory:

1: Be sure all vials have been given an AEL sample#.

2: Locate the appropriate Vial ID and Record the AEL Lab ID.

3: Reweigh the container with cap on and record the weight of the container in column (B).

4: Calculate the weight of the sample: Column(B-A) and record in Column (C).

5: Calculate The ratio of soil/MEOH : Column (C/M).

FIGURE 2

QC SUMMARY VOLATILE SOIL SAMPLES

Procedure:_____ Batch ID#_____

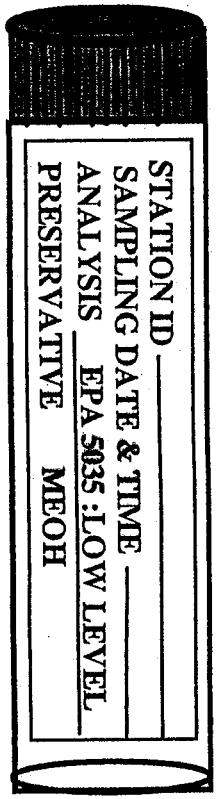
	SAMPLE #	DATE	BLANK	MS	MSD	LCS
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

Rev6 7/97

ATTACHMENT A

analytics

EPA 5035
VOC SOIL
SAMPLING GUIDE



195 COMMERCE WAY
PORTSMOUTH, NH 03801
800-929-9906
603-436-5111
603-430-2151fax

Step 1: Insert the Easy Draw Syringe into the appropriate slot on the Powerstop Handle.

Use one of the 10 gm positions:

-HVY=heavy
(dense clay)

-LT=light
(sandy soil)

-MED=medium
(all others)

Step 2: Remove the cap from the bottom of the syringe.

Step 3: Push the syringe into the freshly exposed soil surface. Remove syringe from handle.

Step 4: Extrude sample into vial by pushing the plunger down. (hold the vial at an angle to prevent splashing)

Step 5: Wipe the threads of the vial clean and cap.

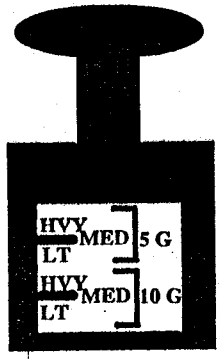
Step 6: Cool vial(s) to 4 °C

Step 7: Fill % Solids vial to top.

Each sample should have:

* 1-40 ml vial for High Level Soil Analysis and
* 1-40 ml vial for % Solid determination.

Please return the Blue Powerstop Handle to Analytics.



analytics environmental laboratory ~~inc.~~ LLC SLL 1/30/01
1680 Commerce Way
Portsmouth, New Hampshire 03801
603-436-5111/800-929-9906
Fax 603-430-2151
email: analytics@analyticslab.com

Laboratory Method for the Determination Of Volatile Organic Compounds - Capillary Column, GC/MS Detection

COPY

Approved By:

QA Officer: _____ Date: 11/2/99

Laboratory Director: _____ Date: 11/2/99

Revision History

Revision	Description of Change	Approval	Date

1. Title

Laboratory Method for the Determination Of Volatile Organic Compounds - Capillary Column, GC/MS Detection. EPA Reference: SW-846, Method 8260B
SW-846, Method 8000B
Federal Register, Method 624

2. Scope and Application

This method describes the technique used to determine volatile organic compounds in a variety of matrices. This method is applicable to most volatile organic compounds that have boiling points below 200°C that are insoluble or slightly soluble in water. Water soluble compounds are included in the method but quantitation limits are approximately five times higher because of poor purging efficiency. The parameters listed in Table 1 can be measured by this method. This method is restricted to use by or under the supervision of analysts experienced in the use of GC/MS and in the interpretation of mass spectra.

3. Safety

The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard. Exposure should be minimized by wearing safety goggles, solvent-resistant gloves and a laboratory jacket when samples or standards are handled, and by working in a fume hood. **The following compounds have been classified as know or suspected human carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, 1,2-dichloroethane, 1,2-dibromomethane, 1,1,2,2-tetrachloroethane, tetrachloroethene, 1,1,2-trichlorethane, trichloroethene, hexachlorobutadiene and vinyl chloride.** Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory, and should be read by any personnel prior to initiating this method.

4. Method Summary

The volatile compounds are introduced into the gas chromatograph (GC) by the purge and trap technique (QA-V009). Purged sample components are trapped in a tube containing sorbent materials. When purging is complete, the sorbent tube is heated and back-flushed with helium to desorb the trapped components to a capillary column. The oven holding the column is temperature programmed to separate the analytes which are detected with a mass spectrometer interfaced to the gas chromatograph. Quantitation is performed using internal standard technique. See Figure 1 for a method summary.

5. Definitions

5.1 ALS = Automated Liquid Sampler
5.2 °C = degrees Celsius
5.3 GC = Gas Chromatograph
5.4 GC/MS = Gas Chromatograph/Mass Spectrometer
5.5 LSC = Liquid Sample Concentrator
5.6 QC = Quality Control
5.7 SOP = Standard Operating Procedure
5.8 µl = microliter
5.9 ml = milliliter
5.10 ppb = parts per billion
5.11 IS/SS = internal standard/surrogate standard
5.12 eV = electron volt
5.13 µg = microgram
5.14 g = gram

5.15 kg = kilogram
5.16 L = liter
5.17 min = minutes
5.18 Rf = response factor
5.19 RSD = relative standard deviation
5.20 m/z = mass to charge
5.21 I = internal standard
5.22 S = surrogate standard
5.23 T = target compound
5.24 M = matrix spike compound
5.25 P = SPCC = system performance check compound
5.26 C = CCC = calibration check compound
5.27 Abs = absolute
5.28 AMU = atomic mass unit
5.29 sec = second
5.30 A/D = analog to digital
5.31 BFB = bromofluorobenzene
5.32 TCLP = Toxicity Characteristic Leaching Procedure
5.33 MS = Matrix spike
5.34 MSD = Matrix spike duplicate
5.35 PSI = pounds per square inch
5.36 ng = nanogram

6. Apparatus and Materials (equivalents may be used)

6.1 5 ml Luerlok Syringe. Rinse twice with reagent grade water before and after use.
6.2 Gastight fixed needle syringes: 2.5 ml, 1 ml, 500 µl, 100 µl, 50 µl, 25 µl, and 10 µl. Rinse three times with methanol before and after use.
6.3 50 ml plastic syringes with Luerlok tips (for rinsing purge cells).
6.4 40 ml VOA vials, pre-cleaned, with Teflon lined caps.
6.5 Autosampler

Tekmar ALS 2016 or equivalent
Tekmar LSC 2000 or equivalent
Tekmar Precept II or equivalent
Tekmar LSC 3000 or equivalent
Tekmar Automatic Sample Heater or equivalent
Trap: Supelco VOCARB 3000, #5, or equivalent
Purge/Desorb gas: Helium (Grade 5)

6.5.1 Method #1 LSC 2000 Inst:C

Standby:	35°C
Prepurge:	0.00
Sample:	35°C
Preheat:	0.00
Purge:	11.00 min
Dry purge:	5.00 min

6.5.2 Method #2- LSC 2000 Inst: I

Standby:	35°C
Prepurge:	0.00
Sample:	40°C
Preheat:	1.50 min
Purge:	11.00 min
Dry purge:	5.00 min

6.5.3 Method #1 and Method #2 LSC 2000 Inst: C&I

Desorb preheat:	250°C
Desorb:	2 min at 250°C
Bake:	5.0 min at 260°C
BGB:	Off
BGB delay:	120 seconds
Autodrain:	Off
Valve:	100°C
Line:	100°C
Mount:	50°C
2016 Valve:	100°C
2016 Line:	100°C

6.5.4 Tekmar 3000 and Precept conditions (water method) 8260W

6.5.4.1 Sample transfer

Sweep needle time:	1.0 min
Syringe fill volume:	5 ml
Transfer sweep time:	0.5 min
Syrine rinse volume:	5 ml
Flush needle time:	2 min
Sweep lines time:	0.5 min
# syringe rinses:	2
Back flush time:	2 min

6.5.4.2 Sample purge

Purge ready temp:	30°C
Purge temp:	30°C
Sample heater:	Off
Purge time:	11 min
Dry purge time:	4 min

6.5.4.3 Sample desorb

GC start:	start of desorb
GC cycle time:	33 min
Desorb preheat:	245°C
Desorb time:	4 min
Desorb temp:	250°C
Glass rinse:	On
Glass rinse time:	2 min
Glass purge time:	2 min

6.5.4.4 Sample bake

Bake time:	8 min
Bake temp:	260°C
BGB:	Off
MCS bakeout temp:	260°C

6.5.4.5 Sample temperatures

Transfer line temp:	100°C
Soil line temp:	100°C
Needle temp:	60°C
Needle purge temp:	60°C
X5 valve temp:	100°C
Vial heater temp:	40°C
Line temp:	100°C
Valve temp:	100°C
Mount temp:	40°C

MCS line temp: 40°C

6.5.4.6 Configuration Precept

Standard 2 option: No
Standby flow: On
Trap turbo cool: Off
GC handshaking: On
GC type: User
User GC type: 31
Halt on bar code: No

6.6 Gas chromatograph: Instrument I Hewlett Packard 5890 Series II or equivalent equiped with a Restek column RTX-502.2, 105m, 0.53mm ID, 3.0µm df or equivalent.

6.6.1 Zone temperatures:

	State	Setpoint
Inlet A:	On	200°C
Inlet B:	Off	
Detector A:	On	200°C
Detector B:	On	220°C

Column head pressure: 30 kPa
Column flow: 25 ml/min Helium (Grade 5)
Make-up flow: 15 ml/min Helium (Grade 5)

6.7 Mass spectrometer: Hewlett Packard 5970 or equivalent.

6.7.1 Set acquisition mode: Scan

6.7.2 Scan aquisition:
EM voltage: varied (Abs.)
MS scan parameters: range 35-300 amu, using 70 eV
Threshold: varied
Sampling A/D: 4

6.8 GC Conditions

6.8.1 Oven equib time: 0.50 minutes

6.8.2 Purge valve settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.75	0.00	Yes

6.9 Oven Program BFB

6.9.1 Initial temperature: 100°C
Initial Time: 0.00 minutes

Level	Rate (°C/min)	Final Temperature (°C)	Final Time (min)
1	10.0	210	0.00 - 16.00

6.9.2 Solvent delay: approximately 2.0 minutes (after elution of solvent front)

6.10 Oven Program Standards and Samples

6.10.1 Initial temperature: 35°C
Initial time: 4.50 minutes
Rate

Level	Rate (°C/min)	Final Temperature (°C)	Final Time (min)
1	8.0	220	3.00-5.00

6.10.2 Solvent delay: approximately 2.00 (after solvent front but before first compound elution)

6.11 Vacuum gauge: 0.5 Torr

6.12 Ionization gauge: 10^{-5} or 10^{-6}

6.13 Gas chromatograph: Hewlett Packard 5890 or 5890 Series II or equivalent equipped with EPC and Restek column RTX-624, 30m, 0.25mmID, 1.0µm df or equivalent.

6.13.1 Zone temperatures:

	State	Setpoint
Inlet A:	On	200°C
Inlet B:	Off	
Detector A:	Off	
Detector B:	On	220°C

Column head pressure: EPC

Column flow: 1.0 ml/min Helium (Grade 5)

6.14 Mass spectrometer: Hewlett Packard 5970, 5971, or equivalent.

6.14.1 Set acquisition mode: Scan

6.14.2 Scan aquisition: EM voltage: varied (Abs.)
MS scan parameters: range 35-300 amu, using 70 eV
Threshold: varied
Sampling A/D: 4

6.15 GC Conditions

6.15.1 Oven equib time: 0.50 minutes

6.15.2 Purge valve settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
B	Off	0.75	0.00	No

6.15.3 Inlet Settings

Inlet A	Constant Flow	On	7.3 psi
Vaccum Comp		On	
Calculated Values	Oven Temp		35°C
	psi		7.3
	Flow		1.0mL/min
	Velocity		36.8cm/sec
	Column Length		30 m
	Split Flow		1.0 mL/min
	Split Ratio		1.00:1

6.16 Oven Program Standards and Samples

6.16.1 Initial temperature: 35°C
Initial time: 4-4.5 minutes
Rate

Level	Rate (°C/min)	Final Temperature (°C)	Final Time (min)
1	8.0	180	2

6.16.2 Solvent delay: approximately 1.6 minutes (after solvent front but before first compound elution)

6.17 Data system: Hewlett Packard Environmental Chemstation Software

6.18 Instrument Run Logbook

6.19 pH logbook

6.20 QC logbook
6.21 Maintenance logbook
6.22 Wide range pH paper, 1-14 pH

7. Standards and Reagents

7.1 Reagent water: distilled then deionized using a Sybron Barnstead three-canister filtering system, or equivalent.
7.2 Methanol: purge and trap grade, or equivalent.
7.3 Tuning standard: refer to QA-V001 for standards preparation.
7.4 Initial and calibration verification standards: refer to QA-V001 for standards preparation.
7.5 Calibration check standards: refer to QA-V001 for standards preparation.
7.6 Internal standards: refer to QA-V001 for standards preparation.
7.7 Surrogate standards: refer to QA-V001 for standards preparation.
7.8 Matrix and TCLP spiking standards: refer to QA-V001 for standards preparation.

8. Procedure

8.1 Instrument Run Logbook. All information pertinent to volatile analysis by GC/MS must be recorded in a permanently bound paginated notebook. At the top of the page the following is recorded: date, instrument ID, voltage and threshold. Each day starts its own page. Information is recorded in columns with the following headings: tube number, method, sample information (standard # or sample #), volume, dilution, comments, data file, and LB (who loaded the sample). Comments could include if the sample foamed or at what dilution the sample should be rerun. See Figure 2 for an example of the run log page.

8.2 Instrument tuning. 2µls of a Bromofluorobenzene standard (25 ng/µl) is spiked into 5 mls of reagent water and loaded into an ALS position. Start the instrument purging Method 1 (QA-V009).

8.2.1 Open MSTOP icon. Edit sample log table of the default sequence from sequence menu and type the following: vial number, data file, method, sample name and miscellaneous information. Click on OK and then save edits. Next, run sequence from the same menu.

8.2.2 When the run has completed, open "ENVDA #1" icon in program manager and load the data file. The chromatogram will appear in window #1. Double click on the peak with the cursor. The scan number and spectrum will be drawn in window #2. Evaluate BFB to the screen. Table 2 gives the criteria to be met to pass tune. These criteria must be demonstrated each 12-hour shift. Two or more spectra can be added together by typing the command ADD, or type SUB to subtract two scans from each other. These two spectra must be in the X and Y registers of the stack to perform these functions. **If background subtraction is used, it must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Subtraction of part of the BFB peak is not allowed.** Evaluate the passing tune to the printer. Label the hardcopy and run log with initials and what scans were used to pass tune. File the printout in the volatiles QC notebook. If the tune fails, a hard tune may need to be performed.

8.3 When using the VOCARB 3000 trap, the amount of thermal decomposition products formed must be routinely tracked by daily monitoring of the formation of chloromethane and bromomethane. A daily check standard containing surrogates , internal standards, and 20 µg/L bromoform must be analyzed prior to the analysis of the daily check standard. If levels of chloromethane or bromomethane exceed the reporting limit, the trap may be too contaminated with salts or tightly bound contamination for analysis to continue. The trap must be replaced and the system recalibrated.

8.4 Initial Calibration. Sample analysis can only begin once an acceptable multi-point calibration has been analyzed. Calibration standards are prepared at the time of loading into purge cells by diluting the standard in reagent water. The listed dilutions are from a 10µg/ml standard (QA-V001). Refer to Table 3 for initial calibration standard preparation.

8.4.1 The procedure to load a standard will be the same for all concentrations. Load samples according to QA-V009. One calibration standard should contain each analyte at the concentration used for the reporting limit.

8.4.2 The method is named by incorporating the date the calibration was run as follows: V8MMDDYX where;

V is the designation for analysis by GC/MS
8 is the designation for 8260
MM is a two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the year
X is the designation for the Instrument ID

This is done by saving the previous 5-point using the new name. Calibration standards will be quantitated automatically upon completion of the analytical run. Load the data file of the first standard in the 5-point. QEDIT quant result and manually integrate any individual compounds which are improperly integrated. Save any changes to the quant report. **Reprint the quant report if any manual integration was performed. Date and initial the hardcopy and maintain in the volatiles QC notebook.**

8.4.3 Under Init Cal in the main menu, go to "update levels." Recalibrate using Calib Level ID for the first standard of the 5-point. Replace the responses. Update the retention times and ion ratios using the midpoint of the calibration. Repeat the above procedures for each standard of the 5-point. Alternatively, all levels can be updated at once using "Quick levels update". Save any changes to the method.

8.4.4 Evaluate the initial calibration to the printer. This will print out the response factors and the RSD. The RF is calculated as follows:

$$RF = (A_X C_{is})/(A_{is} C_X)$$

A_X = Area or height of the characteristic ion for the compound being measured
A_{is} = Area or height of the characteristic ion for the specific internal standard
C_{is} = Concentration of the appropriate internal standard
C_X = Concentration of the compound being measured

8.4.5 The average RF is calculated for each compound using the five RF from the initial calibration. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum mean response factor. Compounds and acceptable criteria are listed in Table 4.

8.4.6 RSDs are also listed on the initial calibration evaluation report. The RSD is calculated as follows:

$$RSD = SD/RF_X * 100\%$$

RSD = Relative standard deviation
RF_X = mean of Rfs for a compound from the initial calibration
SD = standard deviation of the Rfs for a compound from the initial calibration

8.4.7 The RSD should be less than 15% for each compound. However, the RSD for each Calibration Check Compound (CCC) must be less than 30%, see Table 4 for a listing of CCCs. If the RSD of any compound is 15% or less an average response factor can be used

for quantitation. If the RSD is greater than 15%, a calibration curve may be used to quantitate. Alternatively, if the mean of the RSD values for all analytes in the calibration is less than or equal to 15%, the average response factor can be used for all analytes. If neither of these conditions are met, a calibration curve must be used. If a linear calibration is used, the correlation coefficient must be $r \geq 0.995$($r2 \geq 0.990$) to meet acceptance criteria. For a quadratic fit, the coefficient of determination must be 0.995 or greater to meet acceptance criteria and **six** calibration levels are required. **It is never acceptable to force a calibration curve through the origin nor to include the origin (0,0) as a sixth calibration point.**

8.4.8 If Rfs and RSDs criteria are acceptable, file the initial calibration printout and the standard quantitation reports in a ring binder. If criteria fail, correct source of problem, rerun the calibration and evaluate the new calibration for compliance.

8.4.9 Before a multipoint calibration is accepted, a second source standard at the midpoint concentration is analyzed to verify the calibration. The analytes of interest must fall within ± 20% of the expected concentrations for the calibration to be valid for that analyte.

8.5 GC/MS calibration verification.

8.5.1 The initial calibration for each analyte must be verified **once every 12 hours**. This is accomplished by analyzing a calibration standard that is near the midpoint concentration for the working range of the GC/MS.

8.5.2 The calibration verification, as well as the calibration check, are standards that are analyzed near the midpoint concentration for the working range of the GC/MS. They are prepared in an identical fashion. Spike a 5ml aliquot of reagent water with the appropriate amounts of standards (Table 2) for a 20ppb concentration and load on the Tekmar. Begin purging. Set up and run sequence. When sample has finished running, evaluate the data file as continuing calibration to the printer.

8.5.3 Check the calibration to ensure that the system performance check compounds are met (Table 4). CCCs listed in Table 4 are also used to verify the calibration. The percent difference is calculated by the HP software using the following equations;

$$\%\text{Difference} = (Rf_V - \overline{Rf}) / \overline{Rf} * 100$$

Rf_V = Response factor of verification standard

$\overline{Rf}$ = Mean response factor from initial calibration

$$\%\text{Drift} = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theoretical Concentration}} * 100$$

Use percent difference when performing average Rf calibration; use percent drift when calibrating with a curve. If the percent difference or percent drift for each CCC is less than 20%, the initial calibration is assumed to be valid. If any criterion is not met for any one CCC or SPCC, the system must be evaluated, and corrective actions must be taken before sample analysis begins. It may be necessary to analyze a new 5-point calibration.

8.5.4 The internal standard responses and retention times in the calibration check standard or calibration verification standard must be evaluated after data acquisition. If the retention time for any internal standard changes by more than 0.5 minutes from that in the mid-point standard level of the current initial calibration, the system must be inspected for malfunctions and corrections must be made. The minimum internal standard area must be at least 50% to a maximum area of 200% from that in the mid-point standard level of the current initial calibration. Once it has been determined that all criteria are compliant, a

hardcopy of the "continuing calibration report" and the quantitation report are filed in the volatiles QC notebook.

8.6 A method blank consisting of a 5.0ml aliquot of reagent water spiked with IS/SS must be analyzed **once every 12 hours** to ensure that the GC/MS system is free of contamination. If the method blank is contaminated by compounds above the reporting limit corrective action must be taken. Sample analysis cannot begin until an acceptable blank has been analyzed.

8.7 Sample Analysis.

8.7.1 It is highly recommended that the samples be screened. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

8.7.2 Rinse a 5ml Luerlok syringe two times with reagent water and once with the sample if loading 5mls. If a dilution of a sample is being loaded, rinse twice with reagent water then fill with the appropriate volume of reagent water. All analysis will be based on a 5ml volume of sample, water, or some combination of the two. Refer to Table 5 for common dilutions performed. When appropriate dilution is loaded into the syringe, spike the sample with 5µls of the IS/SS mixture (20 µg/ml) and load the sample on the Tekmar according to QA-V009. TCLP extracts are loaded at a minimum dilution of 1:10. Check the pH of the water samples using wide range pH paper and record as < or >2 in the sequence logbook.

8.7.3 If the initial analysis of a sample has a concentration of analytes that exceeds the calibration range, the sample must be reanalyzed at a dilution. The run immediately after the saturated sample should be looked at closely to determine if there has been any cross contamination. A reagent water blank should be run in the position where the saturated sample was previously. If the blank analysis is still not free of interferences, a 1:1 methanol/water blank should be analyzed. If problem persists, replace the tube.

8.7.4 All dilutions should keep the previously saturated peaks in the upper half of the calibration range.

8.8 Quantitative Analysis

8.8.1 All standards, blanks, samples and QC samples are quantitated using the initial calibration response factor or curve.

8.8.2 Quantitation is performed using the internal standard technique.

8.8.3 When MS response is linear (%RSD <15%), and the average response factor is selected; calculate the concentration of each identified analyte in the sample as follows:

Aqueous samples: concentration (µg/L) = $\frac{(A_x)(IS)}{(A_{is})(RF)} \times \frac{(V_N)(D)}{(V_o)}$

A_x = Area or height of characteristic ion for compound being measured.
IS = Concentration of internal standard injected (µg/L).
A_{is} = Area or height of characteristic ion for the internal standard.
RF = Mean response factor for compound being measured from the initial calibration.
V_o = Volume of sample purged (µl).
V_N = Nominal volume (5000 µl).
D = Dilutions performed other than those at instrument

Solid samples: concentration (µg/kg) = $\frac{(A_x)(IS)(V_t)(V_N)}{(A_{is})(RF)(V_i)(W_s)(DRY)}$

A_x, IS, A_{is}, RF, V_N = same as for aqueous sample
V_t = Volume of total extract (L) (usually 0.01L, unless problematic matrix).

V_i = Volume of extract purged (µl).
W_s = Weight of sample extracted (kg).
DRY = % dry weight of sample/100, or 1 for wet-weight basis.

8.8.4 **If the RSD is not <15%, a calibration curve may be used.**

8.8.5 In calculating sample concentrations by the internal standard method, the regression equation is rearranged to solve for the concentration of the target analyte (C_s), as shown below.

$$C_s = \left[(A_s C_{is}) / A_{is} - b \right] / a$$

A_s = Area or height of the peak for the target analyte in the sample
A_{is} = Area or height of the peak for the internal standard
C_s = Concentration of the target analyte in the calibration standard
C_{is} = Concentration of the internal standard
a = Slope of the line (also called the coefficient of C_s)
b = The intercept

8.8.6 Non-linear calibration
In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen should have no more than four parameters, i.e., if the model is polynomial, it may be no more than third order, as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

8.8.7 Target compounds are identified qualitatively based on retention time and relative m/z abundances. The relative retention times of the sample compounds must be within 0.06 relative retention time units of the standard compounds. Furthermore, relative abundances of the compound's key ions must agree with 30 percent of those of the standard's. Sample chromatogram and peak spectra can be analyzed and edited using the "Qedit" option in the "Quant" menu. "False positive" peaks are Q-deleted.

8.8.8 Nontarget compounds may be identified using the NIST library (nbs75k.l). The nontarget peaks can be searched individually or a Library Search Compound (LSC) report can be generated. The LSC report is used to search all peaks in the chromatogram that were not identified by the quantitation database search algorithm. Library search strategies are set by the analyst as well as integration parameters. They may be quantitated using the nearest internal standard as in the equation below.

$$\text{Concentration} = \frac{(As)(Cis)(Vt)}{(Ais)(Vo)(D)}$$

where:
As= Area of the characteristic m/z for the parameter to be measured.
Ais=Area of the characteristic m/z for the internal standard.
D= (100 - per cent moisture/100)for solid samples to be reported on a dry weight basis. Use 1 for water samples or solid samples that are reported on a wet weight basis.
Vt= Dilution.

Cis=Amount of Internal Standard added to each extract (µg/L).
Vo= Volume of water extracted (L), or weight extracted (kg).

Alternatively, the compound may be reported as "present but not calibrated for".

8.9 Matrix spike, matrix spike duplicate and laboratory control sample preparation. 5µL of the 20µg/ml internal/surrogate standard mixture and 20µL of the 5µg/ml matrix spike solution is added to a 5ml aliquot of reagent water or sample.

8.10 TCLP matrix spikes - 5 µl of the 20 µg/ml IS/SS mixture and 25 µL of the 5 µg/ml TCLP matrix spike solution are added to 4.5 ml of reagent water and 500 µL of the TCLP extract.

9. Quality Control

9.1 Every 12 hours

9.1.1 The GC/MS system must be tuned to meet the BFB specifications (Table 2).

9.1.2 A calibration verification must be analyzed and it must meet the SPCC criteria and the CCC criteria listed in Table 4.

9.1.3 A method blank is analyzed after calibration verification and prior to sample analysis. Blanks are analyzed to monitor interferences or contaminants. If a blank is found to contain more than the reporting limit of any compound, another blank must be analyzed. If more than 20 client samples are analyzed per 12 hour period, another blank must be analyzed

9.2 Per Day

9.2.1 A 20µg/L bromoform standard containing internal and surrogate standards is analyzed. Levels of chloromethane and bromomethane must not exceed the reporting limit. If levels exceed the reporting limit a new trap must be installed

9.2.2 A laboratory control sample is performed every 20 samples of the same matrix or per day. The percent recovery of each spiked compound is calculated as in 9.3.2 below. Acceptance criteria are listed in each Instrument method.
Any unacceptable recoveries must be documented and the source of the problem corrected. The laboratory control sample is labeled: LMMDDYX, where;

L indicates a laboratory control sample
MM is a two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the year
X is the Instrument designator

9.3 Per Sample

9.3.1 5µl of the 20µg/ml internal standard is added to each soil sample prior to purging. For aqueous samples, 5 µL of the 20 µg/ml Internal/Surrogate standard mix is added to each sample prior to analysis, including blanks, matrix spikes and laboratory control samples. The minimum internal standard area must be at least 50% to a maximum area of 200% from that in the mid-point standard level of the current initial calibration.

9.3.2 Surrogates are added to each aqueous sample and standard prior to purging. Refer to QA-V002 and QA-V003 for solid samples. Refer to each Instrument method for the acceptance limits for each surrogate used. When a surrogate is outside the acceptance limit, the sample must be re-analyzed to determine if a matrix effect is the cause.

9.3.3 Surrogate recoveries are charted in groups of 20. This information is used to monitor trends that may indicate when instrument maintenance is necessary. The following conditions are considered out of control events:

- Any one point outside control limits
- Any three consecutive points outside warning limits
- Any six consecutive points such that each point is larger (or smaller) than its immediate predecessor

- Any eight consecutive points on the same side of the centerline

9.4 Per Sample Batch (20 samples or 8 hour shift)

9.4.1 A matrix spike and matrix spike duplicate is performed every 20 samples or per month. The percent recoveries of each spiked compound are calculated as below;

$$\text{Percent Recovery} = \frac{[(\text{spike result } (\mu g/L) - \text{native result } (\mu g/L)] * 100}{\text{amount spike added } (\mu g/L)}$$

9.4.2.1 Acceptance limits for spike recoveries are listed in each Instruments analytical method.

9.4.2.2 Relative percent differences (RPD) are calculated for each spike compound.

$$RPD = \frac{(\text{MS \% recovery} - \text{MSD \% recovery}) * 100}{[(\text{MS \% recovery} + \text{MSD \% recovery})]/2}$$

9.4.2.3 Acceptable RPDs are listed in each Instruments analytical method.

9.4.2.4 If either the percent recoveries or RPDs are not within the laboratory acceptance limits, it may be necessary to reanalyze the sample and spikes. Analysis of a calibration verification standard is performed to verify that the system is in control (every 12 hours) before any re-analysis is performed. If the system is in control and recoveries are poor upon re-analysis for both the MS and MSD, matrix effect is listed as the cause. Document the problem on the hard copy chromatograms and in the run logbook.

9.5 Annually

9.5.1 Minimum detection limit studies are performed annually. A series of 7-10 standards are prepared and analyzed. MDL's are calculated using the following formula:

$$MDL=(t)(S)$$

where;

MDL= Minimum Detection Limit
t = Students' t value at the 99% confidence level
S = standard deviation

9.6 Validation Study
Validation studies are performed by each analyst for initial demonstration of capability measuring accuracy and precision.

10. Routine Instrument Maintenance

10.1 Periodic replacement of column is necessary, approximately annually, depending on sample throughput. Poor chromatography is one possible symptom. When a new column is installed, it must be conditioned according to manufacturer specifications. The jet separator connection should be plugged during this phase so no contamination is introduced into the mass spectrometer. Column replacement is documented in the column logbook.

10.2 Periodic replacement of the sorbent trap is necessary. Monitor system performance to determine when a new trap is needed.

10.3 Purge cells are rinsed, using reagent grade water, between analysis. If an unusually concentrated sample (>200ppb) is analyzed, it is recommended to bake the cell and then analyze a cell blank in the purge cell to check for possible carryover. If contamination persists after successive blank runs, replace the purge cell. Refer to QA-M001 for the cleaning of contaminated purge cells.

10.4 Filaments in the mass spectrometer need to be replaced when they burn out or break. There are two filaments on line.

10.5 Replacing filaments on the 5971.

10.5.1 To switch a filament, go under the MS Top icon. Pull down manual tune and load the correct tune file. Pull down MS parameters, and click on filament. Change to new filament and press OK. Before exiting, save the tune file.

10.5.2 Changing filaments. When both filaments are blown, they need to be replaced. Go under MS Top icon and pull down Diagnostics/Vacuum control. Highlight vent and follow instructions. Turn off oven, detectors and injectors. When venting, turn off main power supply and turn off pumps, the jet separator pump first. Refer to the 5971 maintenance manual on how to replace the filaments.

10.5.3 Pump down. Turn on main power supply, followed by the detector pump and the jet pump. Go under MS Top icon, and pull down Diagnostics/Vacuum control. Highlight pump down and follow instructions.

10.6 Replacing filaments on the 5970.

10.6.1 You can switch a filament by flipping the switch located on the outside of the analyzer.

10.6.2 Changing filaments. When both filaments are blown, they need to be replaced. Turn off heater/electronics. Turn off GC oven, injector A and detectors A & B. Wait for the detector temperatures to get safely below 100°C (roughly one hour) before turning off the ~line/pumps switch. Turn off the pumps, starting first with the jet separator pump. It is necessary to manually vent the system using the vent valve before removing the source. Refer to the 5970 maintenance manual on how to replace the filaments.

10.6.3 Pump down. Turn on main power supply, followed by the detector pump and the jet pump. Turn on injectors and detectors and set oven to 210°C.

10.7 It is a good idea to clean the source when the filaments are replaced. Refer to the maintenance manual for the correct sequence. Clean all parts with green rough paper and place the parts in methanol. Sonicate for 2-5 minutes and bake in oven to dry. Reassemble.

10.8 The helium tank is replaced when the tank pressure is below 500 psi.

10.9 The hydrocarbon in-line trap to the Tekmar 2000 or 3000 is replaced annually.

10.10 Occasional baking of the column may be helpful to facilitate the removal of contamination. The setting should not exceed the upper temperature limit of the column.

TABLE 1

Compound	Type	CAS #
Pentafluorobenzene	I	363-72-4
Dichlorodifluoromethane	T	75-71-8
Chloromethane	TP	74-87-3
Vinyl Chloride	TC	75-01-4
Bromomethane	T	74-83-9
Chloroethane	T	75-00-3
Trichlorofluoromethane	T	75-69-4
Diethyl ether	T	60-29-7
1,1,2-Trichloro-1,2,2-trifluoroethane	T	76-13-1
Acrolein	T	107-02-8
Acetone	T	67-64-1
1,1-Dichloroethene	TMC	75-35-4
Methyl iodide	T	74-88-4
Allyl chloride	T	107-05-1
Methylene Chloride	T	75-09-2
Carbon Disulfide	T	75-15-0
Acrylonitrile	T	107-13-1
Methyl-tert-butyl ether	T	1634-04-4
trans-1,2-Dichloroethene	T	156-60-5
1,1-Dichloroethane	TP	75-34-3
Vinyl acetate	T	108-05-4
2-butanone	T	78-93-3
Propionitrile	T	107-12-0
2,2-Dichloropropane	T	590-20-7
cis-1,2-Dichloroethene	T	156-59-2
Methacrylonitrile	T	126-98-7
Methylacrylate	T	96-33-3
Chloroform	TC	67-66-3
Bromochloromethane	T	74-97-5
Tetrahydrofuran	T	109-99-9
1,1,1-Trichloroethane	T	71-55-6
1-Chloro-butane	T	109-69-3
1,1-Dichloropropene	T	563-58-6
Carbon Tetrachloride	T	56-23-5
d4-1,2-Dichloroethane	S	17060-07-0
1,2-Dichloroethane	T	107-06-2
Benzene	TM	71-43-2
1,4-Difluorobenzene	I	540-36-3
Trichloroethene	TM	79-01-6
1,2-Dichloropropane	TC	78-87-5
Methylmethacrylate	T	80-62-6
Chloroacetonitrile	T	107-14-2
Bromodichloromethane	T	75-27-4
Dibromomethane	T	74-95-3
2-Nitropropane	T	79-46-9
1,1-Dichloropropanone	T	513-88-2

Compound	Type	CAS #
2-chloroethylvinylether	T	110-75-8
2-Hexanone	T	591-78-6
4-methyl-2-pentanone	T	108-10-1
cis-1,3-Dichloropropene	T	10061-01-5
d8-Toluene	S	2037-26-5
Toluene	TMC	108-88-3
trans-1,3-Dichloropropene	T	10061-02-6
Ethyl methacrylate	T	97-63-2
1,1,2-Trichloroethane	T	79-00-5
1,3-Dichloropropane	T	142-28-9
Tetrachloroethene	T	127-18-4
Dibromochloromethane	T	124-48-1
1,2-Dibromoethane	T	106-93-4
*d5-Chlorobenzene	I	3114-55-4
Chlorobenzene	TMP	108-90-7
1,1,1,2-Tetrachloroethane	T	630-20-6
Ethylbenzene	TC	100-41-4
p,m-Xylene	T	106-42-3
o-Xylene	T	95-47-6
Styrene	T	100-42-5
Bromoform	TP	75-25-2
Isopropylbenzene	T	98-82-8
1,1,2,2-Tetrachloroethane	TP	79-34-5
Bromofluorobenzene	S	460-00-4
1,2,3-Trichloropropane	T	96-18-4
trans-1,4-Dichloro-2-butene	T	110-57-6
n-Propylbenzene	T	103-65-1
Bromobenzene	T	108-86-1
1,3,5-Trimethylbenzene	T	108-67-8
2-Chlorotoluene	T	95-49-8
4-Chlorotoluene	T	106-43-4
tert-butylbenzene	T	98-06-6
1,2,4-Trimethylbenzene	T	95-63-6
Pentachloroethane	T	76-01-7
sec-butylbenzene	T	135-98-8
4-isopropyltoluene	T	99-87-6
1,3-Dichlorobenzene	T	541-73-1
1,4-Dichlorobenzene-d4	I	3855-82-1
1,4-Dichlorobenzene	T	106-46-7
n-butylbenzene	T	104-51-8
1,2-Dichlorobenzene	T	95-50-1
Hexachloroethane	T	67-72-1
1,2-Dibromo-3-chloropropane	T	96-12-8
Nitrobenzene	T	98-95-3
1,2,4-Trichlorobenzene	T	120-82-1
Hexachlorobutadiene	T	87-68-3
Naphthalene	T	91-20-3
1,2,3-Trichlorobenzene	T	87-61-6

TABLE 2

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%
50	95	15	40
75	95	30	60
95	95	100	100
96	95	5	9
173	174	0	<2
174	95	50	100
175	174	5	9
176	174	95	101
177	176	5	9

TABLE 3

Concentration	Internal Std. (20µg/ml)	Surr.Std. (10µg/ml)	Cal Std. No Gasses (10µg/ml)	Cal Std. Gasses (10µg/ml)	Vol. Reagent Water
2ppb	5µls	1µl	1µl	1µl	5ml
5ppb	5µls	2.5µls	2.5µls	2.5µls	5ml
20ppb	5µls	10µls	10µls	10µls	5ml
50ppb	5µls	25µls	25µls	25µls	5ml
100ppb	5µls	50µls	50µls	50µls	5ml

Note: Equivalent dilutions may be used.

TABLE 4

Calibration Check Compounds - % RSD must be less than 30% for initial calibration
- % difference or % drift must be less than 20% for calibration verification.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

System Performance Check Compounds: Minimum mean response factors (for initial and continuing calibration)

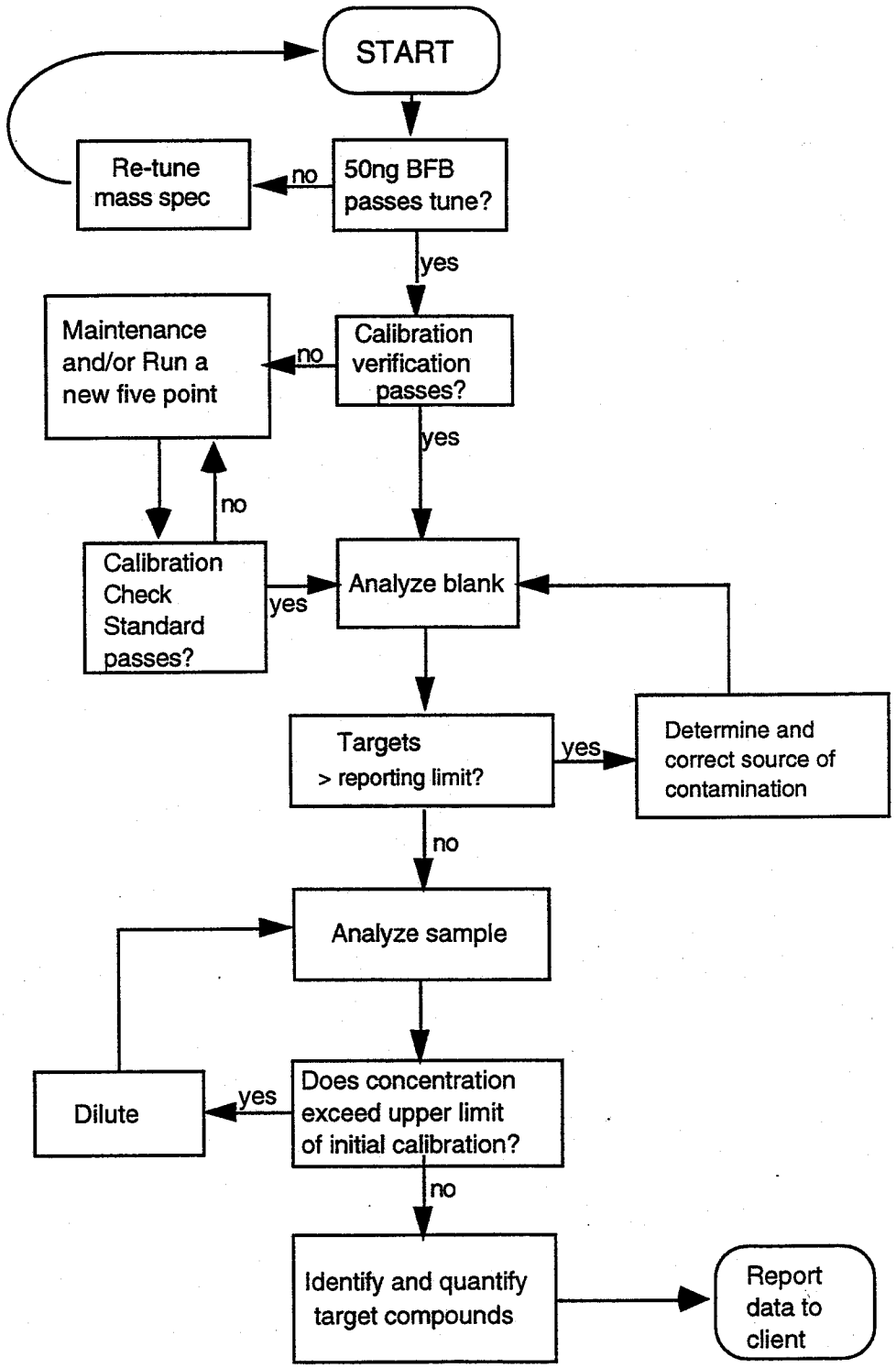
Choromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachoroethane	0.30

TABLE 5

Common dilutions

Dilution	Volume H20(µl)	Volume sample(µl)
1:1	0	5000
1:2	2500	2500
1:5	4000	1000
1:10	4500	500
1:25	4800	200
1:50	4900	100
1:100	4950	50
1:250	4980	20
1:500	4990	10

Figure 1
GC/MS Volatile Organics



FOSTER WHEELER ENVIRONMENTAL CORPORATION

STANDARD OPERATING PROCEDURES

PROJECT PROCEDURE NO. A-1

CATEGORY	PROJECT		
PROJECT TECHNICAL	SITE CHARACTERIZATION PROJECT		
TITLE		LEGEND R-INDICATES REVISION	DATE
HOLLOW-STEM AUGER DRILLING/SPLIT-BARREL SAMPLING/ DIRECT PUSH SAMPLING			Revision 0
	APPROVED BY		REPLACES ISSUE OF
	R. Pennifill		NEW

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PROPRIETARY INFORMATION

1.0 PURPOSE

The purpose of this Project Technical Procedure (PTP) is to describe the methods, the sequence of operations, and the equipment necessary to perform soil borings accompanied by split-barrel (or split-spoon) sampling. The purpose of such drilling and sampling is to determine the type, thickness, and certain physical and chemical properties of the soil, water and rock strata which underlie the site, and to install monitoring wells.

2.0 SCOPE

This PTP addresses the equipment and methods for the use of hollow-stem augers with split-barrel sampling in shallow subsurface exploration. The procedure, generally known as the Standard Penetration Test (SPT), involves driving a split-barrel sampler to obtain a representative soil sample and to measure the resistance of the soil to penetration by the sampler. This PTP does not purport to address the safety problems associated with the use of the described methods.

3.0 MAINTENANCE

The Site Characterization Project Geosciences Manager is responsible for updating this procedure. Approval authority rests with the Program Director and Project Manager of the Site Characterization Project. Suggestions for revision shall be submitted to the Project Quality Assurance Manager.

4.0 DEFINITIONS

Auger Plug - A disk attached to the bottom end of a drill rod, which follows the auger to the sample depth to prevent soil from entering the inside of the augers above the sample depth.

Anvil - That portion of the drive-weight assembly which the hammer strikes and through which the hammer energy passes into the drill rods.

Cathead - The rotating drum or windlass in the rope-cathead lift system around which the operator wraps a rope to lift and drop the hammer by successively tightening and loosening the rope turns around the drum.

Core Catcher - A ring with attached strips arched toward the center, composed of a plastic or steel inserted into the shoe to prevent soil from falling out of split-spoon. The core sample retainer catcher is also referred to as a "basket."

Direct Push Method – Methodology for advancing a drilling rod with sampler attachment using vibratory, hydraulic driven or percussion methods. The sampler is retrieved by removing the drilling rods.

Drill Rod - Steel rod used during borehole drilling for attachment of the split-spoon sampler or auger plug.

Drive-Weight Assembly - A device consisting of the hammer, hammer fall guide, the anvil, and any hammer drop system.

Hammer - That portion of the drive-weight assembly consisting of the 140 ± 2 lb. (63.5 ± 1 kg) impact weight, which is successively lifted and dropped to provide the energy that accomplishes the sampling and penetration. If an increased sample volume is required, a 300 ± 4 lb. (136 kg ± 2 kg) drive-weight assembly should be applied to a 3-inch outer diameter split-spoon sample.

Hammer Drop System - That portion of the drive-weight assembly by which the operator accomplishes the lifting and dropping of the hammer to produce the blow. A manual rope-cathead lift system or an automatic hydraulic hammer system should be utilized.

Hammer Fall Guide - That part of the drive-weight assembly used to guide the fall of the hammer.

Hollow-Stem Auger - A type of earth auger that has its flights welded onto an open pipe so that drill and sample rods may be passed through to the bottom of the boring. A cutter head with a hollow center is mounted on the lowermost section of the hollow drill stem. A plug with an attached bit is inserted into the hollow center of the cutter head to prevent soil from entering the inside of the augers.

N-Value - The blowcount representation of the penetration resistance of the soil. The N-value, reported in blows per foot, equals the sum of the number of blows required to drive the sampler over the sample depth interval of 6 to 18 in. (150 to 450 mm).

Shoe - Cutting head of a split-spoon sampler. It has a threaded annulus that is flush-mounted to the joined spoon halves.

Soil - Soil is sediments or other unconsolidated accumulations of solid particles that are produced by the physical and chemical disintegration of rock. Soil may contain organic matter.

Split-Barrel Sampler - A steel tube (i.e., generally carbon steel, but available in stainless steel for special conditions), split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into soil using a drive-weight mounted in the drill rod.

SPT - Abbreviation for Standard Penetration Test, a term by which engineers commonly refer to this method (Reference 4) of using split-barrel sampling to obtain the N-Value.

Water Table - A surface in an aquifer where groundwater pressure is equal to atmospheric pressure.

5.0 EQUIPMENT

Below is a list of equipment which maybe required to perform the procedures in Section 6.0. Other equipment may be necessary as dictated by field conditions and tasks outside the scope of this PTP.

Drilling Machine - Having sufficient torque and rpm range transformed to the drill rods, to rotate and force the auger to the desired depth or sufficient direct pressure, vibratory or pecussion. capability to advance a drill rod with sampler to the desired depth. The machine shall also be equipped with the accessory equipment needed to perform required sampling, such as split-barrel samplers, drill rods, and drive weight assembly.

Hollow-Stem Continuous Flight Augers - Pipe with spiral flights welded to outer diameters for corkscrew advancement by drilling. Augers are generally five feet in length. The cutting head is on the lead augers, with or without a center bit assembly. The inside diameter of the hollow-stem augers shall provide sufficient clearance for the sampling equipment and/or monitoring well materials, if applicable.

Split-Barrel Sampler - A standard split-barrel sampler used for performing SPTs is 2 inches O.D. and 1 3/8 inches I.D. This standard spoon typically is available in two common lengths providing either 20 inches or 26 inches internal longitudinal clearance for obtaining 18 or 24 inch long samples, respectively. The split-spoon sampler should be made of either stainless steel or carbon steel which is free from rust. In some cases the site-specific sampling plan may specify the use of a 3-inch O.D. split-spoon to allow for the acquisition of a larger sample. The blow counts obtained when using a 3-inch sampler may generate a value differing from the N-Value from the SPT. The use of liners and sample retainer baskets inside the split-barrel sampler is permitted, and should be noted in the field records.

Drive-Weight Assembly

Hammer and Anvil - The hammer shall weigh 140 $\pm$ 2 lb. for 2-inch OD split-spoon samples and standard penetration test (or 300 $\pm$ 4 lb. for 3-inch OD split-spoon samples) and shall be a solid rigid metallic mass. The hammer shall strike the anvil and make steel-on-steel contact when it is dropped. A hammer fall guide permitting a free fall shall be used. For safety reasons, the use of a hammer assembly with an internal anvil is encouraged.

Hammer Drop System - Rope-cathead, trip, semi-automatic, or automatic hammer drop systems should be used, providing that the lifting apparatus shall not cause penetration of the sampler while re-engaging and lifting the hammer.

Accessory Equipment - Accessories such as labels, sample containers, data sheets, and groundwater level measuring devices shall be provided in accordance with the requirements of the project.

6.0 DISCUSSION

6.1 Hollow-Stem Auger Drilling

1. The boring is installed by rotating and advancing the augers into the soil to the desired depth below grade.

2. The boring shall be advanced incrementally to permit intermittent or continuous sampling with the split-barrel sampler. Intervals and locations to be sampled are normally stipulated by the project engineer or project geologist, or as described in the site-specific sampling plan.

3. When taking soil samples for chemical analysis, the hollow-stem auger drilling shall be conducted utilizing a plug. Once the top of the interval to be sampled is reached the plug shall be extracted. Samples can then be taken by driving split-spoon samplers into the formation in advance of the auger. The plug should also be omitted in some cases, or a specially designed plug such as that denoted in Step 4 should be utilized.

4. When drilling below the water table, a plug designed to allow passage of formation water but not solid material should be used. This method should prevent blow back and plugging of the auger when the plug is removed for sampling.

5. Alternately, it may be necessary to keep the hollow-stem augers partially full of water, to prevent blowback and plugging of the auger. If water is added to the hole, it shall be potable and receive proper approval where necessary. In addition, the amount of water introduced, the amount recovered upon attainment of depth, and the amount of water extracted during well development should be recorded.

6. If gravely or hard material is encountered which prevents advancing the auger to the desired depth, augering should be halted and alternative drilling methods should be attempted.

At the option of the Field Operations Leader, or site geologist, a new boring can be attempted when resistant materials prevent the advancement of the auger. The original boring shall be properly abandoned and the new boring started a short distance away at a location determined by the site geologist.

6.2 Split-Barrel Sampling

1. Drill the hole to the desired sampling depth using the hollow-stem auger drilling method (or other approved method). In saturated sands and silts, withdraw the plug slowly to prevent loosening of the soil around the hole and maintain the water level in the hole at or above groundwater level.

2. The 2-inch O.D. split-barrel sampler should be driven with blows from a 140-lb. hammer falling 30 inches in accordance with ASTM D1586-84, Standard Penetration Test. The split-spoon is to be free from rust. When a 3-inch O.D. split spoon is used the same procedure is used except that the sampler shall be driven with a 300-lb. hammer falling 24 inches. The number of blows to drive the 300-lb hammer does not directly relate to the number of blows with the smaller split-barrel sampler and 140-lb. hammer.

3. Repeat this operation at intervals not greater than 5 feet in homogeneous strata, or as specified in the site-specific sampling plan.

   Record on the boring log form or field notebook the number of blows required to achieve each 6 inches of penetration or fraction thereof at the completion of the boring. The first 6 inches are considered to be a seating drive. The sum of the number of blows required for the second and third 6-inch interval of penetration is termed the penetration resistance, N. If the sampler is driven less than 18 inches, the number of blows for each six-inch increment or partial increment shall be recorded, along with the length of penetration. The boring shall be terminated when the specified depth is achieved, or, a total of 100 blows have been applied but less than 6 inches of advancement obtained.

4. Bring the sampler to the surface and remove both ends and one half of the split spoon so that the soil recovered rests in the remaining half of the barrel. Scren the sample using a FID or PID as appropriate. Describe collected soils as appropriate,

including approximate recovery (length), USCS and Burmister classifications (as required), composition, color, moisture, and any impacts noted.

5. Collect samples for laboratory analysis in the following order (as applicable): TCL VOCs, TCL SVOCs, TCL pesticides/PCBs, TAL metals and cyanide, and/or geotechnical parameters. The TCL VOC sample will be collected immediately upon opening the sample tube, from the middle of the soil interval. The remaining parameter samples will be homogenized in a stainless steel bowl prior to sample collection, and all samples will be collected using a dedicated clean stainless steel spatula or spoon.

6. Complete sample logs, labels, custody seals, and chain of custody forms. Record sample information in the field notebook.

7. Place analytical samplers in coolers for shipment and cool to 4°C.

6.3 Direct Push Sampling

1. The carbon steel sampler either unlined or containing a 2 or 3-foot clear acetate sample tube is advanced into the soil to the desired depth below grade.

2. The sampler shall be advanced incrementally to permit intermittent or continuous sampling. Intervals and locations to be sampled are normally stipulated by the project engineer or project geologist, or as described in the site-specific sampling plan.

3. Once the top of the interval to be sampled is reached the locking key shall be removed. Samples can then be taken over the sample interval by advancing the sampler into the formation.

4. Remove the drill rods with the sampler and detach the sampler from the rods. Remove the acetate sample tube if utilized, or else extrude the sample into a decontaminated sampling bowl.

5. If using a sample tube, place the acetate sample tube on clean polyethylene sheeting for inspection/sampling. Each sample tube will be inspected for the presence of groundwater. The tube will be cut lengthwise using a decontaminated utility knife, and the soil will be screened using a FID and PID as appropriate.

6. Describe collected soils as appropriate, including approximate recovery (length), USCS and Burmister classifications (as required), composition, color, moisture, and any impacts noted.

7. Collect samples for laboratory analysis in the following order (as applicable): TCL VOCs, TCL SVOCs, TCL pesticides/PCBs, TAL metals and cyanide, and/or geotechnical parameters. The TCL VOC sample will be collected immediately upon opening the sample tube, from the middle of the soil interval. If the sample was extruded into the sampling bowl, the sample will be mixed lightly and the VOC sample collected immediately from equal

portions of the bowl. The remaining parameter samples will be homogenized in a stainless steel bowl prior to sample collection, and all samples will be collected using a dedicated clean stainless steel spatula or spoon.

8. Complete sample logs, labels, custody seals, and chain of custody forms. Record sample information in the field notebook.

9. Place analytical samplers in coolers for shipment and cool to 4ºC.

10. If gravely or hard material is encountered which prevents advancing the sampler to the desired depth, direct push should be halted and alternative drilling methods should be attempted.

11. If gravely or hard material is encountered which prevents advancing the sampler to the desired depth, direct push should be halted and alternative drilling methods should be attempted.

At the option of the Field Operations Leader, or site geologist, a new advancement can be attempted when resistant materials prevent the advancement of the probe. The original borehole shall be properly abandoned and the new advancement started a short distance away at a location determined by the site geologist.

7.0 RECORDS

Drilling information shall be recorded in the field and shall include the following as applicable:

- Name and location of job
- Names of crew
- Type and manufacturer of drilling machine
- Weather conditions
- Date and time of start and finish of boring
- Boring number and location (station and coordinates, if available and applicable)
- Well permit number (if applicable)
- Surface elevation (if available)
- Method of advancing the boring

- Depth of water surface and drilling depth at the time of a noted loss of drilling fluid, and time and date when reading or notation was made (as applicable)
- Location of strata changes (as observed)
- Equipment and method of driving sampler
- Type sampler and length and inside diameter of barrel (note use of liners)
- Size, type, and section length of the sampling rods
- Materials applied
- Remarks

Data obtained for each sample shall be recorded in the field and shall include the following:

- Sample depth and, if utilized, the sample number
- Description of soil
- Strata changes within sample (relative depths)
- Sampler penetration and recovery lengths
- Number of blows per 6-in. or partial increment (as applicable), and
- Monitoring readings, if applicable

8.0 REFERENCES

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2. USEPA, 1980. Procedure Manual for Ground Water Monitoring at Solid Waste Disposal Facilities. SW-611. Office of Solid Waste, USEPA, Cincinnati, OH.

3. Scalf, M.R., J.F. McNabb, W.J. Dunlap, R.L. Crosby and J. Fryberger, 1981. Manual of Groundwater Sampling Procedures. NWWA/EPA Series. Kerr Environmental Research Laboratory, Office of Research and Development, USEPA, Ada, OK.

4. American Society for Testing and Materials, 1987. ASTM Standards, D1586-84 (1992). and D1452-80 (1990). ASTM Annual Book of Standards, ASTM, Philadelphia PA, Vol. 4.08.

5. Dept. of the Navy, Naval Facilities Engineering Command, 1982. Soil Mechanics Design Manual 7.1.

6. W.L. Acker, III, 1974. Basic Procedures for Soil Sampling and Core Drilling. Acker Drill Co. Inc., Scranton, PA.

7. American Institute of Steel Construction, 1978. Manual of Steel Construction, 7th Edition. American Institute of Steel Construction, New York, NY.

8. Driscoll, Fletcher G., 1986. Groundwater and Wells, 2nd Edition. Johnson Division, St. Paul. MN.

9. PTP A-48. Surface Soil Sampling.

10. PTP A-73. Sampling Equipment Decontamination Procedures

APPENDIX D

PROPOSED SCHEDULE

Proposed Project Schedule

ACTIVITY	DATE
Mobilize for Sampling	4 December 2001
Complete Sampling/Demobilize from Field	1 March 2002
Submit Draft Work Plan	1 April 2002
Receive Comments	1 May 2002
Comment Resolution Meeting	8 May 2002
Submit Final Work Plan	20 May 2002
Mobilize for Interim Remedial Action	28 May 2002
Complete Interim Remedial Action/Demobilize	23 July 2002

APPENDIX E

RESPONSE TO EPA COMMENTS

Foster Wheeler Response to EPA Comments on the
Sampling Plan for the Characterization of PCB Contaminated Soils and Concrete,
Gould Island, Naval Station Newport, Middletown Rhode Island, Rev. 1
Dated September 14, 2001

GENERAL COMMENTS

1. For future reference, the Navy should use the EPA Region 1 guidance for developing Quality Assurance Project Plans. The guidance *Region 1, EPA-New England Compendium of Quality Assurance Project Plan Guidance, Final, October 1999,* Quality Assurance Unit Staff, Office of Environmental Measurement and Evaluation , describes the results of the planning process that is used to identify all the technical and quality aspects throughout the life of the project.

   The Navy has referred to the specified guidance, and revised the document accordingly.

2. The scope of this project is somewhat confusing and needs clarification. For example, the ***Title Page*** defines this as a sampling project. **Section 1.0 Introduction**, also indicates that this is a sampling project. Further, **Section 5.2 DQOs** also relates to sampling to determine contamination extent. However, in **Section 3.0, Project Management**, the term "removal action scope" and in **Section 8.0 Transportation and Disposal**, the terms "removal action" and "segregating" activities are used, which imply other activities may be taking place. Please revise for clarity.

   Agreed. The document was originally prepared as a Work Plan, with sampling to support future removals. The document has been revised to remove activities associated with remediation.

3. Insufficient information on past characterization data, including sampling SOPs, data validation, and analytical methodologies, was provided to determine the adequacy and soundness of this data. This is of concern as this work plan does indicate that the proposed sampling locations are, in part, based upon what has been determined to date.

   Where appropriate, references are made to previous Work Plans. Additionally, the text has been revised to indicate that any area previously sampled, will be sampled again under this scope. The results of the earlier work do not eliminate sampling, but do identify areas where contamination exists in order to focus on future sampling efforts.

4. The statistical comparison described in Section 5.4.1 should include an explanation of how the data set will be examined for the presence of outliers. In addition, the analytical results from the different methods may be statistically correlated while not providing analytically similar results. The EPA Region 1 document Immunoassay Guidelines for Planning Environmental Projects provides recommended comparability acceptance criteria using Relative Percent Difference. This precision measurement should also be included in the report which provides the comparison of the field method results to the laboratory results.

Agreed. The text has been revised to include a discussion of the method for Quantitative Comparison in Section 5.5.1, as well as a method for identifying outliers. In addition to the linear regression described in section 5.4.1, a Relative Percent Difference (RPD) limit will be calculated using the guidance in the EPA Region I document; Immunoassay Guidelines for Planning Environmental Projects

5. Data validation procedures must be more adequately presented and defined in the QAPP, which is attached to the Sampling Plan. According to the EPA Region I Draft Standard Operating Procedure for Sampling Concrete in the Field, all data must be validated to assure that it is of a quality suitable to make project decisions.

   The text has been revised to indicate that a Tier III data package will be supplied from Analytics Environmental Laboratory (AEL), and a Tier I validation will be performed on the data. Analytics will perform in-house QC checks on the data as specified in the Analytics SOPs and the Analytics Quality Assurance Manual (January 2001). A Tier I data validation will be performed by Foster Wheeler and follow the EPA Region I-NE Data Validation Functional Guidelines for Evaluating Environmental Analysis, December 1996.

6. An additional proof read of both the Sampling Plan and the QAPP is needed. In several instances, words have been omitted from sentences, at times making the content of the sentence difficult to interpret.

   Comment noted. The document has been reviewed for typographical and grammatical errors.

SPECIFIC COMMENTS

1. Based on the information provided in the QAPP, there appears to be minimal QC samples proposed for field sampling and IA analysis (e.g. < 5% overall). We do recommend that a minimum of 5% QC be conducted on field analytical samples.

   Approximately 10% of the Immunoassay Samples will be verified by a fixed analytical laboratory, and a minimum of 5% of laboratory analyses will be duplicated.

2. Section 2.3 provides a discussion of activities that have been undertaken in and adjacent to the transformer vaults and the switch house. Please include in the discussion the following information:

   A. The sampling methodology (e.g. technique and depth) for the concrete and soil samples.

   A reference to the original Work Plan has been added that describes the sampling methodology.

   B. The disposition of the following materials:

      1. Building 53 - walls and roof
      2. Building 54 - walls, roof, floor slab, and excavated soils
      3. Building 56 - walls and roof
      4. Building 59 - walls and roof
      5. Building 60 - walls and slab floor. Additionally no mention is made of the roofing materials.
      6. Building 61 - The text indicates that "concrete debris was sent off site as TSCA waste..." It is unclear if this refers only to the floor slab or also includes the walls and roof. Please clarify.
      7. Concrete Roadway discussed in Section 2.3.7 that was removed.

   A brief discussion of the concrete disposition has been added to the text.

3. A major component of this work involves an off-site laboratory. This laboratory and primary contact should be included on the **Figure 3-1, Organizational Chart**. Further, this chart should be updated to included the telephone numbers of these primary contacts shown, and include contacts for both EPA, RIDEM, and the Navy.

   The analytical laboratory and contact, as well as the individual telephone numbers have been added to the chart.

4. P. 17, Section 4.6 - Please indicate the submittal time frame for the comparison study report which will present the results of the laboratory and immunoassay test results.

   The results of the laboratory and immunoassay tests will be provided in the Work Plan for future removal actions. A proposed schedule for the future work has been added as Appendix D.

5. P. 18, Section 5.1 - In the description of the analytical sampling programs, it is stated that real-time air monitoring will be conducted in order to ensure that exposures do not exceed OSHA

requirements. Please indicate the methodology to be used for the real-time monitoring of PCBs and explain how the results will be compared to an 8-hour time-weighted average concentration.

Foster Wheeler will conduct real time dust monitoring to maintain dust levels less than 15mg/m^3. If this level is maintained, the PCBs in the air will be below the 0.5 mg/m^3 PEL. Section 5.5.3 of the text has been modified to discuss the development of this level.

6. Table 5-1 - Analytical methodologies are provided for all analytes of interest; however, the cleanup and extraction procedures are not specified for many of the analytes. Further, there is no discussion of moisture content, which will present significant problems for extraction efficiencies, especially when low PALs are proposed.

Extraction method 3550 – Sonication has been specified for PCB analyses. Table 5-1 has been updated to include 3550B for SVOCs in solids and 3510C for SVOCs in liquids.

Table 5-3 has been expanded to include all the Data Quality Objectives including fixed laboratory PQLs for Sediments, Concrete, Wipes, Liquids and Waste Characterization Analytes. The solid PQLs listed in Table 5-3 have assumed a % Solids value and have a note that the values will change based on actual % solids. With the Project Action Levels (PALs) included in Table 5-3, it is clear that the Lab PQLs are below the PALs even when the % solids are low.

The extraction method specified 3550B uses a drying step with anhydrous Sodium Sulfate to adsorb moisture present in the sample. For very wet samples, additional Sodium Sulfate is added with mixing until a "free-flowing powder" is achieved. This step eliminates any significant problems with high % moisture samples.

The text in Section 5.3.3 has been modified to include this discussion.

7. Section 5.2.1 - We disagree with the statement "Screening data provide analyte identification and quantitation..." Normally, screening data is semi-quantitative and for some analytes of interest (such as PCBs) identification is not definitive.

The statement is qualified in the phrase following the quote. The text has also been revised to clarify.

8. Table 5.2 - Need to include concrete and sediments in the table. Will a separate sample be collected for moisture? If so, please add to table.

Concrete and sediments have been added.
No separate sample will be collected for moisture content.

9. The only SOP mentioned in either the Sampling Plan or the attached QAPP is EPA's Concrete Sampling SOP, December 1997. All SOPs should be included as an attachment to this Sampling Plan, including SOPs that will be used field sampling (both liquid and non-liquid), SOPs for field analyses, and SOPs for fixed laboratory analytical determinations.

Copies of SOPs are included as Appendix C.

10. Please include a discussion of where the generated waste (including the frac tanks) will be stored at the site and the time-frame for off-site disposal.

Foster Wheeler will store the frac tank and roll-off container on the former Building 32 concrete slab. This is noted on Figure 7-1 in the Sampling Plan and Section 8.4.2 of the text.

11. P. 22, Section 5.3.2 - The third paragraph of this section indicates that if PCB concentrations in the "bottom" concrete sample are greater than 1 ppm, the soil beneath the concrete at that location will be sampled and analyzed for PCBs. The anticipated time frame between obtaining analytical results on the concrete sample and collection of the soil sample is not discussed in this section. The use of a mobile laboratory or field GC is not specified. The soil analytical method is not identified in this section. Please define the analytical methods that will be employed for these analyses or refer the reader to the appropriate section of the Sampling Plan that defines the analytical methodology.

The fixed laboratory analysis of the composite concrete sample will be analyzed on an expedited 48-hour turnaround. Faxed data will be evaluated against the 1 ppm PAL to determine if soil sampling will be required below the concrete.

12. P. 23, Section 5.3.5 - Please provide a clear definition of the benchmarks that will be used for decisions concerning the wipe sample results discussed in this section. If a definition has been presented in the Sampling Plan, please refer the reader to the appropriate section of the document.

Wipe sample results will be compared against a 1 ppm standard. This standard has been added to the text.

13. P. 24, Section 5.4 - The use of a flame ionization detector (FID) for measuring airborne PCB concentrations from the head space of soil samples is questionable. It is uncertain that the ionization potential of the contaminant is low enough or that the chemical is volatile enough to be detected by an FID. If a surrogate contaminant has been selected that is detectable when using an FID, this information should be presented in the Sampling Plan.

Foster Wheeler understands that PCBs will not be detected using an FID. However, it may be possible to detect the carrier oils that contained the PCBs. The text has been revised to clarify the use of the FID.

14. Section 5.4 - The procedure described in Section 5.4 does not appear to be consistent with that shown in Figure 5-1 for soils/sediments. According to Figure 5-1 it appears that lab confirmation is proposed for all locations once it has been preliminarily determined that the PALs have been met; however, the narrative in Section 5.4 does not support this. Please revise for accuracy and clarity.

Agreed. The text has been revised for consistency and clarity.

15. Section 5.4.1 - The last paragraph states that 20% of soil samples will be sent to an off-site laboratory. Table 4-2 indicates that 10% of soils will be submitted for analysis to an off-site laboratory. Figure 5-1 does not indicate any % of samples, but rather implies that the last soil sample will be confirmed off-site. Please revise all text and tables for consistency and accuracy.

Agreed. The text and tables have been revised for consistency and clarity.

16. Section 5.4.2 - The text refers to Figure 5-1 for a summary of laboratory analytical methods and Table 5-3 for the TAL and reporting limits for the methods. Figure 5-1 is a decision tree rather than a summary of analytical methods. Further Table 5-3 only provides reporting limits on Aroclors for Soils and Concrete. Table 4-2 indicates other analyses will be conducted for investigation derived waste. The methods and reporting limits (PQLs?) for the analyses specified in Table 4-2 should be included in Table 5-3. In addition, sediment and water matrices and equipment wipe samples need to be added to Table 5-3.

Figure 5-1 has been changed to Table 5-1. Table 5-3 has been expanded to include the Data Quality Objectives including fixed laboratory PQLs for Sediments, Concrete, Wipes, Liquids and Waste Characterization Analytes.

17. It is unclear what the "reporting limit" specified in Table 5-3 refers to. Is this the laboratory quantitation limit or is the project quantitation limit. If it is the project quantitation limit, the Navy should confirm with the laboratory that it is capable of meeting these limits.

Table 5-3 has been expanded to include the Data Quality Objectives including fixed laboratory PQLs for Sediments, Concrete, Wipes, Liquids and Waste Characterization Analytes.

18. The footnote to Table 5-3 infers that the PAL for soil is 10 mg/Kg based on RIDEM standards. As discussed during previous calls, no "final" cleanup standard has been developed for this site. At the Navy's discretion, an interim standard of 10 mg/Kg was derived based on RIDEM's soil standards. Following a full site investigation, a final cleanup strategy will be developed that focuses on all contaminants of concern. This may include additional cleanup of these areas. The project description should include a clarification/discussion to this effect.

Table 5-3 referred to the 10 ppm limit as an "interim" standard. This table has been revised and the reference deleted. However, for clarification, sentences have been added to Sections 1.0 and 6.2.1 stating "Following interim removal actions, a sitewide closure standard will be developed based on a risk evaluation/assessment."

19. Section 5.6 - Please add the following bullet to the decontamination list:

    - *Collect sample from decontaminated equipment as described in [please insert SOP #].*

In lieu of undertaking a huge sampling to confirm decontamination standards have been met, the Navy may wish to consider using the decontamination procedure for sampling equipment described under §761.79(c). This provides for a performance-based decontamination standard rather than a measurement-based standard.

This section has been modified to state that all sample collection equipment (e.g., scoopulas, etc.) will be disposable, therefore no sample equipment decontamination and/or confirmatory sampling will be needed. Heavy equipment (i.e., drill rig) decontamination is discussed in section 7.2.2.

20. Section 6.2.2 - This section references the 50 ppm cleanup standard for regulation under TSCA. This is misleading as the cleanup and disposal regulations for PCB remediation waste at §761.61

may also apply to waste contaminated with PCBs at < 50 ppm. Please see the definition for *PCB remediation waste* at §761.3. Revise text for accuracy.

The text has been clarified to include the regulatory definition of PCB remediation waste from 40 CFR §761.3.

21. Section 6.2.4 - The last sentence states "All liquids generated and stored on-site will be handled in accordance with all applicable state and federal regulations." Please clarify this statement. As discussed previously, any liquids that are generated during this project, including decontamination water, may be regulated under TSCA if it came in contact with PCB-regulated materials and if it contains above 0.5 μg/L PCBs. Please revise this paragraph to describe the management of liquid wastes. For additional information, please see the PCB decontamination standards at §761.79(b).

This text has been revised to indicate that liquid wastes will be considered TSCA, and handled as such, unless they contain less than 0.5 ug/L PCBs.

22. Section 7.2.3 - The last sentence states "...collected PPE and decontamination water will be characterized and disposed of off-site." Section 7.4, last sentence states "...all accumulated waste...will be sampled for waste characterization..." Section 5.3.1 states that "All wastes other than the contaminated water will be disposed as TSCA for this activity." Please revise for accuracy and consistency.

The text has been revised to indicate that additional waste sampling will be conducted at the frequency specified and for the parameters required by the waste disposal facility.

23. Section 7.2.2 - The 2nd paragraph describes a procedure for decontamination of equipment. This procedure appears to differ from that described in Section 5.6. Please revise for accuracy and consistency.

The text in section 5.3 has been revised, and this section clarified to state that this procedure applies to heavy equipment (i.e., the drill rig).

24. Section 7.3 - The 1st sentence states "The interior...vaults...sampled ...to a depth of approximately 2-feet below the water table using a Geoprobe." Section 5.4, 2nd paragraph states that "Samples will be collected...to approximately 2-feet below the water table or 2-feet below the bottom of the vault, whichever is deeper." Please revise for consistency.

Sections 7.3 and 7.3.1 have been revised to be consistent with section 5.4.

25. Section 7.3.1 - The last sentence of the last paragraph indicates that the contamination in the shore-land area will be considered delineated when the analytical results are < 1ppm. Please clarify if this is a single sample (e.g. 1 grid out from shore) determination or if 2-grid intervals will be used for determination.

Where possible, two (2) consecutive grid intervals will be used to delineate contamination. In the shoreline area, this may be limited by the presence of water that will prohibit sampling at a distance from the shore. Text has been revised accordingly.

26. Section 7.3.2, 2nd paragraph - Please clarify if any sampling of the brick and/or concrete rubble will be conducted prior to installation of borings.

No sampling of the rubble is anticipated. Since the borings are 2-inch, minimal disruption of the rubble is expected.

27. Section 8.6.1, 2nd paragraph, last sentence - If removed material is considered a *PCB remediation waste* as defined at §761.3, the disposal requirements at §761.61(b) would apply. If the Navy is concluding that materials contaminated with < 50ppm at not a *PCB remediation waste*, it and the supporting basis should be included in the discussion.

The text has been clarified to state that, for purposes of this sampling event, the solid wastes will be handled and disposed of as TSCA waste.

28. P. 39, Section 8.6.5 - The third sentence in this section should be reworded. One suggested rewording is "Samples containing greater than one pound of PCBs must also comply with U.S. DOT Hazardous Material Regulations."

The sentence has been reworded as specified.

Quality Assurance Project Plan

29. SOPs should be provided for all sampling and analytical determinations to be conducted under this program.

Copies of SOPs are included as Appendix C.

30. Section 2.0, 3rd paragraph - The 2nd sentence states that a 20' x 20' sampling grid will be used for the concrete pad along with visual bias sampling. However, Section 2.1 refers to a 10' x 10' grid. Please clarify and revise for accuracy.

The text has been corrected to indicate an approximate 20' x 20' grid for the concrete pad.

31. Table 1 refers to a PAL of 10 mg/Kg for Aroclor PCBs. However, a PAL of 1.0 ppm for concrete was previously discussed in Section 2.0 and 2.1. Please clarify the various PALs, PQLs, and laboratory MDLs/QLs for concrete and when each will be used. Further PALs for sediments were previously discussed as 1 ppm, but weren't included in Table 1.

Table 1 has been expanded to include all the Data Quality Objectives including fixed laboratory PQLs for Sediments, Concrete, Wipes, Liquids and Waste Characterization Analytes.

32. Table 2 - The Analytical Method/SOP Reference for concrete is given as 4020/8082. This is inconsistent with the information provided in Figure 5-1 and in the text. Further, EPA recommends that MS/MSD samples be included in the QAPP and it is a standard part of the 8082 method.

Table 2 has been revised to be consistent with Table 5-1 in the Sampling Plan. It now includes MS/MSD samples as well as equipment blanks.

33. Table 1 - The Analytical Method MDL for 4020 is shown at 0.20 μg/Kg. This appears low as the manufacturer's information suggests an MDL of 0.5 mg/Kg. This number has been established by the manufacturer under ideal circumstances and may not be achievable in true field conditions.

The Method QL shown for 8082 is also specified in the method, but may not be achievable by the laboratory. Please confirm that these are achievable with the selected laboratory.

Table 1 has been expanded to include all the Data Quality Objectives including fixed laboratory PQLs for Sediments, Concrete, Wipes, Liquids and Waste Characterization Analytes.

34. EPA's QAPP guidelines require that DQOs, DQIs and MPCs be included in this discussion. While to some degree, a discussion of DQOs was included, there was little discussion of DQIs and MPCs and how they were selected. Please discuss each and the established quality limits. Please see *EPA-NE QAPP Manual Sections 7.1 and 7.2* for a further discussion on the concept of project quality objectives (PQOs) and measurement performance criteria (MPC).

A Tier I data validation will be performed by Foster Wheeler and follow the EPA Region I-NE Data Validation Functional Guidelines for Evaluating Environmental Analysis, December 1996. The data package supplied by AEL will be a Tier III package. However, since the data will only be used for determination of contaminated areas to be remediated, and additional confirmatory data will be collected and validated following remedial action, this QAPP is considered sufficient to ensure the Tier I validation and will not be revised to address questions 34, 35, 36, 37, 38 & 39.

35. The ability to generate data to meet PQOs is evaluated through the process of identifying the data quality indicators (DQIs...formerly referred to as PARCCS parameters) to be evaluated, setting MPC for each of the DQIs, and defining the QC samples to be collected to assess whether or not the MPC are met. Then, a sampling process design is developed and both sampling and analytical procedures are chosen that will support achieving the defined PQOs and assessing the MPC (by including the defined QC samples and QC limits that are at least as stringent as the MPC). However, it is not clear that this has been done. For example:

a) **Accuracy/bias:** The MPCs that have been established for the various COPCs appear to be based on those quality controls established by the laboratory. This is not the customary approach. As stated in an earlier comment, the MPC must be defined and then analytical methods are chosen to meet or exceed these criteria.

b) Analysis of performance evaluation (PE) samples should be included for each sample media and analytical parameter (at each laboratory to be performing the analysis) as a measure of accuracy/bias. If PE samples are unavailable, standard reference materials (SRMs) may be analyzed as second source standard samples.

c) **Sensitivity** was not discussed.

d) **Accuracy/Bias-Contamination** and **Precision**- Indicate whether the QL is the PQL or the achievable lab QL. It is recommended that this be the PQL.

e) **Completeness** goal must be established. We recommend that the Navy keep completeness greater than 90%, otherwise the Navy may have very limited data from only a few samples in some areas.

f) **Comparability** was not discussed.

36. Sampling SOPs must be submitted and should include instructions for collecting associated field QC samples (e.g., field duplicates, equipment blanks, etc.).

37. **Data Verification and Validation Tasks**, page 4: The MPCs and the Laboratory QC criteria are the criteria that will be followed by the validator to compare the laboratory QC results. The Use of the Region 1 data validation functional guidelines provide formats on how the validation will proceed and be documented. The criteria that will be checked are based on that criteria established by the DQI and MPC process and should be referenced/documented here.

38. **Data Usability Assessment Tasks**
As stated earlier, the generation of data of necessary quality to meet the PQOs is ensured by setting MPC for each of the DQIs, defining the QC samples to be collected to assess whether or not the MPCs are met, and following prescribed procedures (sampling, analysis, documentation, etc.) selected/designed to generate data to meet the PQOs and MPC. The data is typically validated versus a set of criteria (which may or may not be equivalent to the MPC) and then assessed for usability in meeting the PQOs by evaluating compliance with the MPC. This is not a laboratory function for the project as these goals are not established by the laboratory, but rather by the project team and are based on data quality needs. This section should be revised to reflect these goals.

39. P. 7, Section 5.0 - According to the information presented in this section, approximately 5% of the samples collected will be analyzed using a dual capillary column GC/ECD via Method 8082. The Sampling Plan indicates that at least 20% of the samples (page 25) will be submitted to the laboratory for analysis using Method 8082 for the purposes of performing a statistical comparison of the immunoassay method and the laboratory method. Table 4-2 of the Sampling Plan indicates that 10% of the soil samples will be submitted to the laboratory for confirmatory analysis. Please ensure that statements concerning the specified analytical methods to be utilized are consistent.

40. PP. 8 & 9 - Tables 5 and 6 include footnotes references 1, 2 and 3 but no footnotes are included. Please review and provide descriptions for the footnotes.

The text has been revised.

APPENDIX F

RESPONSE TO RIDEM COMMENTS

Foster Wheeler Response to RIDEM Comments on the
Sampling Plan for the Characterization of PCB Contaminated Soils and Concrete, Gould Island, Naval Station Newport, Middletown Rhode Island, Rev. 1
Dated September 14, 2001

1. **General Comment**

In lieu of performing the proposed investigation, the Navy may elect to utilize the existing information and proceed forward with removal actions. Sampling of the roadways and other suspect area may be conducted at the same time as the remedial efforts.

Comment noted. However, the proposed sampling plan is required in order to comply with TSCA regulations regarding characterization of waste in-situ.

2. **General Comment**

The Office of Waste Management is aware that the Navy is considering conducting a removal action for the contaminated soils at the site. These soils would be disposed of at an off site facility. As a means to reduce disposal cost the Navy may elect to evaluate soil washing. This could be performed either onsite or using the existing Material Handling Area and remediation equipment at Tank Farm # 5.

Comment noted. The current document addresses only the sampling phase of the site activities. Remedial actions will be addressed in Work Plan to be prepared following receipt of sampling results.

3. **General Comment**

As noted in the title this document is supposed to be a sampling plan. A good portion of the document seems to imply that this is a removal action plan. For example, in Section 3.0 it is noted that John Carey will supervise craft labor and site procurement of materials (what craft and procurement of materials are needed to sample?), Section 3.1 states that units of weights and measures on submittals shall be the same as on project drawings and specs (what units of weights and measures are needed for sampling?), Section 4.0 is entitled Construction Quality Control (Again, what construction is needed for sampling?). Please clarify the intent of this work plan.

Agreed. The intent of the plan is for sampling only. The text has been revised to remove references to removal action and/or construction.

4. **General Comment**

Engineering plans, SOPs and other historic documents provide information, which is useful for remedial investigations and actions. Accordingly, the Office of Waste Management requested that the Navy utilize these plans for the proposed remedial investigations/actions to be conducted at the site. Apparently, the Navy was unable to obtain plans for the structures currently under investigation, (the

submitted plans contained piping and other layout schemes for the powerhouse and the salt-water pier). Please indicate whether the following plans were available and or contained useful information:

Power Plant and Substation One Line Relay Connection	06136-068
Deep Well Torpedo Station Gould Island	05932-066

Be advised that as part of the review for this Work Plan, the Office of Waste Management viewed engineering plans for the transformer substations on the southern end of Gould Island. These plans were previously obtained from the Navy for projects being performed on the southern end of the island. The plans provided details concerning transformer substation layouts, foundation layouts, drainage designs for substations, designs for electrical conduits, etc. The Office of Waste Management suggest that the Navy ascertain whether the transformer vaults on the northern end of Gould Island were similar in design to others located on the base.

All available drawings were reviewed in preparation of this plan, however the 2 drawings noted were not available. It was determined that there is insufficient information to determine the route of conduit and/or piping once it exited the vaults. An approximate location of the conduits was included in the drawings, to be field verified. Sampling locations will be field adjusted, as necessary, to attempt to sample adjacent to conduit.

5. Page 1, Section 1.0, Introduction,
Paragraph 2, Sentence 2

This sentence states that this sampling plan is being used to develop an interim removal action. Please be advised that RIDEM does <u>not</u> support the use of interim cleanup goals. The State's PCB standard is 10 PPM and at a maximum, all removal actions at the individual sites should be completed to this standard. Please be advised that at other sites a remedial standard of 1 ppm was employed in order to address ecological concerns. This site is located adjacent to a bird sanctuary and the site itself is actively used as a rookery. Further it is located adjacent to a sensitive environment. The 1-ppm standard is also in concert with potential applicable TASCA regulations. Accordingly, a 1-ppm remediation goal should be considered for the site.

For the purposes of this investigation, soil data will be compared against RIDEM's PCB clean-up standard of 10 ppm. Remediation Standards will be evaluated at a later date as part of Work Plan development.

6. Page 1, Section 1.0, Introduction,
Paragraph 2, Sentence 2

The Work Plan states that interim actions will be conducted at the site. As stated in the above, the Office of Waste Management will not approve interim cleanup standards. Further, the Office of Waste Management also suggests that all of the removal actions be conducted at the same time. This will save the Navy having to incur multiple mobilization costs which in this case are relatively high due to the site being located on an island (the Office of Waste Management is aware however, that logistical, funding or other concerns may affect this option).

See responses to comments 1 and 5 above.

7. **Page 14, Section 4.4.2,**
   Chain of Custody, Paragraph 1

   This paragraph states that the chain of custody form is in Appendix A. It is not, please provide.

   The chain of Custody Form has been added to Appendix A.

8. **Page 19, Table 5-1,**
   Summary of Analytical Procedures and QC Requirements

   The waste characterization of liquids and solids may require more than one sample depending upon the amount and where the investigative derived waste is ultimately going to end up.

   Waste characterization sampling will occur at the frequency required by and for the contaminants specified by the selected waste disposal facility. The sampling provided in Table 5-1 presents the minimum anticipated.

9. **Page 22, Section 5.3.3,**
   Soil Sampling, Paragraph, First Sentence

   The report notes that samples in the field will be analyzed using immunoassays test kits. Considering the number of samples to be run a the site, the Office of Waste Management recommends that the Navy evaluate whether a field GC/MS unit would be more cost effective then field immunoassay kits.

   A preliminary evaluation of the requirements has determined that the use of a field GC/MS will not be cost effective for this application.

10. **Page 23, Section 5.3.4,**
    Sediment Sampling, Paragraph 2, Last Sentence

    Please explain what is meant by field adjustments to sampling methodology and how this could impact the results of the sampling effort.

    The primary field adjustment will be an adjustment to the sampling location based on the ability to obtain a sample at the specified location.

11. **Page 24, Section 5.4,**
    Analytical Methodology, Paragraph 1, Sentence 5

    It is noted that if there is no staining, headspace FID readings will be taken at the four corners of the vault and from the center. Please revise to state that a reading will also take place at any doorway location to the vault and the soils immediately surrounding the vault.

At RIDEM's request, the Navy has agreed to use an FID in the interior of the vaults to determine the sample location if the location of the transformer is unknown. The sampling frequency specified around the perimeter of the vaults will provide adequate coverage to identify potential contamination. The FID will not be used to determine exterior sample locations.

12. Page 24, Section 5.4,
Analytical Methodology, Whole Section.

The Work Plan outlines the sampling decision matrix for borings located inside and outside of the vaults. The matrix proposes two different approaches based upon interior vault samples being above or below 10 ppm, with the more rigorous approach being assigned to sites with interior vault concentrations greater than 10 ppm in the soil. The basis for this approach appears to be the assumption that a sample taken from the bottom of the vault will either be taken in the release and/or the release would have originated from the interior of the structures, (as opposed to releases which may have occurred from conduits leaving the site or disposal of waste PCB oil exterior of the site). This approach would seem to contradict what has been observed at the site. The information presented in Figures 2-1 and 2-2 of the Work Plan show that <u>all</u> of the interior soil samples are below 10 ppm for all of the structures, yet elevated levels of PCBs have been found in the exterior samples collected at all of the vaults. Accordingly, interior soils sample results may not be an appropriate criteria upon which to base a more rigorous sampling scheme.

Please note that the sample results presented in the vault interiors on Figures 2-1 and 2-2 of the Sampling Plan are the results from water samples that were collected from below the vault floors. The only soil data available from below the floor of the vaults is in building 54. This data has been added to the figure.

The Office of Waste Management recommends the following: In the absence of field or other evidence of contamination a minimum of two samples from each boring, one at the water table and one at the surface, would be analyzed for PCBs. The analytical results from these samples will be used to determine if additional sampling is needed. If field or other evidence of contamination is present (that is, presence of staining, oil, olfactory evidence, the presence of preferential migration pathways, conduits, known data from both inside and outside the vaults, etc). Samples would be taken in these areas independent of the results for the samples taken at the surface and at the water table (i.e. a sample would be taken of stained soil or soil containing oil independent of the analytical results for the surface or water table sample).

If there is no contamination in the floor of the vault at depth, and there is no surface contamination around the exterior of the vault, the probability that PCBs have migrated to depth outside the vaults is minimal. Therefore, sampling at depth outside the vault will not be performed unless one of these conditions exists.

13. Page 24, Section 5.4,
Analytical Methodology, Paragraph 2.

"If the lab analysis shows PCB concentrations > 10 ppm samples will be analyzed above and below the groundwater level."

The plan calls for the use of laboratory confirmation of field data for groundwater level data as a criterion to determine how the other samples in a particular boring will be analyzed. This approach will necessitate that a rapid turn around of the laboratory ground level samples in order to insure that the holding times for the other samples in the boring are not exceeded. Please modify the Work Plan to reflect this requirement.

Rapid (48-hour) turnaround for the specified samples has been planned. This turnaround will be adequate to support the 14-day sample holding times. The text has been modified to include this discussion.

14. Page 24, Section 5.4,
Analytical Methodology, Paragraph 2.

The work plan proposes two different sampling methodologies. "If the groundwater sample from the interior of the vault shows PCB concentrations< 10 ppm, the dry samples from one foot or greater above the water table will be analyzed in the field via immunoassays from the surface down until two consecutive samples meet the minimum concentration of 10 ppm.... If the laboratory samples shows PCB > 10 ppm, samples will be analyzed above and below the water table. Wet samples or samples within one foot of the water table will be sent off site for analysis. When the two results are less than or equal to 10 ppm, the area will be considered completely delineated."

The text is incorrect. The word "level" should be inserted between the words "groundwater" and "sample" in the first sentence.

This is a public document, therefore please explain what is meant by the last criteria. That is, for samples collected in areas in which the interior vault concentrations exceed 10 ppm, will all of the samples in a particular boring be analyzed and the site considered delineated when two samples do not exceed 10 ppm, or will the samples in a particular boring be analyzed until two consecutive samples boring samples do not exceed 10 ppm? At that point the analysis of the remaining samples is terminated? If this is true please indicate whether the samples will be collected from the surface to the bottom, as was the case for samples less than 10 ppm, or from the water table up)? Alternatively, will the site be considered delineated when two results within one foot of the water table do not exceed 10 ppm?

The text has been clarified to state that the analysis will be considered complete when 2 consecutive samples in a particular boring do not exceed 10 ppm. For samples where the interior vault sample has exceeded 10 ppm, sample collection will begin with the sample at the groundwater interface and proceed in both directions (up and down) until two consecutive samples less than 10 ppm are identified.

15. Page 31, Section 6.2.2,
Toxic Substance Control Act

This section of the Work Plan notes that concentrations of PCBs above 50 PPM will be regulated under TSCA. Please be advised that concentrations of PCBs above 10 ppm are regulated by RIDEM in accordance with the remediation Regulations, amended 1996. The Work Plan should be modified to reflect this requirement.

Comment noted. The text has been revised to be consistent with RIDEM regulations.

16. Page 32, Section 6.5.1,
Inspection by Regulatory Personnel

The Office of Waste Management request one weeks notification prior to the commencement of field activities, and when possible twenty-four hour notification for the cancellation of scheduled activities. The Office is aware that it is common for investigations of this nature not to follow proposed schedules. According, as was done for other sites on the base, the Office of Waste Management requests that the Navy submit a weekly update of upcoming field activities.

The Navy will provide weekly updates to RIDEM including notifications regarding the commencement of work. The text has been modified to include this notification.

17. Page 3, Section 7.3.1,
Transformer Vaults,

The Office of Waste Management acknowledges that the primary focused of this investigation is to determine the extent of PCB contamination. However, releases of petroleum, solvents, and metals are known to have occurred in this area including locations adjacent to the transformer vaults. Therefore, the Work Plan should stipulate that samples for TPH, metals, VOCs, etc. will be collected <u>if</u> there is field evidence that this contamination exist. As an illustration if there is field evidence of diesel petroleum contamination in a boring an additional sample of the soil should be collected and sent off site for the appropriate analysis. This contingency will merely require that extra sample jars be brought to the site.

If, during field activities, evidence of petroleum or other contamination is noted, it will be considered a changed condition and the procedure to address it will be discussed at that time. Additional sample containers will be available at the site to address such a situation.

18. Page 3, Section 7.3.1,
Transformer Vaults,

The first paragraph in this section discusses sampling in the <u>interior</u> of the vaults. It specifies that the decision to terminate sample <u>analysis</u> at each interior vault boring location will be based upon the results of two consecutive samples exhibiting PCB concentrations less then 10 ppm. The samples in the borings will be collected from the top down. The second paragraph starts with a discussion of sample <u>collection</u> from the <u>perimeter</u> of the vaults. The paragraph then discusses the decision matrix

to be used for analysis of samples in the interior of the vaults. As this is a public document, the please clearly state what decision matrix will be used for samples inside and outside of the vaults.

The second paragraph discusses the sampling procedure for the exterior of the vaults. This procedure is based on the results of the interior samples. The text has been clarified.

19. Page 3, Section 7.3.1,
Transformer Vaults, Whole Section

The primary focus of this investigation is to determine the extent of contamination and investigate potential contaminant migration pathways. In regards to the latter the electrical conduits may represent a preferential pathway via the conduits themselves and or the loose backfill that was placed in the conduit excavations. Accordingly, samples should be taken immediately in these areas. The Office of Waste Management suggest that the following samples points be moved in order to achieve this goal:

Transformer Vault Building 53	Move either or GI –B53-B8 or GI-B53-B9 directly on top of the underground conduit.
Transformer Vault Building 54	Conduit not shown in plan. Determine location of conduit using either historical documents or field methods and move sampling points accordingly.
Transformer Vault Building 56	Move GI–B56-B2 directly on top of the underground conduit
Transformer Vault Building 59	Move GI–B59-B2 directly on top of the underground conduit
Transformer Vault Building 60	Move GI–B60-B2 directly on top of the underground conduit
Transformer Vault Building 61	Move GI–B61-B15 and GI-B61-B14 directly on top of the underground conduit
Switch House Building 59	Move GI–B59-B20 directly on top of the underground conduit

Since no definitive data regarding the exact locations of the conduit was available, these sample locations have not been modified. If visual examination of the vaults indicates the conduit location in the field, and the path from the vault to Building 32 is apparent, the sample location will be field modified as appropriate.

20. Page 3, Section 7.3.1,
Transformer Vaults, Whole Section

The Work Plan notes that each structure contained a sump floor slab. Please include a description of the sump floor slab. At a minimum this description should note the following: whether the sump floor slab was located at the base of the foundation or on the floor which supported the transformers,

whether any drains were located in the sump, whether the sump was pierce by electrical conduits (all are located at the location where the electrical conduit entered the building), whether any staining was observed in the sump, the function of the sump and its operation. That is, information from historic plans or SOPs indicated what was the function of the sump and how was it operated. As an illustration blind sumps, (sumps with no drains) are normally pumped and discharge to a designated area).

No historical drawings or documents have been located to provide the requested information. Since the vault interior is a relatively small area, and headspace readings will be taken in the center and at the corners, the sample will be representative of the worst –case interior contamination.

21. Page 3, Section 7.3.1,
Transformer Vaults, Whole Section

The Work Plan notes that each structure contained a sump floor slab. Please indicate why samples were not historically collected from these sumps, and why the current sampling program does not call for collection beneath these areas.

The entire vault interior comprises an area of less than 9-feet by 16-feet. The sumps were typically located at one end of the vault. Using the agreed to method of collecting FID readings at the center and corners of the vaults, 2 FID readings will be taken in each sump. This data will adequately represent the worst-case vault interior sample and will identify any potential spills in the sump.

22. Page 35, Section 7.3.1,
Transformer Vaults, Paragraph 1

"If groundwater level sample from the interior of the vault shows PCB concentrations < 10 ppm, the dry sample from one foot or greater above the water table will be analyzed in the field via the immunoassay method from the surface down until two consecutive samples meet the minimum concentration of 10 ppm."

The above calls for preferential sampling from the surface down with termination of sampling activities if two consecutive samples do not exhibits signs of PCBs. This procedure would be appropriate if the source of contamination was known to originate from the surface spills. At this site this may not be the case as the contamination may come from releases at the base of vault, release from the electrical conduits or from the surface. Therefore, the Office recommends that this approach be modified as follows: A portion of each split spoon will be evaluate for visual, olfactory, or field instrument (PID/FID) evidence of contamination. Samples exhibiting field evidence of contamination will undergo analysis. In addition, if conduits, pipes, leachfields, or other potential preferential contamination migration routes are uncovered, samples will be taken from these areas. If field evidence of contamination or migration routes are not present then at a minimum the sample in the water table and the surface soil sample will be analyzed for PCBs. Sampling will continue until two consecutive samples meet the minimum concentration of 10 ppm.

If there is no evidence of contamination inside the vault (i.e., if the sample contains less than 10 ppm PCBs), and 2 consecutive surface samples contain no contamination, there would be no contamination to migrate through the conduit. No additional sampling would be necessary.

23. Page 35, Section 7.3.1,
Transformer Vaults, Paragraph 1

"If groundwater level sample from the interior of the vault shows PCB concentrations > 10 PPM, then the groundwater sample will be sent to the lab prior to analyzing the remaining sample. If the lab analysis shows PCBs concentrations > 10 PPM samples will be analyzed above and below the water table."

The above would seem to indicate that all of the samples (from groundwater level to surface) will be analyzed if PCB concentrations are found to be greater then 10 ppm. This is a public document, therefore please clarify.

See responses to Comments No. 14 and 18.

24. Page 35, Section 7.3.1,
Transformer Vaults, Paragraph 1

"If groundwater level sample from the interior of the vault shows PCB concentrations > 10 PPM, then the groundwater sample will be sent to the lab prior to analyzing the remaining sample. If the lab analysis shows PCBs concentrations > 10 PPM samples will be analyzed above and below the water table."

Independent of the analysis at the groundwater table samples the other boring samples should be evaluated for visual, olfactory, or field instrument (PID/FID) evidence of contamination. Samples exhibiting field evidence of contamination will undergo analysis. In addition, if conduits, pipes, leachfields, or other potential preferential contamination migration routes are uncovered, samples will be taken from these areas. This comment applies to all borings, interior, exterior of the vaults, etc. taken at the site. In addition, at a minimum a sample at the surface and in the water table should be analyzed at each boring location.

See responses to Comments No. 14 and 18.

25. Page 35, Section 7.3.1,
Transformer Vaults, Paragraph 2

The work plan calls for the collections of sediment samples to a depth of two feet below the water table using a geoprobe. The shoreline adjacent to the site consists of a cobble stone beach. This will present logistic problems for the use of the geoprobe and its ability to collect split spoons samples with usable material. As the proposed depth for the boring is essentially two feet, samples may be

collected by hand using a wide mouth hand auger or shovel. Sample collection should also be conducted at low tide.

Agreed. A field evaluation will be made at these locations to determine whether use of the Goeprobe rig is appropriate for sample collection. Alternatively, a hand auger will be used. The text has been modified to discuss this alternative.

APPENDIX G

ANALYTICS ENVIRONMENTAL LABORATORY QAP

QUALITY SYSTEMS MANUAL

FOR

ADMINISTRATIVE, ORGANIC AND LIMITED INORGANIC DEPARTMENTS OF ANALYTICS ENVIRONMENTAL LABORATORY, LLC.

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1/22/01
Date

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1.0 Quality Policy Statement

Analytics Environmental Laboratory, LLC., is a commercial laboratory that provides specialty organic analyses in a variety of environmental matrices including ground water, surface water, effluents, soils, sediments, industrial and hazardous wastes, and other selected materials.

Analytics is committed to sound and useful Quality Assurance/Quality Control management practices; generating accurate and precise analytical data of verifiable quality is the first corporate priority.

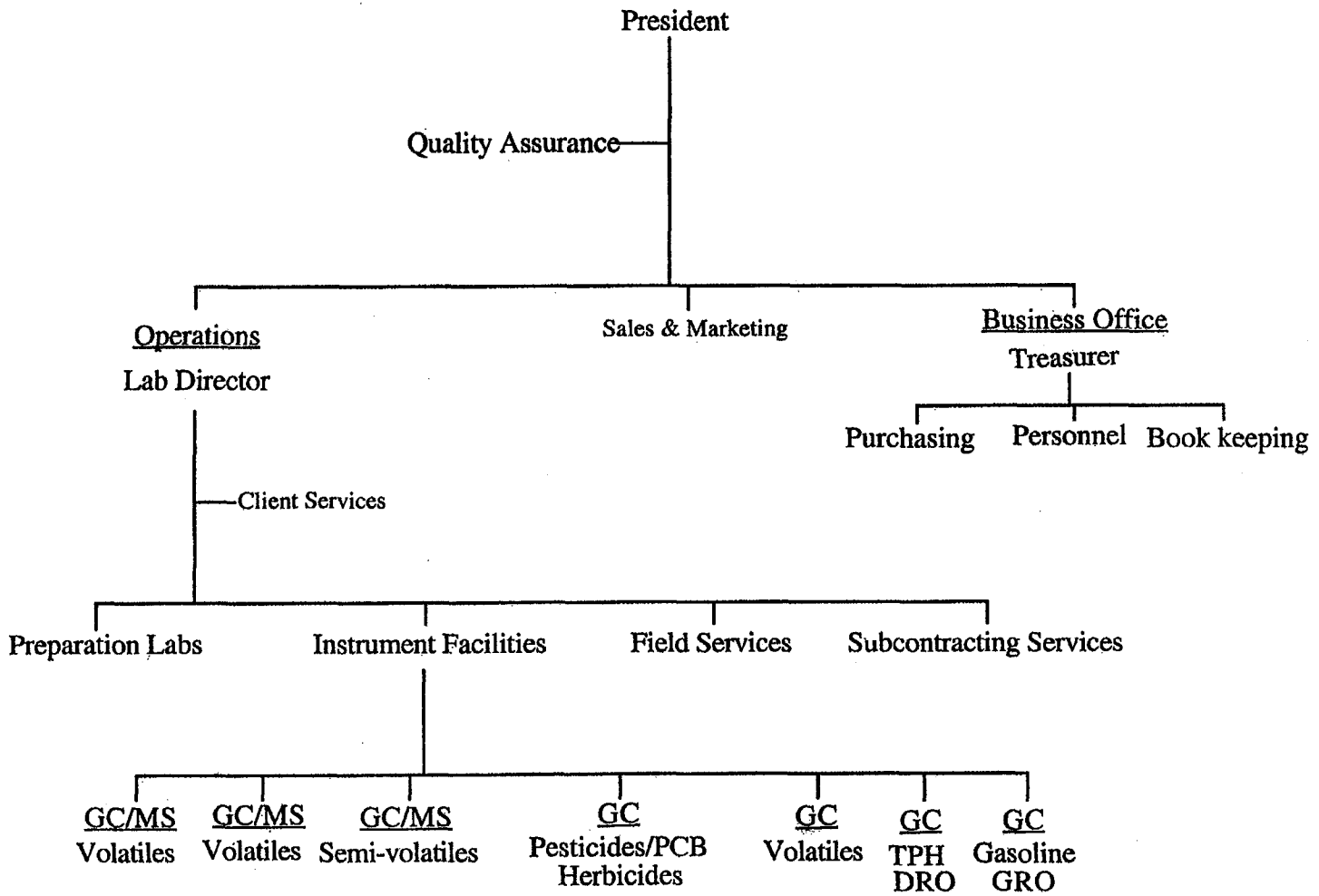
This Comprehensive Quality Assurance Plan presents the policies, objectives, functional activities, and specific Quality Assurance and Quality Control (QA/QC) activities designed by Analytics Environmental Laboratory, LLC. to achieve high quality data. The use of written Standard Operating Procedures provides guidance regarding acceptable practices. The specific quality control procedures outlined in this plan are based on the U.S.E.P.A. Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/4-88-039, July, 1994), and U.S.E.P.A. Test Methods for Evaluating Solid Waste - Physical and Chemical Methods (EPA/SW-846, Third Edition, 1986 and all revisions and updates). The methods outlined in this document are used in the implementation of the services that Analytics provides.

2.0 Organization and Management Structure

Analytics Environmental Laboratory is a New Hampshire Limited Liability Company, established in 2000, to provide organic analyses on environmental matrices. Analytics teams with other laboratories to provide our clientele with full environmental services. The company organizational chart is presented in Figure 4.1, and the names and duties of the laboratory personnel are listed below. Each employee is trained by an analyst proficient in the procedure. Each analyst must successfully complete an Initial Demonstration of Capability before he or she may analyze client samples.

2.1 Organizational Chart

Analytics Environmental Laboratory



2.2 Management Responsibilities

2.2.1 Responsibilities of laboratory management.

a) The laboratory management ensures the supervision of all personnel employed by the laboratory.

b) The laboratory management is responsible for ensuring the quality of data produced by the laboratory.

c) Laboratory management will be responsible for training and keeping personnel up to date on laboratory procedures, operation of instrumentation and laboratory support equipment.

d) The laboratory management will nominate a deputy in the absence of the laboratory director or QAO, or both.

e) It is the responsibility of management to ensure that employees are not under any outside pressure(s) that may affect the quality or objectivity of the employee's work. This is done by following three procedures:

1. All data is reviewed by a second person so that any one employee can not alter data and have it sent to the client.
2. ANALYTICS has an open door policy so that any employee can speak to management about any problem he/she considers important.
3. ANALYTICS has and ethics policy whereby all employees sign a contract stating that they will not falsify data.

2.2.2 Quality Assurance Officer (QAO)

a) The QAO is the person responsible for the laboratory's quality assurance program and it's implementation.

b) The QAO reviews laboratory data and conducts annual internal laboratory audits. The QAO ensures any corrective action arising from internal audits are implemented in a timely manner.

c) The QAO is free from internal and external influences when evaluating data and conducting audits.

d) The QAO have training or experience, or both in quality assurance/quality control procedures and is knowledgeable of the approved analytical methods and quality assurance program requirements.

e) The QAO has direct access to the laboratory management.

2.3 Job Descriptions of Staff Positions

Laboratory Director
The Laboratory Director is responsible for management of the day to day operations of the laboratory. The Laboratory Director establishes and implements policies for administrative and technical operations and quality assurance. The laboratory director or (designee) reviews all final data generated to ensure that the quality of the data meets the required standards. He/she is also responsible for sales, marketing and implementing the fiscal policies. He/she is also the primary contact for project management, quotes and solicitations and client services. He/she is responsible

for hiring/firing of employees and also oversees the training, waste management and safety programs.

Assistant Laboratory Director
The Assistant Laboratory Director is responsible for technical aspects of the laboratory. He/she is responsible for method development and method implementation. He/she is also responsible for supervision and training of the sample custodian and reporting specialists. He/she is also the secondary contact for project management, quotes and solicitations and client services. He/she performs raw data review, reporting and acts as a backup analyst for instrumental analyses when needed.

Laboratory Supervisor
The Laboratory Supervisor is responsible for training and supervision of the laboratory employees. He/she plans and schedules work throughout the laboratory, directs staff in their daily operations and is responsible for the timely completion of analytical work. He/she performs raw data review, reporting and acts as a backup analyst for instrumental analyses when needed.

QA Officer
The QA Officer (QAO) is responsible for the implementation of the QA program which includes initiating and overseeing internal and external audits, validating QC criteria, and updating the QA Plan, scheduling and conducting PE/PT studies, review and updating of SOP's, scheduling MDL and IDC studies. He/she maintains the certification files and is responsible for contact with the certification officers in each state or agency.

GC/MS Analyst
The GC/MS analysts are responsible for performing all aspects of the GC/MS operation including sample loading, tuning, calibration, sample analysis, data reduction, mass spectral interpretation, QC evaluation, adherence to SOPs, and maintenance and cleaning of the GC/MS systems. He/she also is responsible for preparation of calibration and spiking standards, QC Batching of samples, and filing of raw data for either QC samples or client samples. He/she also is responsible for archiving the raw GC/MS data onto backup tapes. After analysis of samples is complete the GC/MS analyst is responsible for removing the samples from the LIMS "Worklist".

GC Analyst
The GC analysts are responsible for performing all aspects of the GC operation including sample loading, calibration, sample analysis, data reduction, chromatogram interpretation, QC evaluation, adherence to SOPs, and maintenance and cleaning of the GC systems. He/she also is responsible for preparation of calibration and spiking standards, QC Batching of samples, and filing of raw data for either QC samples or client samples. He/she also is responsible for archiving the raw GC data onto backup tapes. After analysis of samples is complete the GC analyst is responsible for removing the samples from the LIMS "Worklist".

Organic Preparation Analyst
The organic preparation analyst is responsible for performing organic extractions, extract cleanup and extract concentration in accordance with the established procedures and SOPs. He/she performs daily extractions as directed by the laboratory supervisor. The organic preparation analyst is responsible for placing orders for organic prep. supplies, cleaning of glassware, maintenance of extraction equipment and standard preparation. The Organic Preparation Analyst is also responsible for checking the refrigerator temperatures on a daily basis and maintenance of the MSDS files as directed by the Laboratory Director.

Sample Custodian
The Sample Custodian is responsible for sample receipt, sample log-in, sample management and

Sample Custodian
The Sample Custodian is responsible for sample receipt, sample log-in, sample management and sample disposal. He/she performs sample courier services and sample container preparation and shipment. He/she attends to clients that enter the laboratory with samples for drop-off or sample container pickup. He/she is also responsible for answering the telephone and responding to inquiries for sample container shipment.

Purchasing Agent
The purchasing agent is responsible for placing all purchase orders, selecting vendors, maintaining purchasing budget, establishment of pricing and coordination with bookkeeper.

Reporting Specialist
The reporting specialist is responsible for reporting of the final data using the established LIMS report formats. He/she receives raw data printouts from the instrumentation and is responsible for transferring (either electronically or manually) the data from the instrument file onto the data report forms. The reporting specialist reviews the report for adherence to the laboratory reporting criteria such as significant figures and other reporting policies. He/she prints all final reports and submits then to the Laboratory Director for review. In addition to the data reporting duties, the reporting specialist also is responsible for generation of all required QA/QC forms for preparation of QC Reports. These forms include CLP-type forms for Level # and Level 4 data reports.

Waste Management Specialist
The Waste Management Specialist is responsible for implementation of the waste management policies of the laboratory. He/she performs sample disposal, waste tracking, coordination with waste hauler and waste manifesting activities. He/she is also responsible for waste disposal reporting to state and local authorities as required by the regulations.

Computer Specialist
The Computer Specialist is responsible for maintenance of the Laboratory Information Management System (LIMS) and the supporting software and hardware. He/she performs computer-programming services to the laboratory as directed by management.

Personnel Administrator
The personnel Administrator is responsible for maintenance of the personnel files, payroll and benefits.

Bookkeeper
The bookkeeper is responsible for financial transactions, including accounts payable, accounts receivable, general ledger account maintenance as well as preparation of financial reports.

Owner/Manager
The principal is responsible for implementing corporate policy, overseeing the corporate services, and contract and business administration.

2.4 Personnel Qualifications
All AEL full time employees must possess a 4-year academic degree. Employees working in the Organics laboratory should possess a science degree or have 1-year science related experience.

NAME: Stephen L. Knollmeyer
TITLE: Owner/Manager, Laboratory Director

EDUCATION: B.S. Geology, Univ. of New Hampshire 1984
YEARS OF EXPERIENCE: 17 years
DUTIES: Owner/Manager
Laboratory Director

NAME: Melissa A. Gulli
TITLE: Assistant Laboratory Director/QA Officer
EDUCATION: B.S. Environmental Science, Univ. of Massachusetts 1987
YEARS OF EXPERIENCE: 13 years
DUTIES: Assistant Laboratory Director
QA Officer
Reporting Specialist
Backup GC/MS Analyst for VOCs

NAME: John Raifsnider
TITLE: Laboratory Supervisor, GC/MS Analyst
EDUCATION: B.S. Freshwater Biology, Univ. of New Hampshire 1995
YEARS OF EXPERIENCE: 6 years
DUTIES: Laboratory Supervisor
GC/MS Analyst for VOCs
GC Analyst for Pest/PCBs and Herbicides
Purchasing Agent

NAME: Angelina Richard
TITLE: GC/MS Analyst
EDUCATION: B.S. Animal Science, Univ. of New Hampshire 1986
YEARS OF EXPERIENCE: 12 years
DUTIES: GC/MS Analyst for SVOCs
GC/MS Analyst for EPH/PAHs
GC Analyst for GRO and TPH
GC Analyst for Methane/Ethane/Ethane
Backup GC/MS Analyst for VOCs
Backup GC Analyst for Pest/PCBs and Herbicides

NAME: Matthew Tardif
TITLE: Analyst
EDUCATION: B.S. Environmental Science, Springfield College 1998
YEARS OF EXPERIENCE: 3 year
DUTIES: GC Analyst for DRO and TPH
Organic Preparation Analyst
Waste Management Specialist

NAME: Sara Roberts
TITLE: Analyst
EDUCATION: B.S. Geology, Bates College 2000
YEARS OF EXPERIENCE: 6 months
DUTIES: Organic Preparation Analyst
Assist Sample Custodian

NAME: Allycia Drapeau
TITLE: Analyst
EDUCATION: B.S. Ocean Studies, Maine Maritime Acedemy,2000

YEARS OF EXPERIENCE: 6 months
DUTIES: Organic Preparation Analyst
Assist as Sample Custodian

NAME: Joshua Davenport
TITLE: Computer Specialist
EDUCATION: B.A. Biology, College of Wooster Ohio, 1995
YEARS OF EXPERIENCE: 1 year
DUTIES: Computer Specialist
Assist as Sample Custodian

NAME: Virginia Cusa
TITLE: Personnel Administrator, Bookkeeper
EDUCATION: B.A. Business Administration, Pine Manor College 1986
YEARS OF EXPERIENCE: 6 months
DUTIES: Personnel Administrator
Bookkeeper
Sample Custodian

2.5 Identification of Approved Signatories

The following individuals are authorized to sign laboratory reports:

Stephen L. Knollmeyer
Melissa A. Gulli

3.0 Procedures for Record Management

The laboratory has implemented a record management system, which allows the historical reconstruction of all laboratory activities. The laboratory will keep a record of each environmental analysis for not less than five years. The following records will be maintained by the laboratory:

a) Personnel records

b) Equipment, and reference materials:

1) The name of the item;

2) The manufacture's name, identification and serial number;

3) Date received and date placed in service;

4) Condition when placed in service (new, used, re-conditioned);

5) Current location;

6) Location of manufacturer's instruction manual, when available;

7) Details of maintenance; and

8) Calibrations with acceptance criteria.

c) For reagents and reference materials, the laboratory will retain the manufacturer's statement of purity and traceability (if applicable). The date of receipt, storage conditions, date opened (when applicable), and expiration date (when applicable).

d) Preparation of stock and working standard solutions. The laboratory will maintain a preparation log which will include traceability to purchased reagent or reference material, date of preparation, expiration date (if applicable), and name of analyst.

e) Sample collection and handling. The laboratory will maintain sufficient records to demonstrate samples are properly collected, handled, and preserved. This will include:

1) Date, place, and time of sampling and the name of the person who collected the sample;

2) laboratory sample identification and field sample identification (when available);

3) date and time of sample receipt;

4) person responsible for logging the sample;

5) analysis or analytical method requested;

6) laboratory name or place of analysis; and

7) sampling kit code (when applicable).

8) Sample collection forms

9) Chain of custody forms

10) Sample receiving log

11) Documentation of appropriate sample disposal

12) All information regarding sample integrity and sample acceptance as defined under sample collection, preservation, holding times and handling of this document.

f) Analytical procedures. This will include:

1) Sample identification with date and time of sample preparation and analysis. Initials or signature of the analyst involved in preparation of samples and the analyst involved in the analysis of samples

2) The analytical method used

3) Results of the analysis and the raw data generated. This will include calibrations with acceptance criteria and identification of the instrument

4) Results of quality control and acceptance criteria

5) All calculations

6) Validation of data and initial or signature of the data reviewer

g) Internal audits and Quality Assurance Program reviews. All reviews, audits, audit findings, and corrective action taken will be documented by the laboratory.

h) Proficiency testing. The laboratory will maintain all information resulting from the analysis of proficiency testing samples. This will include a copy of the proficiency testing study reporting forms, and documentation of investigations and corrective actions for failed studies.

i) All electronic hardware and software to reconstruct the analytical data will be available at the laboratory.

j) Records of record storage and access of these records.

k) Copies of final reports and amendments.

l) Copies of historical QA documents.

m) Correspondence relating to laboratory activities for a specific project.

All data not generated electronically, will be recorded in a legible manner using permanent ink.

All corrections to records will be made by crossing the error with a single line, the date of the correction will be noted and the individual making the correction will initial or sign the record.

Records will be stored securely and safely to prevent loss or potential tampering.

Computer and electronic systems used by the laboratory for recording, processing, reporting, storage or retrieval of data will comply with sections 8.1 trough 8.11 of EPA document 2185 - Good Automated Laboratory Practices (1995).

3.1 Document Control

a) Document control is an important process for the management of laboratory documents and to ensure the laboratory staff have knowledge of and access to the most current laboratory adopted procedures.
All Documents will follow an uniformed document control procedure as described in **AEL SOP #QA-D002**.

Each QA document will include a title, a revision number, date of creation and page number of total pages. Such as:

Procedures for Record Maintenance
Revision No. 1
Date: 12/09/1998
Page 1 of 10

All QA documents will be approved and signed by the responsible individuals.
The following are QA Documents that are Controlled:
Standard Operating Procedures
QA Manuals

Worksheets, forms and logbooks will be designed to include all information pertinent to the analysis or task performed. Each worksheet, form, and logbook will include an unique identifier. Worksheets and forms will have a revision number and/or revision date.

4.0 Laboratory Equipment, Reagents, Supplies, and Reference Materials

All equipment, reagents, supplies and reference materials necessary for analyses are kept on hand for the specific analysis for which the ANALYTICS Environmental Laboratory, LLC. (AEL) is accredited.

4.1 General

a) Laboratory Equipment

1) All equipment is properly maintained. Procedures for maintenance of equipment are documented in each of the analytical SOPs in Section #10.

2) Any defective equipment or parts will be removed from service and labeled until repaired. Equipment or parts will not be put back in service until the laboratory demonstrates that it is functioning correctly.

3) All routine and non-routine maintenance and repairs are documented in the specific Instrument Log book.

4) Calibration records are maintained for all measuring equipment.

b) Laboratory Support Equipment.

All laboratory support equipment will be calibrated or verified, or both, before being put into service, and on a continuing basis. The procedures for the calibration and verification of the laboratory support equipment are found in each of the analytical SOPs in Section #10.

c) Reagents and supplies.

1) Glassware will be properly cleaned and maintained as specified in the **AEL Glassware Cleaning SOP # QA-M001**. Any cleaning or maintenance requirements specified in the approved test procedure will be followed.

2) Analytical reagent grade materials, if available, will be used by the laboratory.

3) The laboratory will not use prepared reagents, standards, or purchased chemicals outside the expiration date of the material.

4) All stock and standard solution containers will be labeled with content, preparation date, concentration, and initials of analyst preparing the solution.

5) Compressed gases will meet the requirements specified in the approved method.

6) For the preparation of reagents, standards, and rinsing glassware, the laboratory uses laboratory Milli-Q Grade water.

d) Reference materials.

1) To ensure accurate and precise measurements, the laboratory uses reference materials which are traceable to NIST.

2) The laboratory will keep the calibration certificates of reference materials to demonstrate the traceability to NIST.

3) Reference materials (such as class S weights or equivalent, or thermometers), will go through periodic re-certifications which are traceable to NIST.

4) The original containers will be labeled with the date opened and the expiration date (when applicable).

4.2 Listing of Laboratory Equipment and Reference Materials

See Attachment A

4.3 Calibration and Maintenance Procedures and Frequency

CALIBRATION AND MAINTENANCE			
INSTRUMENT	ACTIVITY	FREQUENCY	DOCUMENTATION
Balance	1. Clean 2. Check alignment 3. Service Contract	1. before use 2. before use 3. annual	Work sheet/log book Post service date on balance
Class S Weights	1. Only use for the intended purpose 2. Use plastic forceps to handle 3. Keep in case 4. Re-calibrate	4. Once every 5 years	4. Keep certificate
Thermometers: 1. Glass and electronic 2. Dial thermometers	Check at the temperature used, against a reference NIST certified thermometer	1. Annual for glass and electronic 2. Quarterly for dial thermometers	Calibration factor and date of calibration on thermometer and work sheet/log book and copy of NIST Certificate
pH electrometers	Calibration: 1. pH buffer aliquot are used only once 2. Buffers used for calibration will bracket the pH of the media, reagent, or sample tested.	before use	Work sheet/log book
pH probe	Maintenance: Use manufactures specifications	As needed	Work sheet/log book

Spectrophotometer.	1. Keep cells clean	2. Annual	1. Note in log book
Refrigerators and Freezers	1. Thermometers are immersed in liquid to the appropriate immersion line 2. The thermometers are graduated in increments of 1 C or less	Temperatures are recorded each day in use	Work sheet/Log book

4.4 Description of facilities and services used by the laboratory.

See Attachment B

b) General

1) The laboratory will be kept clean. Attention is given to good housekeeping at all times.

2) The laboratory has adequate lights and ventilation. Laboratory temperature and humidity will be maintain within adequate range for the analysis performed at the laboratory and for the proper operation of instrumentation. Include description of exhaust hoods were applicable.

3) The laboratory has 4400 sq.ft. of work space which is considered sufficient for conducting all laboratories activities.

4) The laboratory has adequate storage space which is considered sufficient to contain and store all needed supplies, reagents, and equipment.

5) Include physical description of disposal facilities of chemicals, radioactive, and biological wastes, were applicable.

6) The laboratory is designed and activities are conducted so sample contamination is avoided.

7) Access to the analytical and sample storage areas are controlled and only approved personnel are allowed in this area. The sample storage area is located adjacent to the login area and access to this area is controlled by the sample custodian. Only authorized personnel are allowed to pass through the login area into the sample storage room. During non-working hours the only door to the sample storage room is locked.

5.0 Procedures for Dealing with Complaints

See Attachment C

6.0 Procedures for Protecting Client Confidentiality and Proprietary Rights

See Attachment D

7.0 Procedures for Personnel Training

To ensure the quality and integrity of the data generated at AEL all employees participate in a mandatory training program.

a) Upon employment all employees receive a copy of the Quality Systems Manual and training in Chemical Hygiene, data confidentiality and data integrity.

b) Before conducting any analysis, each analyst will receive training by another analyst or supervisor who has completed training. An analyst in training will be supervised by an experienced individual.

c) In addition to in-house training, additional annual training may be provided to the analyst in the form of educational courses, and professional seminars.

d) All employees receive bi-weekly to monthly training sessions which cover a variety of laboratory functions to include but not limited to: Safety issues, SOP training, Documentation training, project coordination and Quality assurance issues

e) Analyst training and performance will be considered complete after the analyst has produced a successful initial demonstration of method performance for the analysis for which they are responsible.

f) All training will be documented and kept in the employees training file. At a minimum, documentation will include the name of the analyst, the reference method/SOP, the dates of training, and data of initial demonstration of method performance (if appropriate). To document the training, ANALYTICS uses a Demonstration of Capability Certification Statement.

g) Waste Management Specialists annually receive mandatory 8-hour Department of Transportation (DOT) training.

8.0 Sampling procedures

To ensure the quality of environmental analysis, the laboratory will ensure each sample is properly collected, handled, and preserved. Procedures are in place for the collection of the samples, sample handling, preservation and holding time and for sample acceptance criteria.

8.1 Sampling Equipment, Containers, Preservation, and Holding Times

a) Samples are collected using the following sampling equipment:

FIELD SAMPLING EQUIPMENT

The following items are routinely employed during ground water, surface water, and soil sampling events. Sampling equipment scheduled for use is inspected to assure that the equipment is clean and in good working order. Backup systems intended to support sampling activities are also examined. Batteries are tested and field instruments are calibrated in accordance with the procedures outlined in the manufacturer's specifications.

Water Sampling Equipment:	Peristaltic pump with Teflon® tubing Teflon® bailers Hand operated PVC inertial pump
Soil Sampling Equipment:	Stainless steel soil sampler (12 inch) JMC Environmentalist's Sub Soil Probe
Auxiliary Equipment:	YSI Model 3000 T-L-C Meter ORS Interface Probe Orion Research Model 201, Digital pH Meter Ferro · Track Audio, Visual Magnetic Locator FT-60
Personal Safety Equipment:	Tyvek® suits MSA and NORTH half-face respirators First Aid kits Disposable nitrile gloves Safety glasses Fire Extinguisher (Type ABC)
Miscellaneous Equipment:	Coolers and ice packs, various sizes Zip-Lok storage bags Labels Measuring tapes Hard hats Chains of custody Sample containers Liquinox, brushes, and other cleaning supplies

b) The Table below depicts the containers, preservation and holding times used for 40CFR Part 136 (Wastewater), 40 CFR Part 141 (Drinking Water) and EPA SW-846 Solid and Hazardous waste analyses.

Recommended Containers, Preservation, Storage, and Holding Time

PARAMETER	METHOD	MATRIX	SAMPLE CONTAINER[1]	PRESERVATION[2,3]	HOLDING TIME[4]	VOLUME
Volatile Organics in Water	EPA 624	Water	G, TefSep	Cool 4°C & HCL pH<2 [5,15]	14 days (7 days w/o HCL)	2 - 40mL [6,7]
Volatile Organics in Drinking Water	EPA 524.2	Water	G, TefSep	Cool 4°C & HCL pH<2 [8]	14 days	3 - 60mL[6,7]
Volatile Organics in Water	EPA 8260	Water	G, TefSep	Cool 4°C & HCL pH<2 [5]	14 days	2 - 40mL [6,7]
Volatile Organics	EPA 8260	Solid/Waste	G, TefCap	Cool 4°C	14 days	100g (2 oz)[7]
Purgeable Halocarbons	EPA 601/8021	Water	G, TefSep	Cool 4°C [5]	14 days	2 - 40mL[6,7]
Purgeable Aromatics	EPA 602	Water	G, TefSep	Cool 4°C & HCL pH<2 [5,15]	14 days (7 days w/o HCL)	2 - 40mL[6,7]
Purgeable Aromatics	EPA 8021	Water	G, TefSep	Cool 4°C & HCL pH<2 [5]	14 days	2 - 40mL[6,7]
Purgeable Halocarbons	EPA 8021	Solid/Waste	G, TefCap	Cool 4°C	14 days	100g (2 oz)[7]
Purgeable Aromatics	EPA 8021	Solid/Waste	G, TefCap	Cool 4°C	14 days	100g (2 oz)[7]
Total Gasoline	EPA 8015	Water	G, TefSep	Cool 4°C & HCL pH<2 [5]	14 days[9]	2 - 40mL[6,7]
GRO	Maine DEP 4.2.17	Water	G, TefSep	Cool 4°C & HCL pH<2	14 days	2 - 40mL[6,7]
Total Gasoline	EPA 8015	Solid	G, TefCap	Cool 4°C	14 days[9]	2 oz jar[7]
GRO	Maine DEP 4.2.17	Solid	G, TefCap	Cool 4°C[14]	14 days[14]	2 oz jar[7]
TPH - Any Fuels	EPA 8015	Water	G, TefCap	Cool 4°C & HCL pH<2	7/40 days[9,10]	1 Liter
DRO	Maine DEP 4.1.25	Water	G, TefCap	Cool 4°C & HCL pH<2	7/40 days[10]	1 Liter
TPH - Any Fuels	EPA 8015	Solid	G, TefCap	Cool 4°C	14/40 days[9,10]	2oz jar
DRO	Maine DEP 4.1.25	Solid	G, TefCap	Cool 4°C	14/40 days[10]	2oz jar
Acid/Base Neutral	EPA 625/8270	Water	AG, TefCap	Cool 4°C[5,17,18,20]	7/40 days[10]	1 Liter
Acid/Base Neutral	EPA 8270	Solid/Waste	AG, TefCap	Cool 4°C	14/40 days[10]	4 oz jar
Pesticides	EPA 608/8081	Water	AG, TefCap	Cool 4°C, pH between 5 and 9[21]	7/40 days[10]	1 Liter
PCBs	EPA 608/8082	Water	AG, TefCap	Cool 4°C	7/40 days[10]	1 Liter

PARAMETER	METHOD	MATRIX	SAMPLE CONTAINER[1]	PRESERVATION[2,3]	HOLDING TIME[4]	VOLUME
Pesticides and PCBs	EPA8081/ 8082	Solid/Waste	AG, TefCap	Cool 4°C	14/40 days[10]	4 oz jar
Chlorinated Herbicides	EPA 615/8151	Water	AG, TefCap	Cool 4°C	7/40 days[10]	1 Liter
Chlorinated Herbicides	EPA 8151	Solid/Waste	AG, TefCap	Cool 4°C	14/40 days[10]	4 oz jar
TCLP Analyses	EPA 1311	Solid/Waste	G, TefCap	Cool 4°C	VOA: 14/NA/14[11] ABN/Pest/Herb: 14/7/40 Metals: 180/NA/180 Mercury: 28/NA/180	500 g- solid 7 L - water[6]
Metals: (For all metals except Chromium VI and Mercury)		Water	P, G/T	HNO_3 to pH<2	6 months	500 mL
Chromium, Hexavalent	7197 / 218.4	Water/Solid	P, G/T	Cool 4°C	24 hours	200 mL (2g)
Mercury (CVAA)	7470/71 / 245.1/5	Water	P,G/T	HNO_3 to pH<2	28 days	500 mL
EP Toxicity	EPA 1310	Solid	P, G/T	Cool 4°C	180 days	100g
RCRA Metals (Total Metals)[12]		Water/Solid	P, G/T	HNO_3 to pH<2	Hg: 28 days Other metals :180 days	500mL (15g)
Priority Pollutant (13 Metals) [12]		Water/Solid	P, G/T	HNO_3 to pH<2	6 months	1 Liter (25g)
Acidity	EPA 305.1	Water/Solid	P,G/T	Cool, 4°C	14 days	100 mL (2g)
Alkalinity	SM18 2320B	Water/Solid	P,G/T	Cool, 4°C	14 days	100 mL (2g)
Available Potassium	AOAC 2.044/6B EPA 258.1	Water	P,G	Cool, 4°C	28 days	100 mL
BOD_5	EPA 405.1	Water	P, G/T	Cool, 4°C	48 hours	1 Liter
Boron	EPA 212.3	Water	P only	Cool, 4°C	180 days	100 mL
$CACO_3$ Equivalence	SM17 2340B	Water	P,G	HNO_3 to pH<2	6 months	100 mL
Chloride	EPA 325.3	Water	P, G/T	none req.	28 days	100 mL
COD	EPA 410.1	Water	G/T	Cool, 4°C, H_2SO_4 to pH<2	28 days	100 mL
Conductivity	EPA 120.1/9050	Water	P,G/T	Cool, 4°C	28 days	100 mL
Corrosivity	SW-846 9045 EPA 150.1	Water/Solid Water	P,G/T	Cool, 4°C	ASAP[13]	100g (8 oz jar)
Cyanide, total	EPA 335.2/9010	Water/Solid	P, G/T	Cool, 4°C, NaOH to pH>12 0.6g ascorbic acid[16]	14 days[13]	500mL (25g)

Flash Point	EPA 1010	Water/Solid	G, TefCap	Cool, 4°C	14 days	4 oz.
Fluoride	EPA 340.2	Water	P	none req.	28 days	100 mL
PARAMETER	METHOD	MATRIX	SAMPLE CONTAINER[1]	PRESERVATION[2,3]	HOLDING TIME[4]	VOLUME
Hardness	SM17 2340B	Water	P, G/T	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months	100 mL
Nitrogen						
Nitrate	EPA 352.1	Water/Solid	P, G/T	Cool, 4°C	48 hours	500mL (20g)
Nitrite	EPA 354.1	Water	P, G/T	Cool, 4°C	48 hours	500 mL
Nitrate-Nitrite	EPA 353.2	Water	P, G/T	Cool, 4°C H_2SO_4 to pH<2	28 days	500 mL
Ammonia	EPA 350.3	Water	G/T	Cool, 4°C, H_2SO_4 to pH<2	28 days	500 mL
Organic	EPA 351.4/350.3	Water	P, G/T	Cool, 4°C, H_2SO_4 to pH<2	28 days	500 mL
Kjeldahl	EPA 351.4	Water	P, G/T	Cool, 4°C, H_2SO_4 to pH<2	28 days	500 mL
Oil & Grease	SW-846 9070 EPA 413.1	Water	G/T	Cool, 4°C, HCL or H_2SO_4 to pH<2	28 days	1 Liter
Paint Filter Liquids Test	SW-846 9095	Water	G, P	Cool, 4°C	6 months	500 mL
Phenols - Total	EPA 420.1	Water	G/T	Cool, 4°C, H_2SO_4 to pH<2	28 days	500 mL
Phosphorus						
Total Phosphorus	EPA 365.3	Water	P,G/T	Cool, 4°C, H_2SO_4 to pH<2	28 days	250 mL
Orthophosphate	EPA 365.3	Water	P,G/T	Filter Immediately, Cool, 4°C	48 hours	250 mL
Available Phosphorus	AOAC 2.044/6B	Water	P,G/T	Cool, 4°C	28 days	250 mL
Reactivity		Water				
Cyanide (solids)	SW-846 7.3.3.2	Water/Solid	P,G	Cool, 4°C	7 days	100mL(100g)
Sulfide (solids)	SW-846 7.3.4.2	Water/Solid	P, G	Cool, 4°C	7 days	100mL(100g)
Solids (Residue)						
Total	EPA 160.3	Water	P, G	Cool, 4°C	7 days	100 mL
Volatile	EPA 160.4	Water	P, G	Cool, 4°C	7 days	100 mL
Suspended (TSS)	EPA 160.2	Water	P, G	Cool, 4°C	7 days	100 mL
Dissolved (Filterable)	EPA 160.1	Water	P, G	Cool, 4°C	7 days	100 mL
Settleable	EPA 160.5	Water	P, G	Cool, 4°C	48 hours	100 mL
Sulfate	SM18 4500S04D	Water	P, G/T	Cool, 4°C	28 days	250 mL
Sulfide	EPA 376	Water	P, G/T	Cool, 4°C, ZnAc and NaOH to pH >9	7 days	500 mL
Surfactants (MBAS)	EPA 425.1	Water	P, G/T	Cool, 4°C	48 hours	500 mL
Total Organic Carbon (TOC)	EPA 415.1	Water	P,G/T	Cool, 4°C, H_2SO_4, HCL or H_3PO_4 to pH<2	28 days	500 mL or 8 oz jar
Total Organic Halides (TOX)	EPA 9020	Water	AG, TefSep	Cool, 4°C, H_2SO_4 to pH<2	7 days	250 mL [7] or 4oz jar

Notes

1. G = glass; AG = amber glass; P = polyethylene; TefSep = Teflon septum; TefCap = Teflon Cap.

2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at $4^{o}C$ until compositing and sample splitting is completed.

3. When any sample is to be shipped by common carrier or sent through the United Stated Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulation do not apply to the following materials: Hydrochloric acid (HCL) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions of concentration of 0.080% by weight or less (pH about 12.30 or less).

4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time.

5. 0.008% Sodium Thiosulfate ($Na_2S_2O_3$) should only be used in the presence of residual chlorine.

6. Samples must be provided in duplicate or triplicate to cover for breakage and to provide sufficient sample for QC procedures.

7. Fill completely to avoid volatile loss.

8. If samples contain residual chlorine, and measurements of the concentrations of disinfection by-products (trihalomethanes, etc.) at the time of sample collection are desired, add about 25 mg of ascorbic acid to the 60 mL sample bottle before filling.

9. EPA does not specifically mention TPH holding times. The holding time given is by analogy to the methodology used (i.e. extractable organics or volatile organics).

10. 7 days from sampling date for extraction, 40 days from extraction date for analysis. 14 days until extraction is listed as 14/40.

11. TCLP Sample Maximum Holding Times are expressed as follows: # of days from field collection to TCLP extraction / from TCLP extraction to preparative extraction / from preparative extraction to determinative analysis. NA = Not applicable. If sample-holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

12. 8 RCRA Metals: Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, and Silver.
13 Priority Pollutant Metals: Antimony, Arsenic, Beryllium, Cadmium, Chromium, Copper, Mercury, Nickel, Selenium, Silver, Lead, Thallium, and Zinc.

13. Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH>12.

14. Samples may be preserved in the field with methanol. The weight of the soil to the methanol should be 1:1 with a minimum soil weight of 10 grams. Both the weight of the soil and volume of methanol used must be provided to the laboratory.

15. Sample receiving no pH adjustment must be analyzed within seven days of sampling.

16. Should only be used in the presence of residual chlorine.

17. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 18, 19 (re the analysis of benzidine).

21. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection.

b) A sample collection form shall be completed for each sampling event. This form shall contain sampling location, date and time of collection, collector's name, method of preservation, and special remarks concerning the sample.

Analytics Field Data Record

Project Name | Date

Address | Time

Project # | Sampler

Sample ID	Preservation	pH	Specific Conductance	Sample Depth	Description of Location

QC Sample	Preservation	pH	Specific Conductance	Sample Depth	Description of Location

d) The laboratory will use a chain of custody procedure from collection through analysis and disposal which can document every person who handled the sample. A copy of the Chain of Custody Form is located in Attachment F.

8.2 Sampling Collection Procedures

An essential part of the sampling event is the clear documentation of the collection procedures, sample identification, and chain of possession of the sample. This information is recorded on a field data record, Chain of Custody form, and sample labels.

Field Data Record: A field data record (Figure 7.1) is filled out at the time of sample collection. Additional pertinent information is recorded in the comments section.

Chain of Custody: The Chain of Custody is completed at the time of sample collection with the following information: client name and address; project name and number; sampler's signature; sample designation; date and time of sample collection; sampling location; number of samples collected at each location; matrix; type of analysis to be performed for each sample; type of preservation; and any other comments or pertinent information. Replicate samples will be designated as such.

When samples are released into the custody of transporters and/or other laboratories, each exchange will have the signatures of both the relinquisher and the recipient, and the date and time of the exchange.

Samples that exhibit unusual characteristics (i.e. reactivity), or evidence of high contamination are labeled as such to prevent undue exposure to conditions that may jeopardize health of the sample or analytical staff or the collection of high quality data. All pertinent information is recorded on the Chain of Custody document depicted above.

8.3 Sample Handling Procedures Including Sub-sampling and Sample Acceptance Criteria

a) Each sample is uniquely identified from collection through disposal and the procedures are contained in **AEL SOP #QA-L001 and QA-L005.**

b) After sample collection and transportation to the facility, the laboratory will check the integrity of the sample by checking the following items:

1) Leakage or breakage.

2) Completeness of sample collection forms.

3) Correct sample identification.

4) Appropriate use of sample labels (such as water resistant) and use of indelible ink.

5) Use of appropriate sample containers, adequate volume, preservation, and holding time as required by section 8.1.

6) Temperature of samples requiring thermal preservation will be checked and recorded.

7) Chemical preservation is checked prior to or during sample preparation or analysis. Results will be recorded.

c) Procedures for handling samples that are improperly collected, preserved or stored are located in **AEL SOP #QA-L001.**

d) Samples analyzed by the laboratory not meeting the sample acceptance requirements will be qualified on the final report.

e) After samples are checked for integrity and sampling forms checked for completeness, the samples are logged in the sample work sheet /log book.

8.4 Storage of Samples in the Laboratory

a) The laboratory will store samples, sub-samples, extracts, and digestates according to the specified conditions in the approved methodology. All samples, sub-samples, and extracts will be protected from all potential sources of contamination.

AEL SOP #QA-L001 includes specific requirements for sample storage and locations.

8.5 Sample Disposal

AEL SOP # QA-W001 includes procedures for sample and extract disposal.

8.6 Forwarding Samples to Another Laboratory for Analysis

When the laboratory forwards collected samples to another laboratory for analysis, a chain of custody form will be used.

When the laboratory forwards collected samples to another laboratory for analysis, the samples must be forwarded to a laboratory holding NELAC accreditation for that analysis.

9.0 Calibration Procedures of Analytical Instrumentation and Frequency

For calibrations of analytical instrumentation, the laboratory will use NIST traceable standards (when available).

Calibration procedures are established for all applicable tests. These procedures are detailed in the SOP for the analysis. The calibration requirements of the USEPA approved procedures are followed by the laboratory.

Many AEL SOPs contain the calibration information specific to the methods. Attachment F contains a list of the SOPs which are underlined for NELAC SOPS and are marked with a "*" for those SOPs that contain calibration information.

10.0 Analytical Methods

Drinking water samples analyzed under the safe drinking water act will be analyzed in accordance with USEPA approved methods.

Wastewater samples analyzed under the clean water act will be analyzed in accordance with USEPA approved methods.

10.1 List of Analytical Tests, Parameters, Method Reference, MDL, and Reporting Limits

SEE Attachment

10.2 Written Procedures for Conducting Analytical Tests

A written procedure for conducting each of the analytical tests performed at the laboratory is available.

10.3 Written Procedures for Conducting Method Validation and/or Initial Demonstration of Performance

a) Prior to implementation of a method, the laboratory will prepare an initial demonstration of method performance in accordance with method specification. When the USEPA approved method does not specify initial demonstration of performance, the laboratory will use the following guidelines:

1) Determination of Method Detection Limits
2) Precision
3) Bias
b) Initial demonstration of method performance will be repeated each time significant changes are made to instrumentation, personnel, or the method. Initial demonstration of performance will be documented. The documentation will include the following initial demonstration of capability certificate:

See Attachment G

c) The process for conducting method validation and/or initial demonstration of performance, are included in the laboratory written procedure for each analysis.

11.0 Internal Quality Control Procedures

11.1 Internal Quality Control Procedures and Frequency of Use

The laboratory will demonstrate the quality of analytical results through the implementation of an internal quality control plan.

a) Chemical Testing

1) Method Blanks. Method blanks will be performed to demonstrate the analytical system is not contaminated. When the result of a method blank exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated batch and 1/10 of the regulatory limit, the laboratory will minimize or eliminate the contamination problem (when possible) and determine the effect of a contaminated method blank on the samples tested. Any samples affected by a contaminated method blank will be re-analyzed (when possible) or sample results will be qualified.

2) Matrix spikes. Matrix spikes will be used to determine analytical accuracy and potential matrix interference. The laboratory will calculate percent recovery and acceptance criteria will be established per matrix. Sample result will be qualified when matrix spike recovery is outside the laboratory's acceptance criteria.

The following calculation will be used to determine Percent Recovery:

$$\%R = [(SSR-SR)/SA] * 100$$

SSR = SPIKED SAMPLE RESULT
SR = SAMPLE RESULT
SA = SPIKE ADDED

Note: Laboratories testing samples for clients, matrix spikes will be rotated among clients.

3) Laboratory control samples. Laboratory control samples will be used to determine the accuracy of the analysis. The laboratory will calculate percent recovery and establish acceptance criteria. The following calculation will be used to determine Percent Recovery:

$$\%R = (CONCENTRATION\ FOUND \div TRUE\ CONCENTRATION) * 100$$

Note: Matrix spikes can be used in place of laboratory control samples.

4) Matrix spike duplicates or laboratory duplicates. Matrix spike duplicates or laboratory duplicates will be used to determine analytical precision. The laboratory will calculate relative percent difference or percent difference. Acceptance criteria shall be established. Sample result will be qualified when relative percent difference or percent difference are outside the laboratory's acceptance criteria. The following calculation will be used to determine percent difference or relative percent difference:

$$\%D = [|S-D| \div ((S+D) \div 2)] * 100$$

S = FIRST SAMPLE RESULT (ORIGINAL)
D = SECOND SAMPLE RESULT (DUPLICATE)
|S-D| = ABSOLUTE DIFFERENCE

$$RPD = [|MSR - MSDR| \div ((MSR + MSDR) \div 2)] * 100$$

MSR = MATRIX SPIKE RESULT
MSDR = MATRIX SPIKE DUPLICATE RESULT

Note: Laboratories testing samples for clients, the matrix spike duplicates or laboratory duplicates will be rotated among clients.

5) Surrogates. For the analysis of organic compounds, surrogate compounds shall be spiked into each sample, each standard, and each blank. Percent recoveries will be calculated and acceptance criteria will be established.

6) Method blanks, matrix spikes, laboratory control samples, and matrix spike duplicates or laboratory duplicates will be performed at a frequency of one per batch.

7) When the approved method does not specify the spiking requirements, the laboratory will spike samples with all parameters of interest for a project or permit.

8) Method Detection Limit (MDL) will be calculated according to 40 CFR Part 136, Appendix B (October 26, 1984) or according to method specifications. Once per year, the laboratory will verify calculated MDL with at least one clean matrix sample spiked at the current MDL concentration. If the current MDL concentration cannot be verified, the laboratory will conduct a new MDL study. Reporting limits will be set at least three times the calculated MDL. The laboratory will document the matrix used for MDL studies and MDL verifications.

9) Selectivity. For the analysis of organic compounds, the laboratory will develop and document acceptance criteria for retention time windows as determined by the approved method. When mass spectrometers are used for the analysis of samples, ANALYTICS will follow the acceptance criteria outlined in the specific method being used. Confirmation is required for all positive results on samples from locations where previous data is not available. Confirmation is not required when mass spectrometers are used for the analysis.

Note: Matrix spikes and determination of MDL's are not required when determining physical properties of a sample or for the analysis of residues, dissolved oxygen, and pH.

11.2 Procedures to Determine Acceptance Criteria.

a) Precision is used as a data quality indicator to determine the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

b) ANALYTICS will determine precision through the use of duplicates and/or matrix spike duplicates. Percent difference (%D) between duplicates and Relative Percent Differences (RPD) between matrix spike duplicates will be calculated.

c) Acceptance criteria for duplicate analysis will be established at a warning limit of two standard deviations, and a control limit of three standard deviations of the %D or RPD. Acceptance criteria will be monitored through the use of control charts. (See Attachment H)
d) Accuracy is used as a data quality indicator to determine the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations.

e) ANALYTICS will determine accuracy through the use of standards of known concentration.

Percent Recoveries (%R) will be calculated.

Acceptance criteria for LCS, MS, and surrogates will be established using warning limits set at two standard deviations, and control limits set at three standard deviations of the %R. Acceptance criteria will be monitored through the use of control charts. (See Attachment I)

f) Acceptance Criteria will be established using the following calculations:

Calculate the average of at least 20 data points.

$$\text{mean} = \frac{x}{n}$$

Calculate the Standard Deviation:

$$SD = \sqrt{\frac{(x - \text{mean})^2}{(n - 1)}}$$

Where:

SD = Standard deviation
x = each data point
mean= average of all data points
n = number of data points

Calculate the Upper Control Limit (UCL) and the Lower Control Limit (LCL) as ± 3 Standard Deviations:

UCL = mean + 3 SD
LCL = mean - 3 SD

Calculate the Upper Warning Limit (UCL) and the Lower Warning Limit (LCL) as ± 2 Standard Deviations:

UWL = mean + 2 SD
LWL = mean - 2 SD

Prepare control charts.

11.3 Acceptance Criteria Reviews

Quality control charts will be updated daily.

Acceptance criteria will be reviewed periodically to determine its adequacy.

12.0 Data Reduction and Reporting

12.1 Procedures for Data Reduction
Procedures for data review and data reduction are contained in AEL SOP #QA-D001.

12.2 Reporting Procedures and Format

All data is reported with all of the following sample information:

a) Report title, name of the laboratory, and accreditation number;

b) Location of sample test;

c) Contact person and phone number at the laboratory;

d) Unique identification of the report;

e) Report pages shall be numbered. The laboratory shall provide the total number of pages on the report.

f) Identification of the client or project name, or both (when applicable),

g) Identification of the sample with field sample number (when available),

h) Description of the sample (water, soil, sludge),

i) Date the sample was received,

j) Date and time of collection,

k) Date(s) of analysis,

l) Time of analysis,

m) Identification of the method used or method number,

n) Reference to sampling procedure when relevant,

o) Data qualifiers to describe analytical conditions,

p) Analytical data is reported in units consistent with monitoring program requirements,

q) Signature and title of the person accepting responsibility for the content of the report,

r) Date issued,

s) Any reports issued by the laboratory with amendments to a previous report, shall be clearly identified as an amended report.

See Attachment J for an example ANALYTICS Report Form

Note: When reporting directly to the state on DES reporting form, the above requirements are not applicable. In this case include a copy of the reporting forms and any additional information about reporting as applicable. The laboratory may report and sign forms for only data generated by the laboratory.

If the laboratory subcontracts part or all of the sample to another laboratory:

a) The test report must indicate any subcontracted data if the data is submitted on the laboratory's reporting form.

b) Only the laboratory actually doing the analysis will sign state forms; copies of the state form will be provided to each subcontracted laboratory, as needed.

12.3 Procedures to Ensure Reported Data Is Free from Errors. Data Validation.

All data is first reviewed by the analyst performing the analysis. The data review will include the following items:

a) Calibration of the instrumentation.

b) Quality control data. Confirm QC meets the acceptance criteria.

c) Calculations. Check for calculation errors.
d) Documentation. Check worksheets/logbooks and printouts for completeness.

Before final reporting is done, data will be reviewed by the QAO or laboratory director described in ANALYTICS **SOP #QA-D001**.

12.4 Procedures for Data Qualifiers. Data qualifiers will be added to all data not meeting collection, analytical or internal QC acceptance criteria.

13.0 Proficiency testing

13.1 The type and frequency of external proficiency testing studies

The laboratory will obtain PT studies from either Accustandard or APG Standards.

PT studies will be performed twice per year at the times scheduled by NH ELAP. The following parameters will be tested:

Drinking Water PT Studies
Method 524 Volatile Organic Compounds
Method 504 EDB, DBCP and 123TCP
pH
Wastewater PT Studies
Method 624 Volatile Organic Compounds
Method 625 Semivolatile Organic Compounds
Method 608 Pesticides and PCBs
PCB's in Oil
pH

PT studies will be analyzed in the same manner and at the same frequency as regular samples.

The same calibration procedures and the same internal QC protocol will be used when analyzing PT studies.

13.2 Procedures for the determination of the cause of a failed study and procedures for corrective action

If the laboratory fails a PT study, an investigation of the cause will be conducted.

Documentation of the PT analysis will be reviewed and the way the analysis was conducted will be investigated. When problems are identified, a corrective action plan will be outlined and put in place in a timely manner.

A corrective action letter will be prepared and sent to NH ELAP within 30 days of receiving the unacceptable results.

14.0 Corrective action

a) The laboratory will take corrective action whenever unacceptable conditions exist. The following indicators will be used to determine unacceptable conditions:

1) QC samples outside of the established acceptance criteria

2) Calibrations outside acceptable criteria

3) Equipment failure

4) PT studies outside acceptable limits

5) Deficiencies identified during internal reviews

6) Deficiencies identified during NH ELAP's on-site assessments

7) Deficiencies or problems identified after receiving a complaint

b) Once an unacceptable condition is identified, the laboratory will investigate the problem and outline a corrective action plan.

c) Corrective action may include any of the following:

1) Re-analysis of samples

2) Re-calculation of results

3) Re-calibration of instrument

4) Preparation of new standards

5) Re-analysis of blanks

6) Dilution of samples

7) Additional analyst training

8) Replace equipment or supplies

9) Re-sampling

d) All corrective actions taken by the laboratory will be documented.

e) If the laboratory determines the validity of the data are compromised, the laboratory will take corrective action and make written notification to the clients affected.

SPECIFIC CORRECTIVE ACTION		
TYPE	RECOMMENDED ACTION	DOCUMENTATION
Contaminated Method Blank	1. Determine source of contamination. 2. Eliminate source of contamination. 3. Re-analyze blank.	Work sheet/log book
LCS outside acceptance limit	1. Check preparation log for errors 2. Check analysis for errors 3. Check calculations 4. Remake standard or use a different standard 5. Re-analyze standard and all affected samples	Work sheet/ log book
Positive/Negative controls	1. Check expiration date of the media 2. Check media preparation 3. Confirm incubator temperatures 4. Prepare new media from same lot, if still not acceptable, prepare new media from different lot 5. Examine analytical technique	Work sheet / log book
Analyst not following the SOP	1. Provide additional training 2. Do demonstration of performance 3. Analyze a PT sample	Analyst training file Work sheet/ log book

15.0 Internal Audits

15.1 Internal Audits of Quality Assurance Plan

The laboratory will conduct annual internal audits to verify that its operations continue to comply with the requirements of this Quality Assurance Plan. Such audits will be conducted by the quality assurance officer or designee(s). Where the audit identifies problems with correctness or validity of the laboratory's calibrations or test results, the laboratory will implement a corrective action to solve the problems.

15.2 Performance Audits

On an on-going basis, ANALYTICS will ensure the quality of the data by implementing the following checks:

The analytical SOPs listed in Attachment F contain the specific procedures for checking the quality of the analytical data and include but are not limited to:

1. Internal quality control procedures using statistical charts;
2. Participation in proficiency testing or other inter laboratory comparisons;
3. Use of certified reference materials and/or in-house quality control using secondary reference materials;
4. Replicate testing using the same or different test methods;
5. Re-testing of retained samples;
6. Correlation of results for different parameters of a sample (for example, total phosphorus should be greater than or equal to orthophosphate).

15.3 Documentation of Audit/Review

All audit and review findings and any corrective actions that arise from them will be documented.

The laboratory management will ensure that these actions are discharged within the agreed time frame.

Where the audit or review identifies problems with correctness or validity of the data, the laboratory will immediately notify, in writing, the client whose work may have been affected.

16.0 Procedures Used by the Laboratory Which Are Not Part of the Approved Method

(NOT APPLICABLE)

ATTACHMENT A

Laboratory Equipment

Item Description	Manufacturer and Model	Serial #	Yr. Purchased
Aggitator,TCLP-2	Associated Design and Manufacturing		1990
Air Compressor-1	Sears 3/4 HP		1990
Auto Mixers-6	Various		
Balance, Analytical-1	Mettler AE 200		1988
Balance, Top Loading-1	Ohaus CT200-S	15524	1992
Balance, Top Loading-1	Ohaus CT200-S	20129	1993
Balance, Top Loading-1	Denver Instrument XL-610	79938	1996
Concentration Workstation-1	Zymark Turbo VapII	TV9421N4053	1994
Concentration Workstation-1	Zymark Turbo VapII	TV9706R7310	1995
Copier,Xerox-1	Xerox 5009 R/E	44277	1994
Deionizer-1	Barnstead Nanopure II	8712078	1988
Dishwasher-1	Labconco Steamscrubber		1988
Extractor, Zeroheadspace-9	Associated Design & Manufacturing		1988
Filtering Apparatus-1	Millipore		1988
Filtering Apparatus-1	Associated Design and Manufacturing	24-890564	1990
Flashpoint Detector-1	Precision Scientific		1990 & 1991
Flowmeter-1	Optiflow 520		1993
Fume Hood,portable-1	Captair5000C		1993
Fume Hood,portable-1	Captair5000C	24625	1993
Fume hood,velocity monitor-1	Kewaunee		1993
Fume hood,velocity monitor-2	St. Charles		1993
Fume Hood-1	Kewaunee		1988
Fume Hood-1	Kewaunee		1990
Fume Hood-1	Kewaunee		1991
Ohmeter-1	Micronta 22-185		1990
Oven,sample-1	VWR 1305U	204490	1988
Oven,toaster-1	Black & Decker B2S02500		1992
pH Meter,portable-1	Orion 230A	OO1080	1990
Refrigerator,Sample-1	General Electric		1988
Refrigerator,Sample-1	Gibson		1992
Refrigerator,Sample-1	Summit	B91200102	1992
Refrigerator,Sample-2	White Westinghouse		1989 &1992
Refrigerator,Sample-2	Whirlpool		1990 & 1991
Refrigerator,Sample-2	Hotpoint		1991 & 1994
Sonicator & Sonibox-1	Heat Systems XL 2020	G2409	1993
Sonicator-1	Branson 2200	B2200R-2	1989
Still, glass-1	Barnstead D0603	8803081	1988
Vacuum pumps-6	Edwards		
Velometer-1	Alnor Velometer,jr		1993
Oven	VWR 1300U	202098	1998
Water baths,sm-2	VWR		

System Identifier: A **Volatile Screening/Air** **4004**

Item Description	Manufacturer and Model	Serial # Yr.	Purchased
Gas Chromatograph	Hewlett Packard 5890	2750A17207	1988
Flame Ionization Detector(FID)	Hewlett Packard		
TDX EZ Flash			
Personal Computer	Dell Optiplex (shares with D)	982903	1999

System Identifier: B **Volatile** **4004**

Item Description	Manufacturer and Model	Serial # Yr.	Purchased
Mass Spectrometer	Hewlett Packard 5970	2807A11068	1988
Gas Chromatograph	Hewlett Packard 5890	2750A17208	1988
Purge and Trap	Tekmar 3000	96060007	1996
Autosampler	Tekmar Precept	96198003	1996
Personal Computer	Dell Optiplex GTI	H4SHC 287-770-08	1999

System Identifier: C **Volatile** **4004**

Item Description	Manufacturer and Model	Serial # Yr.	Purchased
Mass Spectrometer	Hewlett Packard 5971A	3050A01856	1991
Gas Chromatograph	Hewlett Packard 5890 Series II	3033A33637	1991
Purge and Trap	Tekmar LSC-2000	89208001	1992
Autosampler	Tekmar ALS 2016	92163001	1992
Automatic Sample Heater	Tekmar	91289007	1992
Personal Computer	Dell Optiplex GXI	H4SH3 287-769-99	1999

System Identifier: D **Semi-Volatile** **4015**

Item Description	Manufacturer and Model	Serial # Yr.	Purchased
Mass Spectrometer	Hewlett Packard 5972A	3501A02385	1995
Gas Chromatograph	Hewlett Packard 5890 Series II Plus	3336A58810	1995
Robotic Autosampler	Hewlett Packard 7673A	2704A09852	1988
w/ controller		2803A11019	1988
Personal Computer	Dell Optiplex GXT	H4SH2 287-769-98	1999

System Identifier: E **GAS/GRO** **4008**

Item Description	Manufacturer and Model	Serial # Yr.	Purchased
Gas Chromatograph	Hewlett Packard 5890	2750A17209	1988
FID/PID	OI Corp 4430	S2163	1992
Personal Computer	Shares with F		1999
OI 4560 Sample Concentrator			
OI MAM-16			
OI MAC-16			

System Identifier: F **Organochlorine** **4020**

Item Description	Manufacturer and Model	Serial #	Yr. Purchased
Gas Chromatograph	Hewlett Packard 5890 Series II	3140A38807	1991
Electron Capture Detector(ECD) X2	HP 19233, G1223A	M2597,F1595	1991
Personal Computer			1999
Robotic Autosampler w/controller	Hewlett Packard 7673	3225A31440 3229A28898	

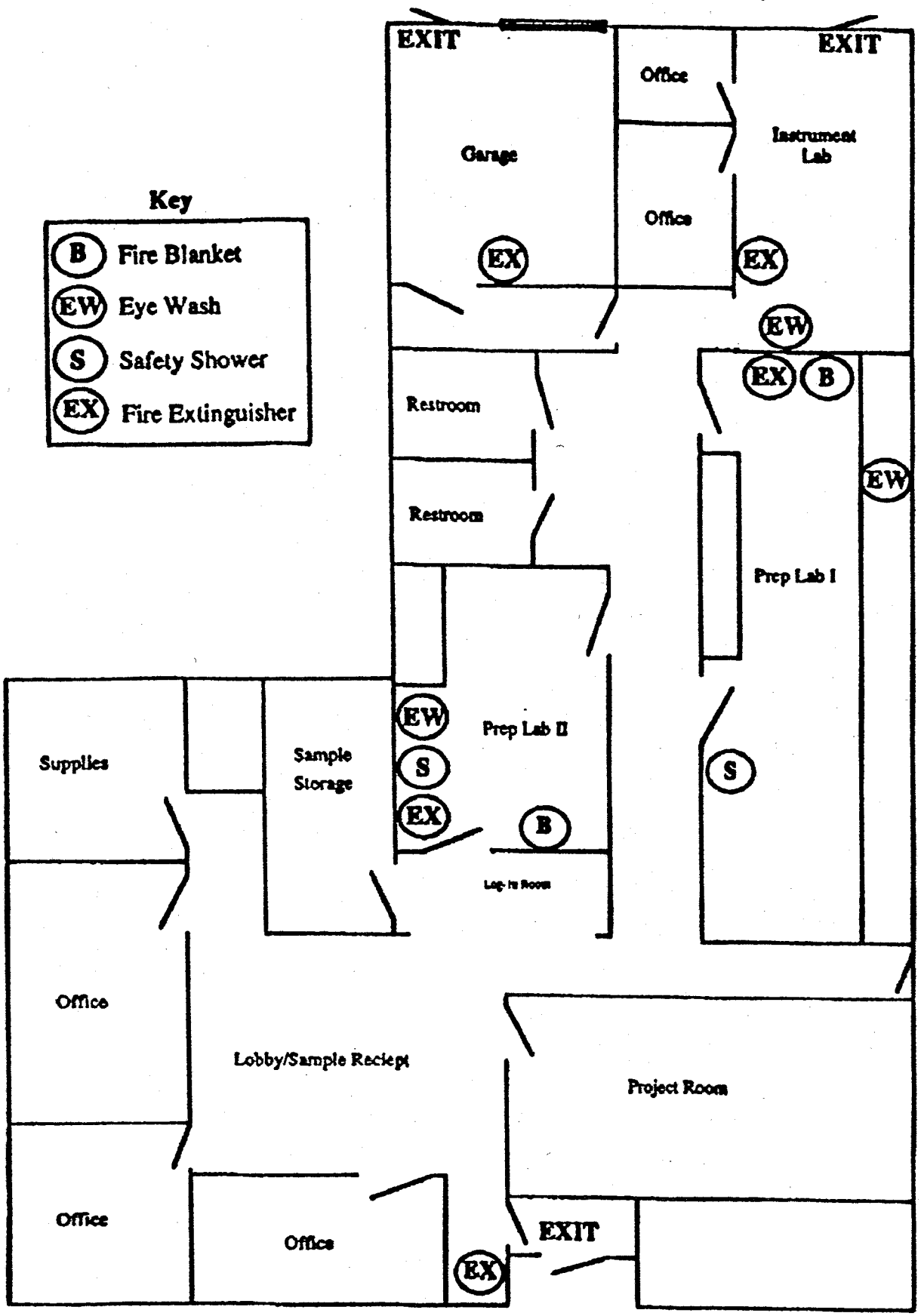
System Identifier: G **TPH/DRO** **4010**

Item Description	Manufacturer and Model	Serial #	Yr. Purchased
Gas Chromatograph (FID)	Hewlett Packard 5890 Series II	3223A42757	1991
Robotic Autosamlper w/ controller	Hewlett Packard 7673	3230A301499 3229A29234	1992
Personal Computer			1999

ATTACHMENT B

ANALYTICS ENV.LABORATORY LAYOUT

Location of Fire Exits, Extinguishers, Blankets, Eye Washes, and Safety Showers



ATTACHMENT C

analytics environmental laboratory inc.

195 Commerce Way
Portsmouth, New Hampshire 03801
603-436-5111 Fax 603-430-2151
800-929-9906

TO: All Laboratory Personnel
FROM: Steve Knollmeyer
DATE: 11/2/99

RE: Policy for Handling Complaints from Clients

Please review this policy for handling complaints from clients.

This policy has been written to cover complaints from outside clients or other entities concerning the analytical activities of ANALYTICS. Complaints are defined as any questions as to the precision, accuracy or correctness of any of the information reported by the laboratory. An example of such a complaint would be a client who feels a compound reported was not actually present in their sample. The following steps shall be followed by ANALYTICS when such instances arise.

1. Initial Contact. Most complaints will be in the form of a phone call from some entity. The person taking the phone call shall direct the call to the Laboratory Director if available. If the Laboratory Director is not available, then a detailed message should be taken and given to the Laboratory Director at the earliest possible time. If the person with the complaint insists on talking to a person, then the call should be directed to the Laboratory Supervisor.

2. Initial Information. In all cases, the first step in a complaint is to obtain information from the client and then conduct a formal evaluation of the information and respond in a formal manner. The information collected shall include; name, title, affiliation, address, phone number and fax number of person with the complaint, nature of the complaint, AEL lab # for all reports involved and reasons for the complaint. If the person calling is not the client the laboratory performed the work for, then we must have authorization from the client to release any information.

3. Evaluation of Complaint. When sufficient information is obtained, then a formal evaluation of the data will be conducted by the QA Officer as follows:

a. Review reported data against the raw data to check for transcription errors.

b. Review all associated QC including blanks, calibrations, matrix spikes and lab control samples.

c. Check for station contamination on the instrument and/or carry over.

d. Check to see if the sample ran more than once at different dilutions. If so, then check to see that all the data correlate.

ATTACHMENT D

analytics environmental laboratory inc.

195 Commerce Way
Portsmouth, New Hampshire 03801
603-436-5111 Fax 603-430-2151
800-929-9906

TO: All Laboratory Personnel
FROM: Steve Knollmeyer, Laboratory Director
DATE: 11/2/99

RE: Policy on Client Confidentiality

Please review this policy for client confidentiality.

This policy has been written to ensure client confidentiality concerning the analytical activities of ANALYTICS.

ANALYTICS has a strict policy of client confidentiality. Employees of ANALYTICS are privy to confidential information during the course of sample receipt, analysis and reporting. It is of the utmost importance for all employees to maintain complete confidentiality of this information.

"Confidential Information" shall mean any confidential or proprietary information that is disclosed to the laboratory employees including but not limited to patents, patent applications, trade secrets, ideas, creations, technical information, business or marketing plans, customer information, analytical results, sample information or information on any environmental activities.

By signing this policy statement, all employees agree to maintain complete confidentiality and shall, not disclose, directly or indirectly, to any third part any portion of the Confidential Information without the prior written consent of ANALYTICS and not use or exploit the Confidential Information.

Signed:

_____ _____
Signature Date

_____
Printed Name

e. Check mass spectra to ensure that the correct identification was made.

f. If the sample containers are still in-house, then the corrected labeling is verified.

4. Reporting of Results of Evaluation The results of the evaluation are reported to the Laboratory Director by the QA Officer. The Laboratory Director communicates the information in writing in the form of a narrative to the client. If the results of the evaluation show that the original data was correct then supporting information is provided. If the data is incorrect, then changes may be made to the report and a revised report issued. If changes can not be made, then resampling and reanalysis will be suggested if possible. The narrative is sent to all parties affected by the data and is filed in the original project file.

ATTACHMENT E

analytics environmental laboratory inc.

195 Commerce Way
Portsmouth, NH 03801
Phone (603) 436-5111
Fax (603) 430-2151

For Analytics Use Only Rev 5/99

Samples were:

1) Shipped or hand-delivered
2) Temp blank °C _____
3) Received in good condition Y or N
4) pH checked by: _____
5) Labels checked by: _____

Project#: Proj. Name:

Company:

Contact:

Address:

Phone: PO# Quote #

Sampler (Signature):

Matrix Key:
W=Water
S=Soil/Sludge
O=Oil
E=Extract
X=Other

Container Key
P=plastic G=glass

Station Identification	Sample Date	Sample Time	Analysis	Preservation: Unpres	4°C	HNO_3	H_2SO_4	HCL	Other	Matrix	Container number/type		pH	Analytics Sample #

FAX RESULTS? YES NO

Fax # _____

Turnaround Request

Standard ☐ Due Date _____

Priority ☐ Due Date _____

Comments / Instructions:

Page____ of____

Received By: Time: Date: Relinquished By Sampler:

Received By: Time: Date: Relinquished By:

Received By: Time: Date: Relinquished By:

ATTACHMENT F

Analytics Standard Operating Procedures

* NELAC SOP BOLDED=Calibration SOP

SEMI-VOLATILE SOPS

*S001 SEPARTORY FUNNEL LIQUID/LIQUID EXTRACTION FOR THE DETERMINATION OF SEMI-VOLATILES IN AQUEOUS SAMPLES
S002 OBSOLETE
S003 WASTE DILUTION METHOD FOR THE DETERMINATION OF SEMI-VOLATILES IN NON-AQUEOUS LIQUID SAMPLES
*S004 LABORATORY METHOD FOR THE PREPARATION OF SEMI-VOLATILE STANDARDS
***S005 LABORATORY METHOD FOR THE DETERMINATION OF ACID AND BASE/NEUTRALS IN ALL MATRICES**
S006 OBSOLETE
S007 SONICATION EXTRACTION FOR THE DETERMINATION OF SEMI-VOLATILES IN SOLID SAMPLES
S008 MICROEXTRACTION OF SOLID SAMPLE FOR THE DETERMINATION OF POLYNUCLEAR AROMATIC HYDROCARBONS (NOT IN USE)

VOLATILE SOPS

*V001 LABORATORY METHOD FOR THE PREPARATION OF GC/MS VOLATILE ORGANIC STANDARDS
V002 PREPARATION OF SOLID SAMPLES (MEDIUM LEVEL) FOR VOLATILE ORGANIC ANALYSIS
V003 PREPARATION OF SOLID SAMPLES (LOW LEVEL) FOR VOLATILE ORGANIC ANALYSIS
V004 OBSOLETE
***V005 LABORATORY METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS - CAPILLARY COLUMN, GC/MS DETECTION**
***V006 LABORATORY METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN DRINKING WATER - CAPILLARY COLUMN, GC/MS**
V007 OBSOLETE
V008 OBSOLETE
*V009 LABORATORY METHOD FOR SAMPLE PREPARATION AND EXTRACTION OF VOLATILE ORGANIC COMPOUNDS BY PURGE-AND-TRAP
V010 OBSOLETE
VO11 LABORATORY METHOD FOR THE DETERMINATION OF METHANE,ETHANE,AND ETHENE IN AQUEOUS SAMPLE- HEADSPACE PREPARATION WITH GC/FID DETECTION

PESTICIDE/PCB SOPS

*P001 LABORATORY METHOD FOR THE PREPARATION OF ORGANOCHLORINE PESTICIDESAND POLYCHLORINATED BIPHENYLS (PCB) STANDARDS
*P002 SEPARATORY FUNNEL LIQUID/LIQUID EXTRACTION FOR THE DETERMINATION OF PESTICIDES AND PCBS IN AQUEOUS SAMPLES
P003 OBSOLETE
P004 WASTE DILUTION METHOD FOR THE DETERMINATION OF PESTICIDES AND PCBS IN NON AQUEOUS LIQUID SAMPLES
P005 MICROEXTRACTION OF SOLID SAMPLES FOR THE DETERMINATION OF PESTICIDES AND PCBS (NOT IN USE)
P006 SONICATION EXTRACTION FOR THE DETERMINATION OF PESTICIDES AND PCBS IN SOLID SAMPLES
P007 FLORISIL COLUMN CLEANUP FOR PESTICIDES AND PCBS
P008 SULFUR CLEANUP FOR PESTICIDE AND PCBS
*P009 ACID CLEANUP FOR PCBS
***P010 LABORATORY METHOD FOR THE DETERMINATION OF PESTICDES IN ALL MATRICES**
P011 PCB WIPE SAMPLE PREPARATION

***P012 LABORATORY METHOD FOR THE DETERMINATION OF PCBS IN ALL MATRICES**

Analytics Standard Operating Procedures

* NELAC SOP BOLDED=Calibration SOP

***P013 LABORATORY METHOD FOR DETERMINATION OF EDB AND DBCP BY GAS CHROMATOGRAPHY AND EPA METHOD 504.1**

HERBICIDE SOPS

H001 LABORATORY METHOD FOR THE PREPARATION OF HERBICIDE STANDARDS
H002 SEPARATORY FUNNEL LIQUID/LIQUID EXTRACTION FOR THE DETERMINATION OF CHLORINATED ACID HERBICIDES IN AQUEOUS SAMPLES
H003 SONICATION EXTRACTION FOR THE DETERMINATION OF CHLORINATED ACID HERBICIDES IN SOLID SAMPLES
H004 EXTRACTION FOR THE DETERMINATION OF CHLORINATED ACID HERBICIDES IN NON AQUEOUS LIQUID SAMPLES
H005 LABORATORY METHOD FOR THE DETERMINATION OF HERBICIDES IN ALL MATRICES

TPH SOPS

T001 LABORATORY METHOD FOR THE PREPARATION OF TOTAL PETROLEUM HYDROCARBON AND DIESEL RANGE ORGANIC STANDARDS
T002 LABORATORY METHOD FOR THE PREPARATION OF TOTAL GASOLINE AND GASOLINE RANGE ORGANIC STANDARDS
T003 SEPARATORY FUNNEL LIQUID/LIQUID EXTRACTION FOR THE DETERMINATION OF TOTAL PETROLEUM HYDROCARBONS IN AQUEOUS SAMPLES
T004 OBSOLETE
T005 WASTE DILUTION FOR THE DETERMINATION OF TOTAL PETROLEUM HYDROCARBONS AND DIESEL RANGE ORGANICS IN NON AQUEOUS LIQUID SAMPLES
T006 MICROEXTRACTION OF SOLID SAMPLES FOR THE DETERMINATION OF TOTAL PETROLEUM HYDROCARBONS (NOT IN USE)
T007 SONICATION EXTRACTION FOR THE DETERMINATION OF TOTAL PETROLEUM HYDROCARBONS IN SOLID SAMPLES
T008 LABORATORY METHOD FOR THE DETERMINATION OF TOTAL PETROLEUM HYDRO CARBONS IN ALL MATRICES
T009 LABORATORY METHOD FOR THE DETERMINATION OF TOTAL GASOLINE IN ALL MATRICES
T010
T011
T012 SEPARATORY FUNNEL LIQUID/LIQUID EXTRACTION FOR THE DETERMINATION OF DIESEL RANGE ORGANICS IN AQUEOUS SAMPLES
T013 SONICATION EXTRACTION FOR THE DETERMINATION OF DIESEL RANGE ORGANICS IN SOLID SAMPLES
T014 LABORATORY METHOD FOR THE DETERMINATION OF DIESEL RANGE ORGANICS IN ALL MATRICES
T015 LABORATORY METHOD FOR THE DETERMINATION OF GASOLINE RANGE ORGANICS IN ALL MATRICES

MISCELLANEOUS SOPS

*M001 LABORATORY METHOD FOR THE CLEANING OF GLASSWARE USED FOR SAMPLE AND STANDARD PREPARATION
M002 LABORATORY METHOD FOR THE DETERMINATION OF PERCENT SOLIDS
M003 LABORATORY METHOD FOR THE DETERMINATION OF FREE LIQUIDS BY THE PAINT FILTER LIQUIDS TEST
M004 OBSOLETE
M005 LABORATORY METHOD FOR THE DETERMINATION OF FLASHPOINT USING THE PENSKY-MARTIN CLOSED CUP TESTER AND THE IGNITABILITY CHARACTERISTIC USING A DIRECT FLAME
***M006 LABORATORY METHOD FOR THE DETERMINATION OF PH IN SOLID AND LIQUID SAMPLES**
*M007 MEASURING HOOD FLOW

Analytics Standard Operating Procedures

* NELAC SOP BOLDED=Calibration SOP

M008
*M009 LABORATORY METHOD FOR BALANCE CALIBRATION
M010 LABORATORY METHOD FOR THE DETERMINATION OF REACTIVE SULFIDE AND CYANIDE
*M011 CLEANING AND SILANIZING OF INJECTION PORT LINERS FOR GAS CHROMATOGRAPHS
*M012 RECEIPT OF SUPPLIES, CHEMICALS, AND STANDARDS
*M013 DATA BACKUP - GC-GC/MS
M014 LABORATORY METHOD FOR REPIPETTER CALIBRATION
*M015 LABORATORY METHOD FOR THE TESTING OF SURROGATE AND SPIKE SOLUTIONS
M016 LABORATORY METHOD FOR THE COMPOSITING OF LIQUID AND SOLID SAMPLES
M017
*M018 THERMOMETER CALIBRATION CHECK
*M020 LABORATORY METHOD FOR PIPETTING
*M021 LABORATORY METHOD FOR DETERMINATION OF METHOD DETECTION LIMITS(MDLS)

TCLP SOPS

TC001 LABORATORY METHOD FOR THE TCLP AND SPLP EXTRACTION OF VOLATILE AND NONVOLATILE COMPOUNDS IN LIQUID AND SOLID WASTES

WASTE MANAGEMENT

*W001 WASTE DISPOSAL

SAMPLE MANANGEMENT

*L001 SAMPLE RECEIPT, PRESERVATION, AND STORAGE
*L002 BOTTLE ORDERS
*L003 PREPARATION OF TRIP BLANKS, HOLDING BLANKS AND VOA VIALS.
*L004 PROCESSING OF NEW CLIENTS
*L005 SAMPLE INFORMATION ENTRY INTO THE DATA BASE
*L006 CLIENT BOTTLE ORDERS

DATA VALIDATION

*D001 VALIDATION OF ANALYTICAL DATA
*DOO2 DATA CONTROL AND ARCHIVING

REPORTING

*R001 INVOICING

ATTACHMENT G

Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix:
(examples: laboratory pure water, soil, air, solid, biological tissue)
Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts 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.

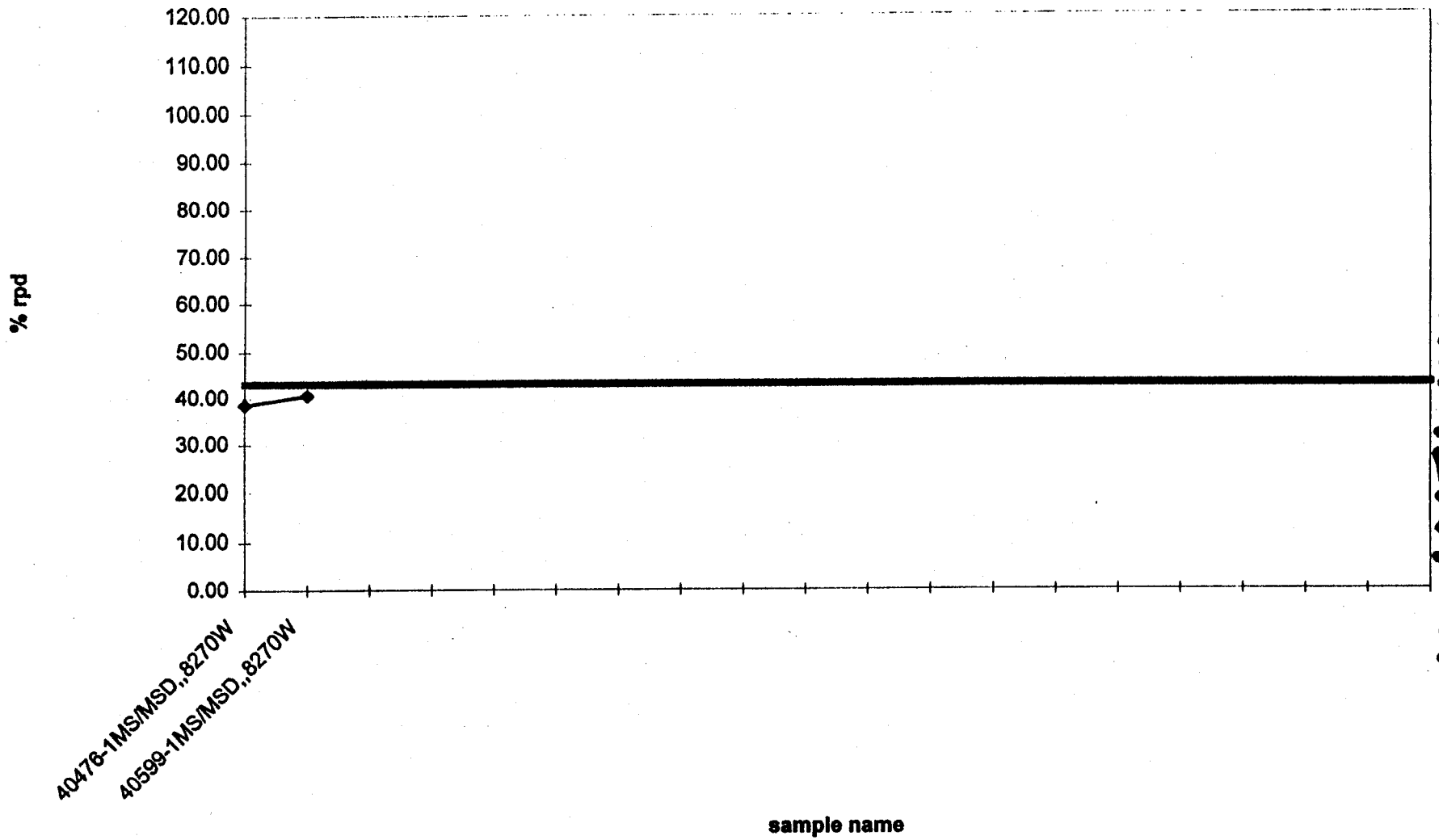
4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Technical Director's Name and Title	Signature	Date
Quality Assurance Officer's Name	Signature	Date

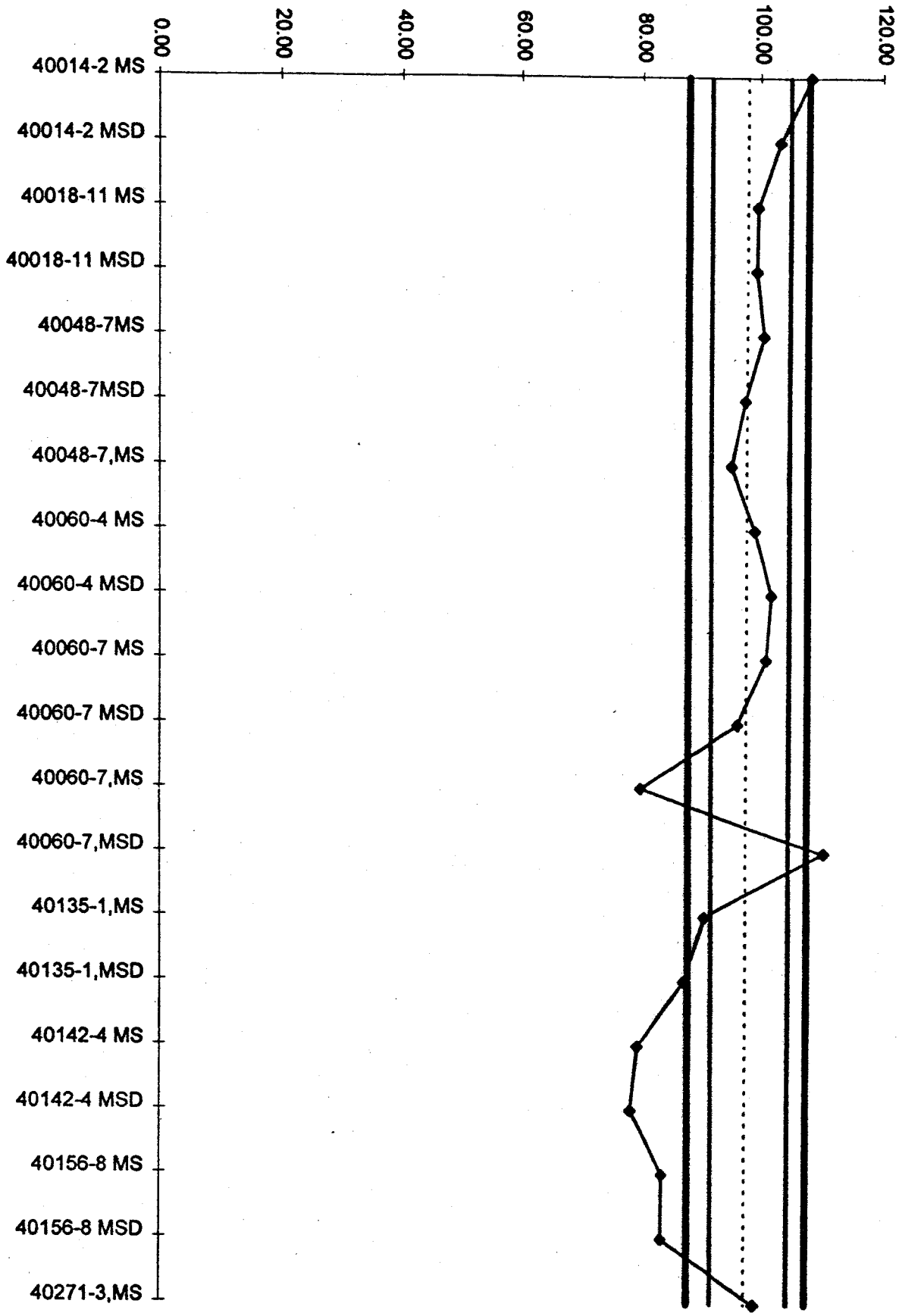
PHENOL

Waters



ATTACHMENT H

ATTACHMENT I



TRICHLOROETHENE

ATTACHMENT J

analytics environmental laboratory inc.
195 Commerce Way
Portsmouth, New Hampshire 03801
603-436-5111 Fax 603-430-2151
800-929-9906

Mr. Herb Kodis
Maine Environmental Laboratory, Inc.
PO Box 1107
Yarmouth, ME 04096-1107

April 29, 1998

SAMPLE DATA

Lab Sample ID: 111
Matrix: Aqueous
Percent Solid: N/A
Dilution Factor: 1.0
Collection Date: 03/17/98
Lab Receipt Date: 03/19/98
Extraction Date: 03/23/98
Analysis Date: 03/26/98

CLIENT SAMPLE ID

Project Name: Sample Report

Project Number:

Field Sample ID: Sample Report

PESTICIDE AND PCB ANALYTICAL RESULTS

COMPOUND	Detection Limit µg/L	Results µg/L	COMPOUND	Detection Limit µg/L	Results µg/L
Aldrin	0.5	ND	Endrin Ketone	0.5	ND
a-BHC	0.5	ND	Heptachlor	0.5	ND
b-BHC	0.5	ND	Heptachlor Epoxide	0.5	ND
d-BHC	0.5	ND	Methoxychlor	2	ND
g-BHC	0.5	ND	Chlordane	0.5	ND
4,4'-DDD	0.5	ND	Toxaphene	2	ND
4,4'-DDE	0.5	ND	PCB-1016/1242	1	ND
4,4'-DDT	0.5	ND	PCB-1221	1	ND
Dieldrin	0.5	ND	PCB-1232	1	ND
Endosulfan I	0.5	ND	PCB-1248	1	ND
Endosulfan II	0.5	ND	PCB-1254	1	ND
Endosulfan Sulfate	0.5	ND	PCB-1260	1	ND
Endrin	0.2	ND			
Endrin Aldehyde	0.5	ND			

Surrogate Standard Recovery

2,4,5,6-Tetrachloro-m-xylene %

Decachlorobiphenyl %

ND=None Detected J=Estimated E=Exceeds Calibration Range B=Detected in Blank

METHODOLOGY: Sample was analyzed according to "40 CFR Part 136 EPA Method 608."

COMMENTS:

Authorized signature _____