# Quality Assurance Project Plan (QAPP) for Eighteenmile Creek PCB Source Trackdown Project Niagara County, New York

**December 2005**

**Prepared for:**

**NIAGARA COUNTY SOIL AND WATER CONSERVATION DISTRICT**
4487 Lake Avenue
Lockport, NY 14094

Niagara County Project Manager — Date: 12/15/05

E & E Project Manager — Date: 12/14/05

E & E QA Director — Date: 12/14/05

ecology and environment engineering, p.c.

BUFFALO CORPORATE CENTER 368 Pleasant View Drive, Lancaster, New York 14086
Tel: 716/684-8060, Fax: 716/684-0844

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# List of Abbreviations and Acronyms

| | |
|---|---|
| ADR | automated data review |
| AOC | Area of Concern |
| ASP | Analytical Services Protocol |
| CLP | Contract Laboratory Procedure |
| COC | chain-of-custody |
| CPR | cardiopulmonary resuscitation |
| DGPS | Differential Global Positioning Systems |
| DOT | United States Department of Transportation |
| DUSR | Data Usability Summary Report |
| EDD | electronic data deliverable |
| EDMS | Electronic Data Management System |
| E & E | Ecology and Environment, Inc. |
| ELAP | Environmental Laboratory Accreditation Program |
| EPA | United States Environmental Protection Agency |
| GIS | geographic information system |
| LCS | laboratory control sample |
| MDL | method detection limit |
| mg/kg | milligram per kilogram |
| MS/MSD | matrix spike/matrix spike duplicate |
| MSB | matrix spike blank |
| NCSWCD | Niagara County Soil and Water Conservation District |

## List of Abbreviations and Acronyms (cont.)

| | |
|---|---|
| NELAP | National Environmental Laboratory Accreditation Program |
| NYSDEC | New York State Department of Environmental Conservation |
| NYSDOH | New York State Department of Health |
| PARCC | precision, accuracy, representativeness, completeness, and comparability |
| PCB | polychlorinated biphenyl |
| PE | performance evaluation |
| PPE | personal protection equipment |
| QA/QC | quality assurance/quality control |
| QAM | Quality Assurance Manual |
| QAPP | Quality Assurance Project Plan |
| QMP | Quality Management Plan |
| RAP | Remedial Action Plan |
| RPD | relative percent difference |
| SDG | sample delivery group |
| SOP | Standard Operating Procedure |
| SPDES | State Pollutant Elimination System |
| STL | Severn Trent Laboratories |
| TOC | total organic carbon |

## Distribution List

| Party | Affiliation and Title | Revision | Date Sent |
|---|---|---|---|
| **Eighteenmile Creek QAPP Original Distribution** | | | |
| Marcia Meredith Galloway | E & E QA Director | 0 | |
| Kris Erickson | E & E Project Manager | 0 | |
| Victor DiGiacomo | NCSWCD Project Manager | 0 | |
| Tony Bogolin | STL-Buffalo | 0 | |
| Field Team Leader | Gene Florentino | 0 | |

## Revision List

| Revision | Modifications | Distribution |
|---|---|---|
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# 1 Project Management

This Quality Assurance Project Plan (QAPP) has been prepared by Ecology and Environment, Inc. (E & E) for the Niagara County Soil and Water Conservation District (NCSWCD) in support of the Eighteenmile Creek Polychlorinated Biphenyl (PCB) Trackdown Study located in Niagara County, New York.

This QAPP has been prepared in accordance with "United States Environmental Protection Agency (EPA) Requirements for Quality Assurance Project Plans," final, EPA QA/R-5 (March 2001) and EPA Region 2 Guidance for the Development of QAPP for Environmental Monitoring Projects (April 2004); and also incorporates New York State Department of Environmental Conservation (NYSDEC) requirements. This QAPP presents the policies, organization, objectives, functional activities, and specific quality assurance/quality control (QA/QC) procedures that will be employed by E & E to ensure that all technical data generated for the Eighteenmile Creek PCB Trackdown Study are accurate, representative, and ultimately capable of withstanding judicial scrutiny. These activities will be implemented under the requirements of E & E's comprehensive QA program as documented in the corporate Quality Management Plan (QMP).

The QAPP is formatted to address the four major sections listed in the EPA QAPP guidance document: Project Management, Data Generation and Acquisition, Assessment and Oversight, and Data Validation and Usability.

## 1.1 Project Organization

The organizational chart for the project work is presented on Figure 1-1. The QA Director independently reports to the NCSWCD Project Manager on all QA/QC issues. The specific names and contact information for the current project team are provided in Table 1-1. The roles and specific QA responsibilities of key project personnel are described below.

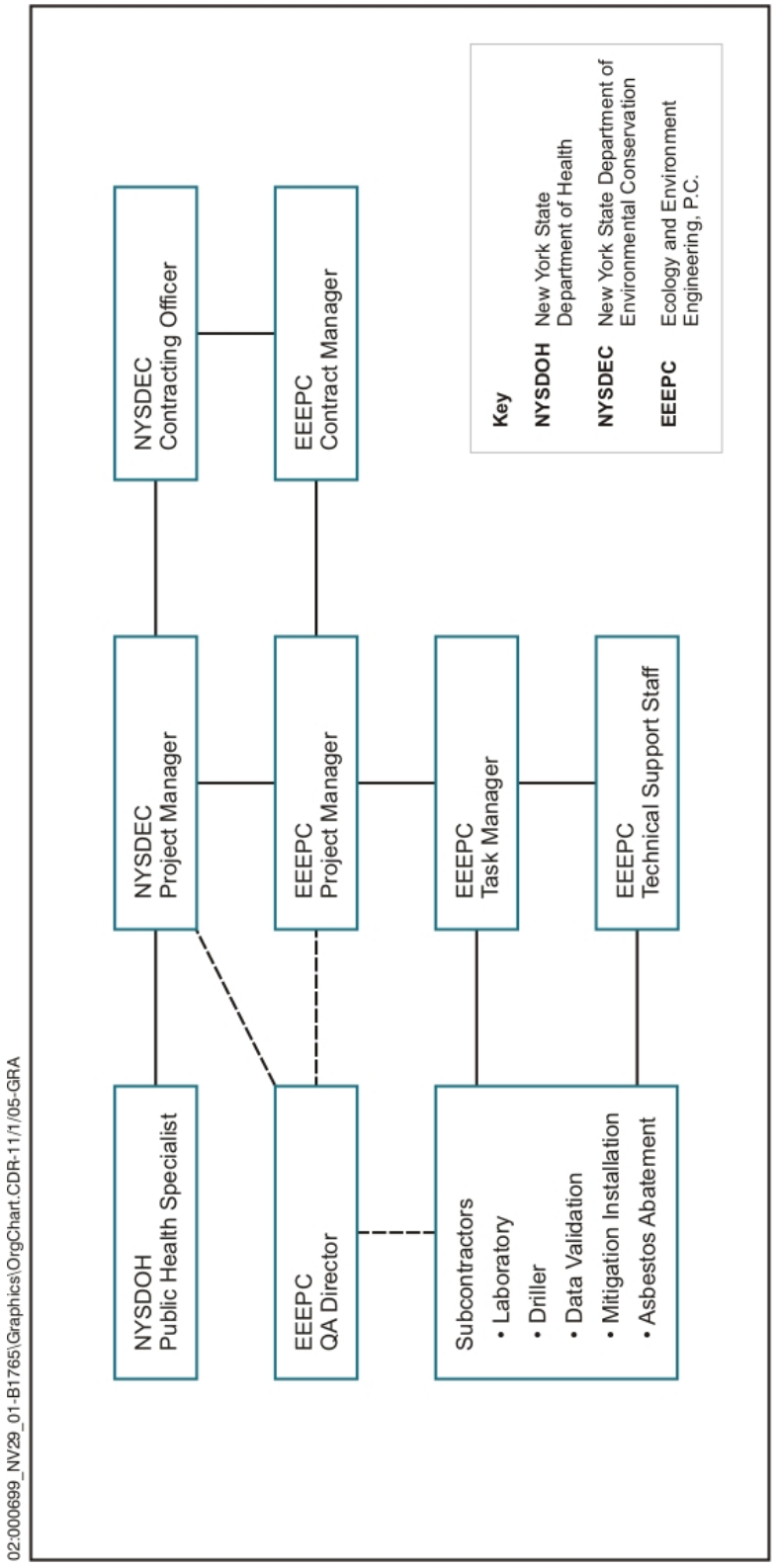


**Figure 1-1 Eighteenmile Creek PCB Trackdown Study Project Organization Chart**

**Table 1-1 Project Organization, Eighteenmile Creek PCB Trackdown Study**

| Key Team Member | Contact Information | |
|---|---|---|
| NCSWCD Project Manager | Victor F. DiGiacomo | 716-434-4949 |
| EPA Region 2 Project Officer | Marie O'Shea | |
| E & E QA Director/Program QA Officer | Marcia Meredith Galloway | 716-684-8060 |
| E & E Project Manager | Kris Erickson | 716-684-8060 |
| E & E Task Manager | Gene Florentino | 716-684-8060 |
| E & E Project Chemist | Rebecca Humphrey | 716-684-8060 |
| **Subcontractors** | | |
| Laboratory (sediment) | Tony Bogolin<br>Project Manager<br>Severn Trent Laboratories, Inc.<br>10 Hazelwood Drive<br>Amherst, NY 14228 | 716-504-9822 |

### Project Manager

The Project Manager is responsible for QA/QC functions for all task-specific operations on the Eighteenmile Creek PCB Trackdown Study project, and the overall quality of E & E's performance on the NCSWCD contract.

The Project Manager will also be responsible for the overall quality of work performed under project activities as it relates to the following specific roles:

- Overseeing day-to-day performance including all technical and administrative operations;
- Interfacing frequently with the NCSWCD Project Manager;
- Tracking schedules and budgets and management of mobilization and contract closeout activities;
- Selecting and monitoring technical support staff; and
- Reviewing and approving all final reports and other work products.

### QA Director/Project QA Officer

The QA Officer is responsible for oversight of all QA/QC activities for NCSWCD projects. The QA Officer will remain independent of day-to-day, direct project involvement but will have the responsibility for ensuring that all project and task-specific QA/QC requirements are met. The QA Officer will have direct access to corporate executive staff, as necessary, to resolve any QA/QC problems, disputes, or deficiencies. The QA Officer's specific duties include:

- Reviewing and approving the QAPP;
- Conducting field and laboratory audits in conjunction and keeping written records of the audits;
- Coordinating with the NCSWCD Project Manager, field team, and laboratory management to ensure that QA objectives appropriate to the project are set and that laboratory and field personnel are aware of these objectives; and
- Recommending, implementing, and/or reviewing actions taken in the event of QA/QC failures in the laboratory or field.

### Project Chemist

The Project Chemist is responsible for data validation and verification, generation of Data Usability Summary Reports (DUSRs), and independent assessment of the hard copy and electronic analytical data. The Project Chemist will report nonconformance with QC criteria (including an assessment of the impact on data quality objectives) to the appropriate managers.

### Technical Support Staff

The technical support staff for this program will be drawn from E & E's pool of corporate resources. The technical support staff will implement project and site tasks, analyze data, and prepare reports/support materials. All support personnel assigned will be experienced professionals who possess the degree of specialization and technical competence necessary to perform the required work effectively and efficiently.

### Laboratories

Laboratory analyses of all environmental matrices will be performed by Severn Trent Laboratories (STL) Buffalo. STL-Buffalo is certified by both the New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP) for environmental analysis of water, solid and hazardous wastes, and air and National Environmental Laboratory Accreditation Program (NELAP).

The laboratory Project Manager QA duties include:

- Reviewing the QAPP to verify that analytical operations will meet project requirements;
- Reviewing receipt of all sample shipments and notifying the E & E Project Manager and Project Chemist of any discrepancies within one day of receipt;

- Rapidly notifying the E & E Project Manager and Project Chemist regarding laboratory nonconformance with the QAPP or analytical QA/QC problems affecting project samples; and

- Coordinating with the E & E Project Manager and Project Chemist, and laboratory management to implement corrective actions approved by NCSWCD.

## 1.2 Problem Definition/Background

### 1.2.1 Problem Definition

The purpose of this trackdown project is to further evaluate contamination in Eighteenmile Creek sediments. The specific objectives of this investigation are to:

- Review all historical sampling data available to identify potential PCB sources and future sampling locations of interest, utilizing Geographic Information System (GIS) technology to accurately depict the data spatially;

- Further evaluate the nature and extent (horizontally and vertically) of PCB, arsenic, copper, chromium, lead, zinc and mercury contamination in Eighteenmile Creek sediment in an attempt to determine the source or sources of these contaminants; and

- Assist and make progress towards the de-listing of Eighteenmile Creek as an Area of Concern (AOC).

PCBs contaminate the sediments of Eighteenmile Creek and its AOC. PCBs are factors in restrictions on fish and wildlife consumption, bird and animal deformities, or reproductive problems and degradation of benthos. A surface sediment sample taken in the 1994 Olcott Harbor Sediment Sampling from the AOC contained PCBs at a concentration greater than the NYSDEC guidance for screening of contaminated sediments. Ten of 15 fish flesh samples from the creek contained PCBs at levels above the Food and Drug Administration action level of 2.0 milligrams per kilogram (mg/kg).

Sources and potential sources of PCBs to Eighteenmile Creek have been identified as industrial and municipal wastewater discharges, combined sewer overflows, inactive hazardous waste sites, the New York Barge Canal discharge, contaminated sediments already present in the creek and an unknown source between Olcott Street and North Transit Road. Extensive progress has been made by monitoring discharges and updating State Pollutant Elimination System (SPDES) permits for industrial and municipal wastewater dischargers and de-listing inactive hazardous waste sites. NYSDEC conducted a sediment study in the area of the unknown source of PCBs located between Olcott Street and North Transit Road in April of 2005. It is anticipated that the results from this study will sup-

plement the work completed by the NYSDEC and further lead to defining this unknown source of contamination.

### 1.2.2 Background

Eighteenmile Creek, located in the heart of Niagara County is surrounded by six highly residential townships. Many citizens own creek-front property from the start of its headwaters in the town of Lockport to its discharge to Lake Ontario in Olcott, New York. The creek is used extensively for fishing, boating, and recreation. The projected sampling location is primarily in a residential neighborhood. It is suspected that sediment contamination in this area has impacted residential properties adjacent to the creek.

Currently supported by numerous stakeholders, the NCSWCD, United States Army Corps of Engineers, and Niagara County are in the preliminary stages of developing a Comprehensive Watershed Management Plan for Eighteenmile Creek. It is apparent that the creeks recovery and management will be undermined by the continuing influence of unknown contaminant sources and the creek's contaminated sediments.

Clarifying the source or sources of continuing PCB contamination in Eighteenmile Creek will greatly enhance EPA and NYSDEC's ability to eliminate these contaminants and their impacts on the residential properties adjacent to the creek, the Eighteenmile Creek AOC and Lake Ontario. Reducing contamination in the Eighteenmile Creek watershed will directly:

- Support regional Eighteenmile Creek watershed and AOC restoration efforts;
- Reduce the risk to human health and the environment; and
- Restore Eighteenmile Creek's beneficial uses by improving sediment quality, restoring the health of benthic communities, fish and wildlife, and improving the potential for human consumption of fish.

The project will provide outputs that include a GIS map of known PCB data and identified potential sources, 80 PCB screening samples at a detection limit of 0.25 mg/kg and 36 sediment core samples to a depth of 3 feet. The project will also produce a report that will publish the results of the research, conclusions, and final recommendations. Intermediate outcomes accomplished through the completion of this project would include the identification of unknown sources of PCBs contaminating Eighteenmile Creek sediments and identifying the extent of PCB contamination in these sediments.

This would contribute to an end outcome of forward progress in implementing the Remedial Action Plan (RAP) for the Eighteenmile Creek AOC, by completing a

preliminary step necessary to make improvements in overall water quality, habitat and the health of fish and wildlife. The RAP's mission is to restore the chemical, physical and biological integrity of the AOC ecosystem. Locating upstream sources of contamination to the AOC will aid in this mission and make progress towards the overall goal of de-listing Eighteenmile Creek as an AOC.

## 1.3 Project Description

The project has two main tasks:

### 1.3.1 Review of Historical Data

A detailed GIS map and database of the source area will be developed. All sources of existing data will be collected and subject to quality overview. The data will be entered into the GIS database and where possible sampling locations will be geo-referenced. Meta data files will be created for each data source to indicate pertinent quality information. The United States Army Corps of Engineers - Buffalo District will help to develop this effort.

Potential PCB sources in the area will be investigated and identified on the GIS map. A final map depicting known PCB data and identified potential sources will be created. The map will be used to identify, and later map, key sampling locations by E & E and NCSWCD. The historical data will be combined with the data collected on the PCB source trackdown to provide final contour maps of historical contamination and project potential sources. All data will be used by NCSWCD and United States Army Corps of Engineers to implement the RAP.

### 1.3.2 Source Trackdown Sampling in Eighteenmile Creek

Sediment core sampling in Eighteenmile Creek will be conducted from the Clinton Street Dam to a location downstream that is deemed sufficient at the completion of a preliminary screening round. Sampling will be conducted in the general area of the former Flintokote Plant Site. Based on the review of historical data, some additional samples may be collected farther downstream of the Clinton Street Dam area. A map of the project area is presented in Figure 1-2.

The sampling effort will quantify upstream-downstream differences in total concentrations at an 18-inch depth using a small coring device. Collection will be conducted around potential sources and at defined intervals along the route. Samples will be screened for PCBs using a modified laboratory screening procedure. A total of 80 samples will be collected for PCB screening at a detection limit of 0.25 mg/kg. Some of these samples may be collected in the Eighteenmile Creek bed downstream of the sampling site if additional source data are needed and suggested at the completion of the NYSDEC Eighteenmile Creek sediment investigation, conducted in April 2005.

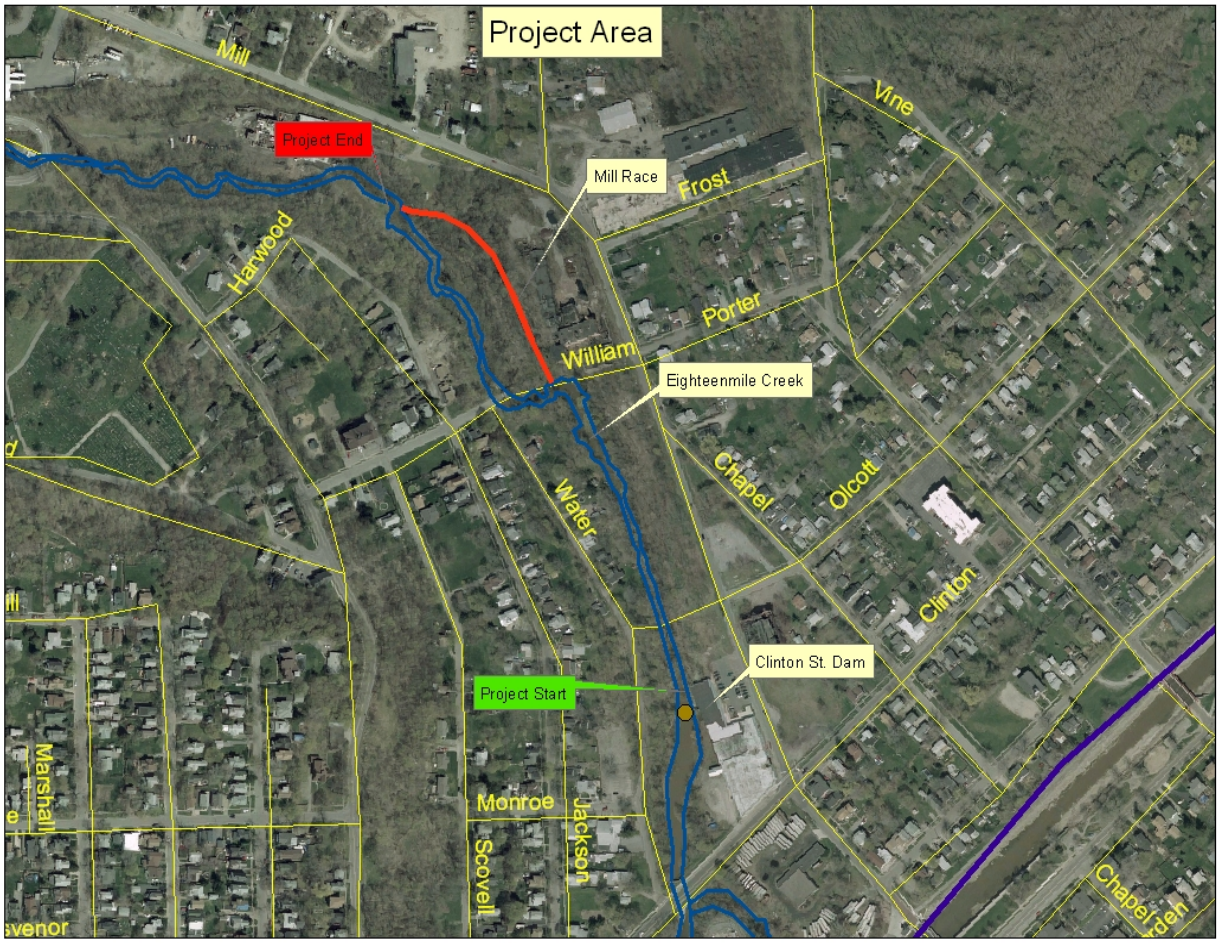


**Figure 1-2 Project Location**

PCB screening sample results will be analyzed within 72 hours. The screening results will be used to determine locations for sediment core samples. A total of 12 sediment core samples (3-foot) are planned approximately every 500 feet. Sediment cores will be analyzed for low level PCBs, select metals and total organic carbon (TOC). PCBs will be analyzed at 1 foot intervals and metals and TOC will be analyzed as a composite of all 3 feet. Metals will include the following: arsenic, chromium, copper, lead, zinc, and mercury. A summary of the sample collection is provided on Table 1-2.

The project schedule will depend on the QAPP approval date. Weather is a crucial factor in determining when the sampling may be completed. It is anticipated that sampling will commence in early spring and the project end date is September 2006.

**Table 1-2 Sampling and Chemical Analysis Summary Table, Eighteenmile Creek PCB Source Trackdown, Lockport, NY**

| Analysis | Method No. | No. of Field Samples | Field Duplicates | Trip Blanks | Rinsate Blanks | MS/MSD | Total No. of Samples |
|---|---|---|---|---|---|---|---|
| **Sediment Sampling** | | | | | | | |
| TCL PCBs-Screening | SW 8082 Mod | 80 | 4 | – | – | 4 | 84 |
| TCL PCBs | SW 8082 | 36 | 0 | – | – | 0 | 36 |
| TAL As, Cu, Cr, Pb, Zn, and Hg only | SW 6010B/ 7471A | 12 | 0 | – | – | 0 | 12 |
| Total Organic Carbon (TOC) | Lloyd-Kahn | 12 | 0 | – | – | 0 | 12 |
| Percent Solids | ASTM D2216 | 116 | 4 | – | – | 4 | 120 |

Project milestones are scheduled to include:

- Project Start 11/1/2005
- QAPP Submittal 12/15/2005
- Historical Data Analysis and GIS Mapping 2/2006
- PCB Screening Round 4/2006
- Sediment Core Sampling 5/2006
- Preparation of Semi-annual Report 4/2006
- Data Review and Analysis 6/2006
- Draft Report Preparation 7/2006
- Review and Approval of Final Report 8/2006
- Project End 9/2006

## 1.4 Quality Objectives and Criteria

General quality objectives for the Eighteenmile Creek PCB Trackdown Study are summarized in Table 1-3. Acceptance and performance criteria for field and analytical QC samples are outlined in Section 2.4. Appendix A of this QAPP provides detailed acceptance and performance criteria for analytical methods.

**Table 1-3 General Data Quality Objectives, Eighteenmile Creek PCB Trackdown Study Projects**

| Data Collection Activity | Quality Objectives | Standards[a] | Acceptability/ Performance Criteria[b] |
|---|---|---|---|
| Historical Data Collection | To incorporate all existing data that meets quality objectives for the RAP. Data must be geo-referenced. | ■ EPA or NYSDEC sampling and analytical procedures | ■ Data are generated using EPA or NYSDEC sampling and analytical methods or alternative methods approved under a RAP project.<br>■ Data must be from the original source.<br>■ Data must be geo-referenced or able to be digitized into a GIS system. |
| Sampling and Analysis | To have samples and analytical results that accurately represents the nature and extent of contamination at the site. Data must be of sufficient quality to meet all regulatory requirements and allow assessment of impacts by comparison to New York State criteria or background values. Data must present Aroclor results to allow comparison of PCBs in sediment to potential source areas. | ■ NYSDEC Division of Fish, Wildlife and Marine Resources, Technical Guidance for Screening Contaminated Sediments, January 25, 1999<br>■ NYSDEC TAGM 4046 | ■ Data must be collected under an approved QAPP.<br>■ Data must meet the acceptance and performance criteria documented in Section 2 of this QAPP.<br>■ Reporting limits should be below risk-based screening values for 90% of target analytes and 100% of critical analytes of concern.<br>■ Data must be compared to standards.<br>■ Data must be compared to potential source areas to evaluate for type of PCBs present in hot spots. |
| Mapping | To relate project work locations (including sample, monitoring well, and test pit locations) to existing local benchmarks. | ■ GPS data | ■ Relation of all survey points to existing/known benchmarks.<br>■ Accurate horizontal coordinates (±3 feet for GPS locations). |
| Field Records | To document all field activities and to allow accurate representation field events in the final report. Records must be capable of withstanding legal scrutiny. | ■ Section 2 of the QAPP<br>■ E & E SOPs (Field Activities Logbooks) | ■ Consistency between field and laboratory data.<br>■ Clear and legible documentation for sample collection and equipment decontamination for final report. |
| Outside Records | To use the most current reference values, reports, or data from outside sources in data assessments and recommendations for the site. | None | ■ All versions of data or standards must be the most current values available.<br>■ Data or standards must be accurately incorporated into the final report. |

## Table 1-3 General Data Quality Objectives, Eighteenmile Creek PCB Trackdown Study Projects

| Data Collection Activity | Quality Objectives | Standards[a] | Acceptability/ Performance Criteria[b] |
|---|---|---|---|

Notes:

[a] Major standards.
[b] Major or noteworthy acceptability criteria. All performance criteria must be verified using procedures listed in the QAPP.

Key:

EPA = Environmental Protection Agency.
GPS = Global Positioning System.
NYSDEC = New York State Department of Environmental Conservation.
SOP = Standard Operating Procedure.
TAGM = Technical and Administrative Guidance Memorandum 4046 Soil Cleanup Objectives.
QAPP = Quality Assurance Project Plan.

### 1.4.1 Data Assessment Definitions

Acceptance and performance criteria are often specified in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters. Numerical acceptance criteria cannot be assigned to all PARCC parameters, but general performance goals are established for most data collection activities. Numerical goals for analytical methods are presented in Section 2.4. Data assessment procedures throughout the QAPP clearly outline the steps to be taken, responsible individuals, and implications if QA objectives are not met. PARCC parameters are briefly defined below.

#### Precision

Precision measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared to their average value, usually stated in terms of standard deviation or coefficient of variation. It also may be measured as the relative percent difference (RPD) between two values. Precision includes the interrelated concepts of instrument or method detection limits and multiple field sample variance. Sources of this variance are sample heterogeneity, sampling error, and analytical error.

Field duplicates will be used to assess precision for this site as described in Section 2.5. Because the data will used to determine potential sources of PCBs, precision is a critical data quality indicator for the screening results. For PCB core data precision will be assumed to be as good as the screening samples.

#### Accuracy

Accuracy measures the bias of the measurement system. Sources of this error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis. Data interpretation and reporting may also be significant sources of error. Typically, analytical accuracy is assessed through the analysis of spiked samples and may be stated in terms of percent recovery or the average (arithmetic mean) of the percent recovery. Blank samples are also analyzed to assess sampling and analytical bias (i.e., sample contamination). Background measurements similarly assess measurement bias. Background samples may be collected for the screening analysis.

#### Representativeness

Representativeness expresses the degree to which data represent a characteristic of a population, a parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative parameter, which is most concerned with proper design of the measurement program. Sample/measurement locations may be biased (judgmental) or unbiased (random or systematic).

Representativeness of the sampling scheme will be determined with evaluation of the historical data.

### Completeness

Completeness is defined as the percentage of measurements performed which are judged to be valid. Although a quantitative goal must be specified, the completeness goal is the same for all data uses—that a sufficient amount of *valid* data be generated. A completeness goal of 90% is established for this project.

### Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set may be compared to another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through the use of standard techniques to collect and analyze samples. Historical data will be evaluated to ensure the methods and reporting limits are comparable the to the proposed sampling. Data will only be evaluated if it is determined to be comparable.

## 1.5 Special Training/Certification

E & E training requirements for the Eighteenmile Creek PCB Trackdown Study activities are as follows:

- E & E employees that participate in on-site activities must have completed the 40-hour health and safety training program and the cardiopulmonary resuscitation (CPR)/first aid certification course. To continue such participation, each employee must successfully complete a minimum of eight hours of refresher training, annually; and

- All personnel shipping samples must complete the United States Department of Transportation (DOT) hazardous materials transportation training and certification, including training in specific International Air Transport Association (IATA) regulations (air shipments).

## 1.6 Documentation and Records

The E & E Program QA Officer will approve the QAPP and maintain the most current approved version of the document. The E & E Project Manager is responsible for providing the most current copy of the QAPP and other planning documents to the project team members.

In addition to the QAPP and other planning documents, the primary documentation for the project are field records and analytical data packages. Requirements for field records are documented in E & E Standard Operating Procedures (SOPs) for Field Activities Logbooks and are described briefly below. Requirements for analytical data packages are also described below. The remainder of the QAPP

describes additional project documentation and record requirements for QA/QC assessments, data validation, data management, and other areas.

### 1.6.1 Field Documentation

#### Sample Identification

Samples will be identified using the format described below. Each sample will be labeled, chemically preserved (if required), and sealed immediately after collection. To minimize handling of sample containers, labels will be completed prior to sample collection as practicable. The sample label will be completed using waterproof ink and will be firmly affixed to sample containers and protected with clear tape. The sample label will give the following information:

- Date of collection;
- Unique sample number;
- Analyses requested; and
- Preservation.

Each sample will be referenced by sample number in the logbook and on the chain-of-custody (COC) record.

Individual samples will be identified by a unique alphanumeric code. Normal field samples (non-quality-control) will be numbered according to the following convention:

EMC-###-SD-Q

EMC - Three letter code for site name
SD - Matrix code of SD for Sediment. This code also could be CO for concrete or WP for wipe.
### - Sequential sample number
Q - Quality control sample code such as D for duplicate.

Samples collected with an additional volume for matrix spike/matrix spike duplicates (MS/MSD) will be designated on the COC.

#### Daily Logs

The Daily Log is the responsibility of the site Team Leader and will include a complete summary of the day's activities at the site and any communications outside the project team. The Daily Log will include:

- Name of the person making the entry (signature);
- Names of team members, subcontractors, and visitors on site;
- Levels of personal protection equipment (PPE):
  - Level of protection originally used,
  - Changes in protection, if required, and
  - Reasons for changes;
- Time spent collecting samples;
- Drilling information, including:
  - Method employed,
  - Diameter of borehole and well casing,
  - Materials used, and
  - Depth of borehole;
- Documentation on samples collected, including:
  - Sampling location and sample identification number,
  - Sampling depth,
  - Flow rate of water,
  - Sampling date, time, and personnel,
  - Sample sequence (order in which samples were collected),
  - Equipment used,
  - Type of sample (e.g., grab, composite, QC) and matrix,
  - Amount of each subsample or aliquot (if sample is a composite); and
  - Sample preservation and verification of preservation;
- Types of field QC samples, including when and where they were collected.
- Field equipment used, equipment identification numbers, and calibration information;
- On-site measurement data;
- Field observations and remarks;
- Weather conditions;
- Decontamination procedures;
- Unusual circumstances or difficulties; and

- Initials of person recording information (if a team member other than the Team Leader records information).

### Corrections to Documentation Notebook

As with any data logbooks, no pages will be removed for any reason. If corrections are necessary, they must be made by drawing a single line through the original entry (so that the original entry can still be read) and writing the corrected entry alongside. The correction must be initialed and dated. Most corrected errors will require a footnote explaining the correction.

### Photographs

Photographs will be taken of the core locations. The following information will be noted in the daily log concerning photographs:

- Date, time, location, and direction photograph was taken;
- Description of the photograph taken;
- Sequential number of the digital photo; and
- Camera system used.

### 1.6.2 Laboratory Data Reporting

The data packages for all analytical services must be consistent with NYSDEC Analytical Services Protocol (ASP) Category B (July 2005) for the sediment core samples. Laboratory reports for PCB screening do not need to be fully compliant with NYSDEC Category B CLP summary forms. Any laboratory standard reporting forms includes results of screening samples and associated QC is acceptable. The laboratory must provide copies of the chromatograms for the samples and standards against which they were calibrated. The laboratory will provide an electronic data deliverable that matches all data reported on the hard copy analytical report. Electronic data report requirements are described in Section 2.10.

The analytical summary report will include the sample aliquot analyzed, final extract volume, and dilution factor. The analytical summary data report also will include the laboratory reporting limit and method detection limit (MDL) for all target compounds. These limits will be corrected for percent moisture and all dilution factors. Any compounds found less than the reporting limit, but greater than the MDL will be reported and qualified with a “J” flag as estimated.

QC reports will provide a summary report or batch identifier clearly linking all QC results to actual field sample results. QC summary reports will include the laboratory control limits and flag any result reported outside control limits. The case narrative must include an explanation of all QC results reported outside con-

trol limits. The laboratory must provide copies of any nonconformance or corrective action forms associated with data in the laboratory report.

For Category A, the laboratory should provide copies of chromatograms for any samples for which elevated reporting limits are used because of sample matrix, but no target compounds are found above the reporting limit.

### 1.6.3 Record Retention

All records related to the project must be stored in secure areas consistent with requirements in E & E's QMP. All records related to the analytical effort will be maintained at the laboratory in lockable filing cabinets for at least one year, except those stored in the computer. All records must be maintained in a secure area for a period of six years after the end of the calendar year in which the final report is issued.

# 2 Data Generation and Acquisition

This section of the QAPP contains descriptions of all aspects of the implementation of field, laboratory and data handling procedures to meet the requirements of the Eighteenmile Creek PCB Trackdown Study activities.

## 2.1 Sampling Process Design

The purpose of the sampling project is to further determine the nature and extent of PCB contamination in the work area sediments, as well as determine the source(s) of this contamination. In order to obtain a full picture of the contamination present in the project area a large number of samples need to be collected. To defray the substantial costs associated with PCB sampling and analysis, a modified laboratory screening procedure or immunoassay test kit will be used to screen 80 preliminary sediment sore samples. Four field duplicate samples will be collected to document analytical sampling integrity of the results. Screening samples will quantify a total concentration of PCB's at a depth of 18 inches. All screening samples will also be analyzed for percent solids. Upon completion of sediment screening sampling and analysis, potential hot spots will be identified and sediment chemistry samples will be collected. These samples will allow project personnel to determine the levels of contaminants present in the sediments and further lead to pinpointing possible sources of contamination.

The collection of preliminary screening samples will begin just downstream of the remnants of the Clinton Street Dam, located just south of the creek's intersection with Olcott Street. Preliminary screening will also be completed in the Mill Race, located to the east of the creek's main branch. The collection of screening samples will end at a location just north of the creek's confluence with the Mill Race, located south west of the Mill Street and Center Street intersection. Figure 1-2 presents an overview of the project area. Screening samples will be collected approximately every 30 feet along the 2,500-foot project area. Screening samples will be collected in a phased approach so that the sampling locations can be modified based on the results of the screening samples.

Samples of the transformer area also may be collected for screening. The samples could potentially include sample wipes or concrete cores to assess if there is historical contamination on the pad itself.

Sediment core sample locations will be determined based upon the results of the preliminary PCB screening round. A total of 36 sediment core samples will be collected to a depth of 3 feet. PCB's will be analyzed at 1-foot intervals. All sediment core samples will also be analyzed for percent solids. A smaller subset (12) of the core samples taken will be analyzed for arsenic, chromium, copper, lead and mercury as a composite of all 3 feet. A second subset (12) of the core samples collected will be analyzed for Total Organic Carbon (TOC) as a composite of all 3 feet. PCB samples are critical to this study. TOC data are used to correct the PCB results for comparison to sediment criteria.

The analysis of lead contamination in the project area is also critical because of elevated levels of lead detected in this area. All other metal analyses and TOC composites are secondary in nature and are useful for trend analysis of other contaminants present in the AOC. A full description of the types and number of samples planned to be collected can be found in Table 1-2.

All preliminary screening samples as well as sediment core samples will be referenced using a Differential Global Positioning System. Core sample locations identified at the completion of the screening results are tentative and may be relocated by the on-site field coordinator or project manager during sampling. This will be dependent upon, but not limited to; site characteristics and ability of the sampling team to collect sufficient sample material required for a 3-foot core sample.

The results of all sediment data collected will be evaluated against the New York State Technical Guidance for Screening Contaminated Sediments to determine whether sediments can be removed from the environment and its ultimate fate after removal. Table 2-1 summarizes the types of data and analyses to be collected at each type of sampling location.

**Table 2-1 Summary of Data and Analysis Collected at Sampling Locations**

| Screening Samples (80 Locations) | Core Samples (36 Locations) |
|---|---|
| Sediment Chemistry (PCB) | Sediment Chemistry |
| Sediment Depth | Sediment Depth |
| Physical Description of Samples | Physical Description of Samples |
| Sample Locations (Lat.,Long.) | Sample Location (Lat,Long) |
| | Photographs of Samples |

### Field Data Collection

Two sets of field data will be collected that are critical to the data quality objectives for this project.

Latitude/Longitude Location: This data is critical for use in determining where sediment samples were collected. A Differential Global Positioning Systems (DGPS) capable of ascertaining horizontal locations with < 5 meters of accuracy will be utilized. To achieve this accuracy, it is important that the DGPS is in good working order and are obtaining strong satellite signals. The field team will be responsible for checking the satellite signal strength for the DGPS system prior to recording this data and for ensuring that the system records equivalent horizontal locations. Any problems with signal strength shall be recorded in the field boring log. If problems are noted, the field team should provide a qualitative description of the sampling location utilizing any available, permanent landmarks.

Sediment Depth: Sediment depth data is critical for determining the volume of sediments with a potential for contamination. Sediment depth will be measured to the nearest 0.1 foot.

## 2.2 Sampling Methods

The sampling methods are documented in E & E's sampling SOPs. In general, sampling at a site will progress from clean areas to contaminated areas. This minimizes the potential for cross contamination of samples and, subsequently, eliminates data anomalies or misinterpretation of the extent of contamination.

### Screening Sediment Sampling Methodology

- Collect sufficient sample volume to be analyzed using a disposable stainless-steel spoon or trowel or small hand coring device. Mix the sample thoroughly removing all stones and debris in disposable pie tin, and fill the appropriate sample containers;

- Decant excess liquid as necessary and secure jar;

- Upon collection, place the samples in a cooler maintained with ice at 4°C; and

- Package and ship samples in accordance with the procedure specified in Section 2.3.

Samples for concrete will involve scraping the concrete pad with trowel or coring device. Samples will be placed directly in the container.

## Sediment Core Sampling Methodology

### Equipment and Supplies

- Boat (including life preservers, oars, and two anchors) where needed;
- Weighted measuring tape;
- A 2-inch diameter by 3-foot long hand core sediment sampler (and extension rod for deep areas);
- Disposable/dedicated acetate core liner tubes;
- Dedicated plastic sediment catchers;
- Hand shovel;
- Hand saw;
- Dedicated stainless-steel spoons;
- Dedicated stainless-steel bowls;
- Plastic sheeting;
- Precleaned glass containers (see Table 2-2), equipped with teflon-lined lids;
- Coolers with ice; and
- Decontamination solutions (alconox, 10% nitric acid, and deionized water).

### Sampling Procedures

- Assemble the sampler by placing a dedicated liner and catcher in the core tube;
- Measure and record the depth of water at each sample location;
- Push hand core sediment sampler to a depth of 3 feet below channel bottom or refusal;
- Extract sampler and remove catcher and plastic liner;

- Either extrude sediment sample from liner onto clean dedicated plastic, or cut the liner and sediment core with a hand saw at the appropriate depth intervals. If a location is accessed by boat, then place caps on both ends of the liner and extrude on shore;

- Segregate sediment core into three depth intervals (0 to 1 foot, 1 to 2 feet, and 2 to 3 feet below the channel bottom), place each portion in separate stainless-steel bowls, homogenize, then place in appropriate jars, and label accordingly;

- Immediately place the sample containers in a cooler with ice to maintain sample temperature at 4°C; and

- Clean sampler with an alconox wash, 10% nitric rinse, and triple deionized water rinse prior to collecting the next sample.

## 2.3 Sample Handling and Custody

### 2.3.1 Sample Containers

The volumes and containers required for sampling activities are indicated in Table 2-2. Prewashed sample containers will be provided by the laboratory and will be wide-mouth jars with Teflon-lined caps unless otherwise indicated. The laboratory must use an approved specialty container supplier, which prepares containers in accordance with EPA bottle-washing procedures. The laboratory must maintain a record of all sample bottle lot numbers shipped in the event of a contamination problem.

### 2.3.2 Samples Preservation and Holding Times

A list of preservatives and holding times for each type of analysis are indicated in Table 2-2. Additional preservation requirements and holding times for non-target analyses are listed in the NYSDEC ASP.

Field personnel should record the date used in the field, site name, and E & E project number on the label or in the site logbook. Fresh sample containers and preservatives will be obtained from laboratory stocks prior to mobilization for each sampling event. Sample preservation will be recorded in the logbook and verified in the laboratory. If samples are improperly preserved, a corrective action form will be submitted to the laboratory project manager for follow-up action. The laboratory will notify the Project Manager to implement corrective action in the field.

Samples will be delivered to the laboratory within two days of sample collection to ensure the holding times listed in Table 2-2 are met.

**Table 2-2 Summary of Analytical Methods, Preservatives, and Holding Times**

| Parameter | Method[c] | Containers for Solid Samples | Preservatives[a] | Holding Time[a] |
|---|---|---|---|---|
| **Site Characterization Analysis** | | | | |
| TCL PCBs | 8082 | One 8-oz. glass jar | Cool to 4°C | 7 days/40 days[b] |
| Arsenic, Chromium, Copper, Lead, and Mercury | 6010/7470 | One 8-oz. glass jar | Cool to 4°C | 180 days/28 days for mercury |
| TOC | Lloyd Kahn | One 2-oz. glass jar | None | 28 days |
| **Screening Samples** | | | | |
| PCBs | Screening (modified SW8082) | One 4-oz. glass jar | Cool to 4°C | 7 days/40 days[b] |

[a] All samples to be cooled to 4°C. Holding times listed above are from the date of sample collection and for purposes of data review and are consistent with NYSDEC validation requirements. NYSDEC ASP holding times are based on verified times of sample receipt and will be applied for contractual purposes.

[b] Holding time is 7 days from collection to extraction and 40 days from extraction to analysis.

[c] Method references are from EPA publications SW-849, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," unless otherwise noted.

Key:

EPA = U.S. Environmental Protection Agency.
oz. = Ounce.
PCBs = Polychlorinated biphenyls.
TCL = Target Compound List.

### 2.3.3 Sample Handling

The transportation and handling of samples must be accomplished in a manner that not only protects the integrity of samples but also prevents any detrimental effects due to the possible hazardous nature of the samples. Regulations for packaging, marking, labeling, and shipping of hazardous materials are promulgated by the DOT in 49 CFR 171 through 177. E & E trains all staff responsible for the shipment of samples in these regulations. Procedures for sample packing and shipping are documented in an E & E SOP.

#### Sample Packaging

Samples must be packaged carefully to avoid breakage or contamination and must be shipped to the laboratory at proper temperatures. Samples will be transported and hand delivered to the laboratory at the end of the sampling day will reduces the need for extensive packaging.

### 2.3.4 Sample Custody

Formal sample custody procedures begin when the precleaned sample containers leave the laboratory or upon receipt from the container vendor. The laboratory must follow written and approved SOPs for shipping, receiving, logging, and internally transferring samples. Sample identification documents must be carefully prepared so that sample identification and COC can be maintained and sample disposition controlled. Sample identification documents include:

- Field notebooks;
- Sample labels;
- Custody seals; and
- COC records.

The primary objective of COC procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from sampling through completion of all required analyses. A sample is in custody if it is:

- In a team member's physical possession;
- In a team member's view;
- Locked up; or
- Kept in a secured area that is restricted to authorized personnel.

### Field Custody Procedures

Precleaned sample containers will be relinquished by the laboratory to the Field Team Leader. The Field Team Leader will record receipt of the sample containers in the project logbook. The following field custody procedure will be used for collection of samples:

- As few persons as possible should handle samples;
- Coolers or boxes containing cleaned bottles should be sealed with a custody tape seal during transport to the field or while in storage prior to use;
- The sample collector is personally responsible for the care and custody of samples collected until they are transferred to another person or dispatched properly under COC rules;
- The sample collector will record sample data in the field logbook; and
- The Field Team Leader will determine whether proper custody procedures were followed during the fieldwork and decide if additional samples are required.

### Chain-of-Custody Record

The COC form must be fully completed in duplicate by the field technician designated by the Project Manager as responsible for sample shipment to the appropriate laboratory for analysis. In addition, if samples are known to require rapid turnaround in the laboratory because of project time constraints or analytical concerns (e.g., extraction time or sample retention period limitations), the person completing the COC record should note these constraints. The custody record also should indicate any special preservation techniques necessary or whether samples need to be filtered. Copies of COC records are maintained with the project file.

### Custody Seals

Custody seals are preprinted, adhesive-backed seals with security slots designed to break if the seals are disturbed. Custody seals are placed over the cap of individual sample bottles by the sampling technician. DOT-approved sample shipping containers are sealed in as many places as necessary to ensure security. Seals must be signed and dated before use. Upon receipt at the laboratory, the custodian must check and document on a cooler receipt form that seals on boxes and bottles are intact.

## 2.4 Analytical Method Requirements

Analytical method requirements are documented in Table 2-2. The specific implementation of analytical methods will be documented in laboratory SOPs.

Laboratory SOPs and the QA program will be reviewed and approved as part of the procurement process. The laboratory SOP for PCBs is including in Appendix B.

The laboratory is certified by NYSDOH for all analytical methods and NELAP approved by NYSDOH.

Table 2-1 lists all analyses that may be performed for the Eighteenmile Creek PCB Trackdown Study. Reporting limits for analyses are provided in Appendix A to this QAPP.

## 2.5 Quality Control

QC data are necessary to determine precision and accuracy and to demonstrate the absence of interferences and/or contamination of glassware and reagents. Field QC will include duplicates, trip blanks, field equipment blanks, and miscellaneous field QC samples. Field QC samples will be preserved, documented, and transported in the same manner as the samples they represent. Laboratory-based QC will consist of standards, replicates, spikes, and blanks. Method QC limits for analyses are provided in Appendix A to this QAPP.

### 2.5.1 Field Quality Control Samples

Field QC samples will be collected and analyzed at the frequency listed in Table 2-3.

**Table 2-3 Field Quality Control Guidelines, Eighteenmile Creek PCB Trackdown Study**

| QC Sample | Description | Acceptance Criteria and Corrective Action |
|---|---|---|
| Field Duplicate | One per matrix per 20 samples for each screening analysis. | RPDs are less than 50% for screening samples. If RPDs are high in the initial phase, then field procedures will be modified and samples in critical locations recollected. |
| Field Equipment Blank | One per equipment set per day for each analysis. Only equipment sets that are subject to decontamination require equipment blanks. Dedicated or disposal equipment does not require equipment blanks. | Equipment blanks are not anticipated for this project. If required blanks should be below the reporting limits or associated data flagged if less than 5 times blank. If significant contamination is present, field procedures may be modified and samples recollected. |

#### Duplicate Samples

Duplicate samples will be collected at the rate one duplicate per 20 project samples of the same matrix. Duplicate soil samples will be prepared by collecting equal aliquots from the same sample source and placing them in separate sample

bottles. Duplicate samples will be shipped with the samples they represent and will be analyzed in the same manner.

The RPD between the concentration in the original and duplicate sample measures the overall precision of the field sampling and analytical method. Field duplicates are evaluated by using two times the laboratory QC criteria for duplicates. If all other laboratory QC criteria are met, RPD results outside control limits indicate potential matrix effects. Significant deviations in RPD results of field duplicates are assessed to evaluate whether data can be used to contour PCB sources.

### Field Equipment Blanks

Field equipment blanks are blank samples (also called rinsate blanks) designed to demonstrate that sampling equipment has been properly prepared and cleaned before field use and that cleaning procedures between samples are sufficient to minimize cross-contamination. Field equipment blanks will be prepared in the field using an approved water source. Sampling of the water source may also be required if analyte-free water is not obtained from the lab. The field equipment blank will be preserved, documented, shipped, and analyzed in the same manner as the samples it represents. Equipment blanks will be collected at the rate of one sample per day, per equipment set.

An equipment set is all sampling equipment required to collect one sample. For example, one soil sample equipment set may include a stainless-steel bowl, a stainless-steel trowel, and a bucket auger. Samples collected with dedicated or disposable equipment do not require equipment blank samples.

Field equipment and trip blanks serve to demonstrate contamination-free procedures in the field and during sample transport. The goal is for field blanks to be free of contamination. Low-level contamination may be present, but must be less than five times the level found in associated samples. If contamination is greater, the sample results are qualified as non-detect at an elevated-reporting limit. If field blank contaminants are also present in the method blank, or are typical laboratory contaminants, or are not present in project samples, then no further action is required. All other sources of contamination must be investigated as part of the corrective action process. Sample results that do not meet quality objectives after qualification, re-sampling may be required. The Project Manager must determine potential changes in field procedures to eliminate contamination sources prior to re-sampling.

## 2.5.2 Laboratory Quality Control Analyses

Analytical performance is monitored through QC samples and spikes, such as laboratory method blanks, surrogate spikes, QC check samples, matrix spikes, matrix spike duplicates, duplicate samples, and duplicate injections (see Table 2-4).

**Table 2-4 Laboratory Quality Control Sample Guidelines, Eighteenmile Creek PCB Trackdown Study Projects**

| QC Sample | Description |
|---|---|
| MB | One per matrix per preparation batch for each analysis. |
| MSB | One per matrix per preparation batch for each analysis. The MSB must contain all target analytes of concern at the site. |
| Surrogate Spikes | All samples analyzed for organic methods. |
| MS/MSD | One per matrix per SDG for each analysis. The spike solution must contain a broad range of the analytes of concern at the site. The overall frequency of MS/MSD on project samples must be at least one set per 20 samples. |
| MS/MD | One per matrix per SDG for metals and general chemistry methods. The spike solution must contain a broad range of analytes of concern at the site. The overall frequency of MS/MD on the project samples must be at least one set per 20 samples. |

Key:

SDG = Sample Delivery Group.
MSB = Matrix Spike Blank.
MS/MD = Matrix Spike/Matrix Duplicate.
MS/MSD = Matrix Spike/Matrix Spike Duplicate.
MB = Method Blank.
TAL = Target Analyze List.

All QC samples are applied on the basis of a laboratory batch. Batches do not exceed 20 samples excluding associated field and laboratory QC samples. The QC samples associated with sample preparation include method blanks, laboratory control samples (LCSs) (also called matrix spike blanks [MSB] by NYSDEC), matrix spikes, and duplicates. The run batch represents all samples analyzed together in the run sequence. The run sequence is typically limited to 24 hours unless defined differently for the analytical method. The QC samples associated with the run sequence include calibration standards, instrument blanks, and reference standards. Sample delivery group (SDG) is all samples delivered in a seven day period, up to a total of 20 samples. Analytical Criteria are listed in Appendix A.

Instances may arise where high sample concentrations, nonhomogeneity of samples, or matrix interferences preclude achieving detection limits or associated QC target criteria. In such instances, data will not be rejected *a priori* but will be examined on a case-by-case basis. The laboratory will report the reason for deviations from these detection limits or noncompliance with QC criteria in the case narrative.

### Laboratory Method Blank

Laboratory method blanks serve to demonstrate a contamination-free environment in the laboratory. The goal is for method blanks to be free of contamination.

Low-level contamination may be present, but must be less than the level in samples as defined by the method SOP. If contamination is greater, samples are re-analyzed. If contaminants are present in the method blank but not in project samples, no further action is required. All sources of contamination that are not common laboratory contaminants as defined in the method SOPs must be investigated as part of the corrective action process. Sample results must not be blank subtracted unless specifically required by the analytical method.

### Surrogate Standards

Surrogate recoveries must be within QC criteria for method blanks and LCSs to demonstrate acceptable method performance. If surrogate recoveries are outside QC criteria for method blanks or LCSs, corrective action is required and the Project Chemist should be notified. Surrogate recoveries in the samples indicate the method performance on the particular sample matrix. Surrogate recoveries that are outside QC criteria for a sample indicate a potential matrix effect. Matrix effects must be verified based on review of recoveries in the method blank or LCS, sample reanalysis, or evaluation of interfering compounds. Sample clean-up procedures are required by the NYSDEC ASP must be implemented to alleviate potential matrix problems.

### Laboratory Control Sample

LCS recoveries must be monitored on control charts for all non-Contract Laboratory Procedure methods. Laboratory QC criteria must be established for each method and matrix using a minimum of 30 points. QC criteria should be updated annually for all non-CLP methods. The LCS recovery must be within the control limits to demonstrate acceptable method performance. Sporadic marginal failures of a few target analytes reported when greater than five target analytes are required are allowed as part of the data review guidance. If LCS recoveries are outside QC criteria for more than a few target analytes, recoveries are significantly low, or the compounds were detected in the samples, then corrective action is required. After corrective action is complete, sample re-analysis is required for failed parameters. If LCS recoveries exceed the QC criteria, and that parameter is not found in any samples, re-analysis is not necessary. For any other deviations from LCS control limits that can not be resolved by sample re-analysis within holding times, the Project Chemist must be notified immediately. If critical samples are affected, the Project Manager may determine that re-sampling is required.

### Matrix Spike Sample

MS recoveries are a measure of the performance of the method on the sample being analyzed. Field and trip blanks must not be chosen for spiking. MS recoveries outside the control limits applied to the LCS indicate matrix effects. Sample clean-up procedures may be warranted for samples with severe matrix effects. The laboratory should notify the Project Chemist of these instances to determine an appropriate corrective action.

### Matrix Spike Duplicate Sample

The MSD sample is commonly prepared in conjunction with the MS sample. The MSD is prepared from a separate portion of the sample and processed with the same additions as the MS. The MSD is prepared for methods that do not typically show concentrations of target analytes above MDLs, such as organic methods. The RPD between the recoveries in the MS and MSD measure the precision of the analytical method on actual project samples. For this project, QC criteria for RPDs are 20% for waters and 35% for soils unless the laboratory provides additional statistical criteria.

### Duplicate Sample

The duplicate is prepared for methods that typically show concentrations of target analytes above MDLs, such as metals and wet chemistry methods. The RPDs between recoveries in the original and duplicate measure the precision of the analytical method on the actual project samples. For this project, QC criteria for RPDs are 20% for waters and 35% for soils unless the laboratory provides additional statistical criteria.

If all other QC criteria are met, RPD results outside control limits indicate potential matrix effects. The laboratory should investigate significant deviations in the RPD results by observing the sample to determine any visual heterogeneity or reviewing sample chromatograms for matrix interference. If visual observation does not indicate a potential problem, the sample may be reanalyzed. Potential matrix effects are reported in the case narrative.

### Blind QC Check Samples

Types of blind QC check samples include external performance evaluation (PE) samples provided by an outside certifying agency and internal QC samples submitted for routine analysis by the laboratory QA officer. The laboratory must pass NYSDOH samples as part of the approval process. If methods are used that are not included in NYSDOH approval process, blind QC samples may be submitted to the laboratory to evaluate method performance.

## 2.6 Instrument/Equipment Testing, Inspection, and Maintenance

All laboratory and field instruments and equipment used for sample analysis must be serviced and maintained only by qualified personnel. Laboratory instrument maintenance procedures will be evaluated to verify that there will be no impacts on analysis of project samples due to instrument malfunction. For example, the laboratory must have duplicate instrumentation and/or major laboratory instruments (e.g., gas chromatograph/mass spectrometer, inductively coupled argon plasma spectrometry, atomic absorption spectroscopy) maintained under service

agreements with the manufacturer that require rapid respond by manufacturer-approved service agents.

### 2.6.1 Laboratory Equipment Maintenance

The laboratory must maintain a stock of spare parts and consumables for all analytical equipment. Routine preventive maintenance procedures should be documented in SOPs. Maintenance performed on each piece of equipment must be documented in a maintenance logbook. Daily checks of the laboratory deionized water and other support systems are required. The laboratory must operate backup instrumentation for most of its analytical equipment in the event of major instrument failure or have an alternative approached to ensure analytical work proceeds within holding times with no adverse impacts on data quality.

## 2.7 Instrument/Equipment Calibration and Frequency

All instruments and equipment used during sampling and analysis will be operated and calibrated according to the manufacturer's guidelines and recommendations, as well as criteria set forth in applicable analytical methodology references. Personnel properly trained in these procedures will perform operation and calibration of all instruments. Documentation of all field maintenance and calibration information will be maintained in the task logbook. Table 2-5 lists typical monitoring equipment used during fieldwork. All field personnel receive annual refresher training on the field operation of all health and safety related equipment, which includes calibration procedures. Brief descriptions of calibration procedures for major field instruments are listed on Table 2-4.

## 2.8 Inspection/Acceptance of Supplies and Consumables

Measures are established in E & E's QMP to assure that purchased material, equipment, and services, whether purchased directly or through contractors or subcontractors, conform to procurement documents. Documentation regarding the purchase of material, equipment, and services is prepared, reviewed, and approved in accordance with requirements set forth in the QMP and E & E subcontracting procedures.

Procedures for the procurement, inspection, maintenance and management of equipment and supplies for NCSWCD activities are documented in E & E's Government Property Procurement SOP. All field supplies and equipment will be procured as part of the contract and maintained by the technical team. Supplies and equipment will be inspected on receipt at the site to verify that the correct materials were received.

**Table 2-5 General Field Equipment and Calibration Procedures, Eighteenmile Creek PCB Trackdown Study**

| Instrument or Equipment | Description[a] | Field Calibration Procedure | Acceptability/ Performance Criteria | Responsible Personnel |
|---|---|---|---|---|
| Organic Vapor Analyzer (OVA) | Foxboro OVA 128 Flame Ionization Detector to provide continuous data on organic vapor concentrations. Unit must be Class I, Division 1, Grade A,B,C,D. Unit must have rechargeable battery, range of 0 to 1,000 ppm, and ultra-high purity hydrogen as fuel source. | Units are factory calibrated to remain with performance specification for an excess of 6 months. During field use, a carbon filter is used with the OVA to distinguish methane from other organics. When the OVA is used to screen samples (except samples for headspace analysis), periodic ambient air readings will also be recorded in the logbook. | A carbon filter must remove source of organic vapors other than methane (i.e., marker). Instrument must detect organic vapors without filter. | Site Safety Officer, Project Geologist |
| $O_2$ Explosimeter | Gastech Model GT302 gas monitor designed to simultaneously monitor areas for oxygen deficiency and dangerous levels of combustible gas. Units must be equipped with sample pumps and hoses to measure gases in a confined space. Range $O_2$ - 0 to 25%, LEL - 0 to 100%, $H_2S$ - 0 to 200 ppm, and CO - 0 to 999 ppm. Not all units have the additional capability to detect hydrogen sulfide or $H_2S$ or carbon dioxide. | Procedures for field calibration of the $O_2$/Explosimeter are as follows:<br>■ Inspect instrument to ensure entry and exit ports are clear;<br>■ Turn the switch to ON position;<br>■ Allow the meters to stabilize and then press the reset button;<br>■ Check the battery level;<br>■ Calibrate the oxygen meter to 20.8% by using the calibrate knob;<br>■ Adjust the explosimeter to zero by using the zero knob; and<br>■ Check alarm levels by adjusting the calibrate knob for oxygen levels and the zero knob for explosimeter levels and note the readings when the alarm sounds. Return readings to normal and depress the reset button. | Alarm must sound during calibration procedure. Battery must have sufficient charge for operation. Blocking the sample line probe and observing the drop of the flow indicator float checks flow system. If flow system is not functioning, return unit for repairs. | Site Safety Officer, Project Geologist |
| pH/Conductivity, Temperature, Dissolved Oxygen (DO), Oxidation Reduction (REDOX) Meter | Myron MYR-6P Meter designed for field use with battery operation. The unit contains separate pH, temperature, conductivity, DO, and ORP probes in one unit. | Before use, pH, specific conductance, DO, and ORP probes need to be calibrated or tested for responsiveness. The pH probe will be calibrated first. This is done by placing the probe in pH 7, then pH 4, standard solutions and adjusting the pH calibration knobs until the correct measurement is obtained. The ORP probe is then calibrated with the ORP standard solution (Zobell), and the DO probe is checked with a zero DO solution (solution of 20 ml of deionized water, 20 ml of sodium sulfite, and a trace of cobalt chloride). The probes should be rinsed with deionized water between each calibration solution and following calibration. Used calibration solution is to be discarded. Finally, the conductivity probe is checked with a solution of known conductivity. | Turbidity and DO ± 10%<br>pH ± 0.01 pH<br>Conductivity at ± 2% FSD<br>The instrument will be checked with a pH standard every 4 hours and at the end of the sampling day. If the response is greater than 0.2 unit more or less than the standard, complete calibration will be conducted. | Project Geologist, Sampler |

**Table 2-5 General Field Equipment and Calibration Procedures, Eighteenmile Creek PCB Trackdown Study**

| Instrument or Equipment | Description[a] | Field Calibration Procedure | Acceptability/ Performance Criteria | Responsible Personnel |
|---|---|---|---|---|
| Turbidity Meter | HACH 2100P Nephelometer designed for field use with battery operation. Range 0.01 to 1,000 NTU. | The unit is factory calibrated. Field procedures involve checking the unit's responsiveness at least once a day using factory supplied standards. The responsiveness should be checked on the 0 to 10 range, 0 to 100 range, and 0 to 1,000 range. | ± 10% | Sampler |
| Organic Vapor Meter (photo-ionization detector) | The photoionization detector is a portable, non-destructive trace gas analyzer. Units for site characterization must have a range of 0 to >2,000 ppm and a 10.6 or 11.7 eV lamp (e.g., MiniRAE 2000). Units for indoor air monitoring must have a range of 1 ppb to 2,000 ppm and a 10.6 eV lamp (e.g., ppbRAE Plus).Calibration check gas (e.g., isobutylene) must be provided with unit. | In the field, PIDs will be calibrated at the start of each day. If a significant change in weather occurs during the day (i.e., change in humidity or temperature) or if the unit is turned off for an extended period, then it will be recalibrated at that time. When a PID is used to screen samples in the field, periodic ambient readings will also be recorded in the logbook. The calibration procedure is described in the instrument operations manual that must be supplied with each unit. | Meter must be able to adjust properly using the span knob or the lamp may require cleaning. | Site Safety Officer, Project Geologist |

[a] Description is for typical equipment; equivalent units may be used.

## 2.9 Non-Direct Measurements

For data acquired from non-direct measurement sources include the following:

- Physical information such as descriptions of sampling activities and geologic logs;
- State and local environmental agency files;
- Reference computer databases and literature files; and
- Historical reports on a site and subjective information gathered through interviews.

Data from non-direct measurements will be reviewed and used as indicated in Section 1 and Table 1-2.

## 2.10 Data Management

The Field Team Leader will review all field data for accuracy. Any field data not provided by the laboratory will be entered into a database or spreadsheet.

The laboratory will provide an electronic data deliverable (EDD) for all analytical reports that is consistent with Automated Data Review (ADR) format.

The E & E technical team will process the EDD using ADR software to verify that criteria established in this QAPP (Appendix A) are met. The Project Chemist will review all laboratory and field data to verify the results against the hard copy and check for transcription errors. The Project Chemist will verify qualifiers added by ADR and add any data qualifiers. The ADR files will be processed to the Electronic Data Management System (EDMS). EDMS will be used to store all reviewed and approved data. Data that will appear on data tables for the report will be generated from EDMS. EDMS will serve as the central data source for all data handling operations.

The central EDMS database will be stored in a secure area on E & E's network with access limited to data management specialists designated by the Project Manager. The central EDMS database can be electronically linked to E & E's GIS/CAD systems, risk assessment programs, and other final data user models and statistical programs. Data users may enter additional electronic data such as risk-based criteria for comparison of results. This data will be stored in separate tables in the database and linked to the actual results. Any data from outside sources will include a description of the data, a reference to the source, and the date updated. Outside data will be checked prior to use verify that current values are used. The central EDMS database will be used to create tables for the final report.

# 3 Assessment and Oversight

E & E's assessment and oversight procedures will be implemented in accordance with the QMP. The QMP outlines general roles and responsibilities for the project team.

## 3.1 Assessment and Response Actions

E & E's overall assessment activities include management assessments, development of SOPs, and performance evaluations. Management assessments include weekly meetings and conference calls to evaluate project readiness and staff utilization. Assignment of qualified personnel, maintenance of schedules and budgets, and quality of project deliverables are verified as part of these assessments. The development of SOPs and performance evaluations are used to provide trained and qualified personnel for the project.

E & E's technical assessment activities include peer review, data quality reviews, and technical system audits (i.e., laboratory and field). Procedures for assessment and audit of data quality are described in Section 4 of this QAPP. Procedures for peer review and technical assessments are summarized briefly below.

Both overall and direct technical assessment activities may result in the need for corrective action. E & E's approach to implementing a corrective action response program for both field and laboratory situations is summarized briefly below. The NCSWCD QA Officer has stop work authority on all NCSWCD projects that may have negative quality impacts prior to completion of corrective actions.

### 3.1.1 Peer Review

E & E implements peer review for all project deliverables including work plans, QAPPs, draft and final reports, and technical memoranda. The peer review process provides for a critical evaluation of the deliverable by an individual or team to determine if the deliverable will meet established criteria, quality objectives, technical standards, and contractual obligations. The Project Manager will assign peer reviewers when the publications schedule is established. The publications staff will be responsible for ensuring all peer reviewers participate in the review process and approve all final deliverables. For technical memoranda and other project

documents, the Project Manager will be responsible for obtaining principal review and approval.

### 3.1.2 Technical Systems Assessments

The entire project team is responsible for ongoing assessment of the technical work performed by the team, identification of nonconformance with the project objectives, and initiation, implementation and documentation of corrective action. Independent performance and systems audits are technical assessments that are a possible part of the QA/QC program. The following describes types of audits conducted, frequency of these audits, and personnel responsible for conducting audits.

#### Field Audits

Field audits, if performed, are under the direction of the QA Officer. No field audit will be performed for this field program.

#### Field Inspections

The Project Manager will be responsible for inspecting all field activities to verify compliance of activities with project plans.

#### Laboratory Audits

The laboratory must implement a comprehensive program of internal audits to verify compliance of their systems with SOPs and QA manuals.

NYSDOH must certify the laboratory and will perform external systems audits at an approximate frequency of once a year. External audits include reviews of analytical capabilities and procedures, COC procedures, documentation, QA/QC, and laboratory organization. These audits also include analysis of blind samples for PE.

No laboratory audits are planned for this project.

### 3.1.3 Corrective Action

Corrective actions will be implemented as needed. In conjunction with the QA Officer and Laboratory QA Coordinator, the Project Manager is responsible for initiating corrective action and implementing it in the field and office, and the laboratory project manager is responsible for implementing it in the laboratory. It is their combined responsibility to see that all sampling and analytical procedures are followed as specified and that the data generated meet the prescribed acceptance criteria. Specific corrective actions necessary will be clearly documented in the logbooks or analytical reports.

### Field Situations

The need for corrective action in the field may be determined by technical assessments or by more direct means such as equipment malfunction. Once a problem has been identified, it may be addressed immediately or an audit report may serve as notification to project management staff that corrective action is necessary. Immediate corrective actions taken in the field will be documented in the project logbook. Corrective actions may include, but are not limited to:

- Correcting equipment decontamination or sample handling procedures if field blanks indicated contamination;
- Recalibrating field instruments and checking battery charge;
- Training field laboratory personnel in correct sample handling or collection procedures; and
- Accepting data with an acknowledged level of uncertainty.

After a corrective action has been implemented, its effectiveness will be verified. If the action does not resolve the problem, appropriate personnel will be assigned to investigate and effectively remediate the problem. Corrective actions recommended by NCSWCD personnel will be addressed in a timely manner.

### Laboratory Situations

Out-of-control QC data, laboratory audits, or outside data review may determine the need for corrective action in the laboratory. Corrective actions may include, but are not limited to:

- Reanalyzing samples, if holding times permit;
- Correcting laboratory procedures;
- Recalibrating instruments using freshly prepared standards;
- Replacing solvents or other reagents that give unacceptable blank values;
- Training additional laboratory personnel in correct sample preparation and analysis procedures; and
- Accepting data with an acknowledged level of uncertainty.

The laboratory corrective actions must be defined in analytical SOPs. Any deviations from approved corrective actions must be documented and approved by the Project Chemist.

## 3.2 Reports to Management

For reports to management include the following:

- **Data Usability Summary Report** - A DUSR will be completed by the Project Chemist and provided to the NCSWCD technical staff in the appendix of the report. Impacts on the usability of data will be tracked by adding qualifiers to individual data points as described in Section 4.

- **Project Status Reports** - Project status reports are completed by the Project Manager to document the overall assessment of the project on a monthly basis.

Upon completion of a project sampling effort, analytical and QC data will be included in a comprehensive technical report that summarizes field activities and provides a data evaluation. A discussion of the validity of results in the context of QA/QC procedures will be made and the DUSR will be provided.

Serious analytical problems will be reported immediately to NCSWCD personnel. Time and type of corrective action (if needed) will depend on the severity of the problem and relative overall project importance. Corrective actions may include altering procedures in the field, conducting an audit, or modifying laboratory protocol.

# 4 Data Validation and Usability

E & E will implement procedures for data validation and usability described below.

## 4.1 Data Review, Validation, and Verification Requirements

All data generated will be reviewed by comparing accuracy and precision results for the samples QC samples to QC criteria listed Appendix A using ADR. The following types of data will be reviewed through ADR:

- Analytical reporting limits and target compounds will be compared to limits listed in the ADR library;
- Holding times will be verified against Table 2-2;
- QC summary data for surrogates, method blanks, LCS, and MS/MSD samples. Acceptance and performance criteria will be compared to criteria in the project library as listed in Appendix A. The project library may be updated if current laboratory control limits are updated and then differ from the limits listed in the QAPP;
- Field QC results for duplicates and blanks will be compared to criteria listed in Table 2-3;
- Calibration summary data will be checked by the laboratory to verify that all positive results for target compounds were generated under an acceptable calibration as defined by the analytical method. Any deviations will be noted in the case narrative and reviewed by the Project Chemist;
- Field data such as sample identifications and sample dates will be checked against the laboratory report; and
- Any raw data files from the field and laboratory will not be reviewed unless there is a significant problem noted with the summary information.

## 4.2 Validation and Verification Methods

The laboratory is responsible for performing internal data review. The laboratory data review must include 100% analyst review, 100% peer review, and 100% review by the laboratory project manager to verify that all project-specific requirements are met. The laboratory QA officer must perform review on 10% of the data packages. All levels of laboratory review must be fully documented and available for review if requested or if a laboratory audit is performed.

After receipt from the laboratory, project data will be validated using the following steps:

### Evaluation of Completeness

The Project Chemist checks the ADR electronic files for compliance with ADR format and the project library. If errors in loading are found, the ADR files will be returned to the laboratory. The Project Chemist also verifies that the laboratory information matches the field information and that the following items are included in the hard copy data package:

- COC forms and NYSDEC Sample Summary forms;
- Case narrative describing any out-of-control events and summarizing analytical procedures;
- Data report forms (i.e., Form I);
- QA/QC summary forms; and
- Chromatograms documenting any QC problems.

If the data package is incomplete, the Project Chemist will contact the laboratory, which must provide all missing information within one day.

### Evaluation of Compliance

The ADR validation procedures process the electronic data and assign qualifiers if outliers are found. The Project Chemist will review all ADR processed files and approve all data qualifiers. Additional compliance checks on representative portions of the data are briefly outlined below:

- Review chromatograms and other raw data if provided as backup information for any apparent QC anomalies;
- Ensure that all analytical problems and corrections are reported in the case narrative and that appropriate laboratory qualifiers are added;

- For any problems identified, review concerns with the laboratory, obtain additional information if necessary, and check all related data to determine the extent of the error; and

- Project chemists will follow qualification guidelines in *USEPA CLP National Functional Guidelines for Organic Data Review, EPA 540/R-99-008* (October 1999) or *USEPA CLP National Functional Guidelines for Inorganic Data Review, EPA 540/R-94/013* (July 2002). The DUSR will be completed as specified in NYSDEC *Guidance of the Development of DUSRs* (July 1999).

### Data Review Reporting

The Project Chemist will perform the following reporting functions:

- Alert the Project Manager to any QC problems, obvious anomalous values, or discrepancies between the field and laboratory data, that may impact data usability;

- Discuss QC problems in a DUSR for each laboratory report. The DUSR will include a short narrative and print out of qualified data from ADR;

- Prepare analytical data summary tables of qualified data that summarize those samples and analytes for which detectable concentrations were exhibited including field QC samples; and

- At the completion of all field and laboratory efforts, summarize planned versus actual field and laboratory activities and data usability concerns in the technical report.

## 4.3 Reconciliation with User Requirements

Any deviations from analytical performance criteria or quality objectives for the project will be documented in the DUSR provided to the data users for the project.

The QA Officer or Project Chemist will work with the final users of the data in performing data quality assessments. The data quality assessment may include some or all of the following steps:

- Data that are determined to be incomplete or not usable for the project will be discussed with the project team. If critical data points are involved which impact the ability to complete project objectives, data users will report immediately to the Project Manager. The Project Manager will discuss resolution of the issue with NCSWCD technical staff and implement necessary corrective actions (for example re-sampling);

- Data that are non-detect but have elevated reporting limits due to blank contamination or matrix interference will be compared to screening values. If reporting limits exceed the screening values, then results will be handled as incomplete data as described above; and

- Data that are qualified as estimated will be used for all project decision making. If an estimated result is close to a screening value, then there is uncertainty in any conclusions as to whether the result exceeds the screening value. The data user must evaluate the potential uncertainty in developing recommendations for the site. If estimated results become critical data points in making final decisions on the site, the Project Manager and NCSWCD technical staff should evaluate the use of the results and may consider the data point incomplete.

The assessment process involves comparing analytical results to screening values and background concentrations to determine if the contamination present is site-related (i.e., above background levels) or significant (i.e., above screening values).

# A

# Laboratory Performance Criteria

# *Project Reporting Limits and QC Criteria*

**Project** PCB Trackdown Study

**Method: 6010B** **SO** Metals by Inductively Coupled Plasma-Atomic Emission

| *Analyte Name* | *Client Analyte ID* | *RL* | *RL Type* | *RL Units* | *MS Lower* | *MS Upper* | *MS RPD* | *MS Units* | *LCS Lower* | *LCS Upper* | *LCS RPD* | *LCS Units* | *Lab Dup* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ARSENIC | 7440-38-2 | 2.0 | PQL | mg/Kg | 75.00 | 125.00 | 20.00 | Percent | 79.71 | 120.18 | 20.00 | Percent | 20 |
| CHROMIUM | 7440-47-3 | 0.50 | PQL | mg/Kg | 75.00 | 125.00 | 20.00 | Percent | 78.59 | 121.18 | 20.00 | Percent | 20 |
| COPPER | 7440-50-8 | 1.0 | PQL | mg/Kg | 75.00 | 125.00 | 20.00 | Percent | 82.29 | 117.71 | 20.00 | Percent | 20 |
| LEAD | 7439-92-1 | 1.0 | PQL | mg/Kg | 75.00 | 125.00 | 20.00 | Percent | 80.59 | 119.49 | 20.00 | Percent | 20 |
| ZINC | 7440-66-6 | 2.0 | PQL | mg/Kg | 75.00 | 125.00 | 20.00 | Percent | 79.24 | 120.76 | 20.00 | Percent | 20 |

**Method: 7471A** **SO** Mercury in Solid or Semi-solid Waste by Manual Cold Vapor Technique

| *Analyte Name* | *Client Analyte ID* | *RL* | *RL Type* | *RL Units* | *MS Lower* | *MS Upper* | *MS RPD* | *MS Units* | *LCS Lower* | *LCS Upper* | *LCS RPD* | *LCS Units* | *Lab Dup* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| MERCURY | 7439-97-6 | 0.017 | PQL | mg/Kg | 75.00 | 125.00 | 20.00 | Percent | 67.22 | 130.00 | 20.00 | Percent | 20 |

**Method: 8082** **SO** Polychlorinated Biphenyls (PCBs) by GC using ECD

| *Analyte Name* | *Client Analyte ID* | *RL* | *RL Type* | *RL Units* | *MS Lower* | *MS Upper* | *MS RPD* | *MS Units* | *LCS Lower* | *LCS Upper* | *LCS RPD* | *LCS Units* | *Lab Dup* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| AROCLOR 1016 | 12674-11-2 | 17 | PQL | ug/Kg | 70.00 | 130.00 | 40.00 | | 70.00 | 130.00 | 40.00 | Percent | 0 |
| AROCLOR 1221 | 11104-28-2 | 17 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1232 | 11141-16-5 | 17 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1242 | 53469-21-9 | 17 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1248 | 12672-29-6 | 17 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1254 | 11097-69-1 | 17 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1260 | 11096-82-5 | 17 | PQL | ug/Kg | 70.00 | 130.00 | 40.00 | | 70.00 | 130.00 | 40.00 | Percent | 0 |

## Surrogates

### Method: 8082 SO

| *Analyte Name* | *Client Analyte ID* | *Surrogate Lower* | *Surrogate Upper* | *Surrogate Units* |
|---|---|---|---|---|
| DECACHLOROBIPHENYL | 2051-24-3 | 70 | 130 | Percent |
| TETRACHLORO-M-XYLENE | 877-09-8 | 70 | 130 | Percent |

### Method: 8082-Screen SO Polyclorinated Biphenyls (PCBs) Screening by GC

| *Analyte Name* | *Client Analyte ID* | *RL* | *RL Type* | *RL Units* | *MS Lower* | *MS Upper* | *MS RPD* | *MS Units* | *LCS Lower* | *LCS Upper* | *LCS RPD* | *LCS Units* | *Lab Dup* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| AROCLOR 1016 | 12674-11-2 | 250 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1221 | 11104-28-2 | 250 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1232 | 11141-16-5 | 250 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1242 | 53469-21-9 | 250 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1248 | 12672-29-6 | 250 | PQL | ug/Kg | | | | | | | | | 0 |
| AROCLOR 1254 | 11097-69-1 | 250 | PQL | ug/Kg | 60.00 | 140.00 | 40.00 | Percent | 60.00 | 140.00 | 40.00 | Percent | 0 |
| AROCLOR 1260 | 11096-82-5 | 250 | PQL | ug/Kg | | | | | | | | | 0 |

## Surrogates

### Method: 8082-Screen SO

| *Analyte Name* | *Client Analyte ID* | *Surrogate Lower* | *Surrogate Upper* | *Surrogate Units* |
|---|---|---|---|---|
| DECACHLOROBIPHENYL | 2051-24-3 | 60 | 140 | Percent |
| TETRACHLORO-M-XYLENE | 877-09-8 | 60 | 140 | Percent |

# B

# PCB Laboratory SOP

# STL Buffalo

## LABORATORY STANDARD OPERATING PROCEDURES

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| SOP No. | Revision No. | Effective Date | Page |
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**TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES**

**SUPERCEDES: Revision 8**

| REVIEWED & APPROVED BY: | SIGNATURE | DATE |
|---|---|---|
| Kathleen E. Aldrich, Supervisor | Kathleen E. Aldrich | 10/25/04 |
| Verl D. Preston, Quality Manager | Verl D. Preston | 10/25/04 |
| Christopher Oprandi, Laboratory Director | Christopher R. Oprandi | 10/25/04 |



## 1.0 IDENTIFICATION OF TEST METHOD

1.1 Method 3550B: Ultrasonic Extraction.

## 2.0 APPLICABLE MATRIX

2.1 Soils, sediments and wipes

## 3.0 REPORTING LIMIT N/A

## 4.0 SCOPE AND APPLICATION

4.1 This method is used for the extraction of nonvolatile and semivolatile organic compounds from solids and wipes. The ultrasonic process used ensures thorough contact of the sample with the extraction solvent.

## 5.0 SUMMARY OF THE TEST METHOD

5.1 Low Level

5.1.1 A 30 gram sample is mixed with anhydrous sodium sulfate. This is solvent extracted three times using ultrasonic extraction. The extract is then filtered and

concentrated. The extract may then be subject to clean-up procedures or sent directly for analysis.

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5.2 **Medium/High Level**

5.2.1 A 2 gram sample is mixed with anhydrous sodium sulfate and solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

5.3 **Wipes**

5.3.1 A wipe sample is mixed with anhydrous sodium sulfate and solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

## 6.0 DEFINITIONS

6.1 Standard definitions are found in Section 3.0 of the Laboratory Quality Manual.

## 7.0 INTERFERENCES

7.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

7.2 Matrix interference may be caused by contaminants that are co-extracted from the sample.

## 8.0 SAFETY

8.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

8.2 All parameters of this extraction must be performed in an operational fume hood or within an extraction apparatus that is ventilated by the fume hood system.

8.3 Any excess unextracted sample (including dry weights) waste will be disposed of in "BE" waste. Solid waste generated in the extraction process will be disposed of in "BC" waste. All solvent and extract waste is disposed of in "C" waste.

8.4 Safety glasses, gloves, and lab coats must be worn at all times. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP and should not be used.

8.5 All solvents, reagents, and standards must be handled inside a fume hood and with proper personal safety equipment due to their hazardous properties. All samples must be opened inside a fume hood due to their unknown hazardous properties.

8.5.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|---|---|---|---|
| Methylene Chloride | Carcinogen Irritant | 25 ppm-TWA 125 ppm-STEL | Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin. |
| Acetone | Flammable | 1000 ppm-TWA | Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. |
| Hexane | Flammable Irritant | 500 ppm-TWA | Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

8.6 Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection.Each lab technician working in the general area for extended periods of time should wear earplugs.

## 9.0 EQUIPMENT AND SUPPLIES

9.1 Aluminum Dishes, Foil
9.2 Metal Spatula
9.3 Toploader Balance

9.4 Syringes
9.5 ¾ in. dual horn Sonicators® with Sonabox® acoustic enclosures
9.6 16 oz. french squares
9.7 Ovens - 104°C and 400°C
9.8 16 oz. wide mouth jars
9.9 Turbovap concentrators and vessels
9.10 Stainless steel filter funnels
9.11 Graduated cylinders
9.12 Ear Protection
9.13 2,10 and 24ml vials, septa and caps
9.14 Disposable pipets and pipet bulbs
9.15 18.5 cm #41 filter paperMicrotip horn Sonicators® with Sonabox® acoustic enclosures

## 10.0 REAGENTS AND STANDARDS

10.1 All solvents are pesticide grade or equivalent.

10.1.1 Hexane
10.1.2 Compressed Nitrogen
10.1.3 Granular sodium sulfate, previously baked in a 400°C oven for a minimum of 4 hours, cooled and dried in a dessicator, and rinsed with methylene chloride.
10.1.4 Methylene Chloride
10.1.5 Acetone
10.1.6 Surrogate and spike solutions appropriate to the final determinative procedures as assigned by test profile (See Table 2).

## 11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 All samples must be stored in a glass amber sample container and stored at 4°C. The sample is stored unpreserved and unfiltered unless otherwise requested by the client.

11.2 Typical method holding time for soil samples is fourteen days from sampling. However, the client may impose a more strict time constraint.

## 12.0 QUALITY CONTROL

12.1 All batches (20 samples or less) will contain a matrix spike blank (MSB) and method blank (MBLK) when a matrix spike (MS) and matrix spike duplicate (SD) are supplied. When client-specific QC is not assigned or there is not sufficient volume to assign QC samples, a matrix spike blank (MSB), matrix spike blank duplicate (MSBD) and a method blank (MBLK), will be assigned. All reagent blanks and matrix spike duplicates will undergo the same procedure as the samples.

**TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES**

**SUPERCEDES: Revision 8**

## 13.0 CALIBRATION AND STANDARDIZATION N/A

## 14.0 PROCEDURE

**Note:** All samples must be signed out of the sample logbook and signed back in when returned. If the entire sample is to be used for the extraction, write "disc" for discard in the "TIME IN" column.

### 14.1 Low Level Extraction:

**14.1.1** Decant and discard any standing water on sample. On the batch sheet, mark a "Y" for "yes" or an "N" for "no" in the "Decant" column. Discard any sticks, leaves, rocks or other foreign matter.

**14.1.2** Transfer at least 90 to 100 grams of the sample to the labeled sonication jar. Homogenize the sample thoroughly. Transfer all but 30 grams of the sample back to the original sample jar.

**14.1.3** Mark aluminum dish on bottom with the last three digits of the STL vial number. Weigh and record the weight of the dish.

**14.1.4** Weigh 5 – 9 grams of recently homogenized sample into the dish and record the combined sample plus dish weight. Place in the dry weight oven for at least four hours prior to dry weight determination.

**14.1.5** Add granular sodium sulfate to the 30g sample and blend with a spatula until the sample is free flowing.

**14.1.6** If the sample is excessively wet or needs to be decanted prior to homogenization, add the sodium sulfate to the sample, mix and let the sample sit for ten minutes. This time allows the sodium sulfate to absorb the water from the sample, however it will also harden the sample. After the sample sits, it will be necessary to break up the sample with the spatula until a free flowing consistency is again achieved.

**14.1.6** Add surrogate (See Table2) to the samples using the appropriate surrogate as designated on the batch sheet. Write an "X" on the label after adding the surrogate.

**14.1.8** Add appropriate spike (See Table2) to samples designated MS, MSD, MSB, MSBD. (The spike code to be used appears on the preparation batch logbook sheets.) Circle the "X" after adding the appropriate spike.

**14.1.9** For blank samples, approximately 30g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

**14.1.10** Add 100mls of appropriate solvent to the sample; the solvent for determinative methods is as follows:

8080,8081,8082 - 1:1 acetone/hexane
8270 – Methylene chloride
DRO - Methylene chloride
*8270 soils for specific clients (Columbia) and USACE projects will be extracted with 1:1 Methylene chloride/Acetone

**14.1.11** Fold an 18.5 cm filter paper into quarters and place it in a powder funnel.

**14.1.12** Place this funnel in a labeled french square bottle, wrapped in a napkin to prevent condensing water from entering the sample.

**14.1.13** Place the 16 oz. Glass sonication jar under the sonication horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch. In the case of excessively wet samples that needed a great deal of $MeCl_2$, more solvent may be added and the position of the sonicator jar adjusted to the ideal parameters.

**14.1.14** Sonicate for 3 minutes at out put setting 10, pulsed mode, 50% duty cycle, using ¾ inch horn.

**14.1.15** Collect the extract in a labeled french square jar by first decanting the extract through the filter funnel containing the 18.5 cm filter paper folded inside. When using solvents with acetone, add a little sodium sulfate to the filter paper to reduce the amount of water in the extract.

**14.1.16** Add 100ml of appropriate solvent to the sample.

**14.1.17** Repeat steps 14.1.13, 14.1.14, 14.1.15, and 14.1.16

**14.1.18** Repeat steps 14.1.13, 14.1.14, and 14.1.15

**14.1.19** After the third sonication, rinse the contents of the sonication jar into the funnel.

**14.1.20** After sample has drained, rinse down the funnel with 20-30mLs of the extraction solvent being used.

**14.1.21** Clean the sonicator horn between samples with DI water, acetone, and solvent being used. Wipe the horns with paper towels after the DI water rinse.

**14.2 Concentration Procedure**

**14.2.1** Pour the extract into a labeled turbovap vessel that is pre-rinsed with $MeCl_2$, rinse the french square with the appropriate solvent and add this to the turbovap vessel.

**14.2.2** Place the vessel in the turbovap at a water temp of 32°C and turn on the nitrogen to concentrate the extract to approximately 1ml. During concentration, the turbovap vessel should be periodically rinsed with the extraction solvent.

**14.2.3** For 8270 and DROs, bring the volume to exactly 1ml using the calibrated 1.0ml mark on the turbovap vessel. Transfer this to a 2ml vial using a dipsosable 9 inch pipette. The entire 1.0mL volume must be transferred to the vial. If any sample is spilled during the transfer to the vial, it must be noted in the comment section of the batch sheets. 8270 samples can be relinquished to GC/MS for analysis and DRO samples can be relinquished to GC for analysis.

**14.2.3** For 8080, 8081, 8082 the extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives. If proceeding directly to analysis, bring the volumeto 1.0ml using the calibrated 1.0ml mark on the turbovap vessel then adjust the final volume to 10.0ml by adding 9.0ml of Hexane to the turbovap vessel with a repipetter. Transfer 1ml to a 2ml vialusing a disposable pipette, mark the meniscus on the vial and relinquish to GC for analysis. Vial a second one mL aliquot and store this in the sample incubator with the necessary label for future reference. This needs to be stored for a period no less than one month. The remaining 8 mLs can now be disposed of by trained personnel.

**14.3 MEDIUM LEVEL EXTRACTION:**

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**14.3.1** Decant and discard any standing water on sample. On the batch sheet, mark a "Y" for "yes" or an "N" for "no" in the "Decant" column. Discard any sticks, leaves, rocks or other foreign matter.

**14.3.2** Homogenize the sample by transferring all contents of the sample jar into a clean disposable 16oz wide mouth jar and mixing thoroughly. Transfer 2 grams of the sample into a tared 24 mL extraction vial. Return the remaining sample back to the sample jar and discard the 16oz wide mouth jar.

**14.3.3** Mark aluminum dish on bottom with the last three digits of the STL vial number. Weigh and record the weight of the dish.

**14.3.4** Weigh 5 – 9 grams of recently homogenized sample into the dish and record the combined sample plus dish weight. Place in the dry weight oven for at least four hours prior to dry weight determination.

**14.3.5** Add granular sodium sulfate to the 2g sample and blend with a spatula or disposable tongue depresser until the sample is free flowing.

**14.3.6** If the sample is excessively wet or needs to be decanted prior to homogenization, add the sodium sulfate to the sample and let the sample sit for ten minutes. This time allows the sodium sulfate to absorb the water from the sample, however it will also harden the sample. After the sample sits, it will be necessary to break up the sample with the spatula until a free flowing consistency is again achieved.

**14.3.6** Add surrogate to the samples using the appropriate surrogate (See Table 2) as designated on the batch sheet. Write an "X" on the label after adding the surrogate.

**14.3.8** Add appropriate spike (See Table 2) to samples designated MS, MSD, MSB, MSBD. (The spike code to be used appears on the preparation batch logbook sheets.) Circle the "X" after adding the appropriate spike.

**14.3.9** For blank samples, approximately 2 g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

**14.3.10** Add 9.0 mLs of hexane to the sample if 100.0 □l of spike is used. If 1.0 mL of spike is used, add 8.0 mLs of hexane to the sample.

**14.3.11** Sonicate each sample once for 3 minutes on pulse mode of half power using a microtip sonicating horn.

**14.3.12** Decant the sample into a 24mL vial that is pre labeled with the appropriate vial number. Add 10 mLs of concentrated $H_2SO_4$, cap and shake for 2 minutes. Allow the solvent layer to separate from the acid layer (at least 10 minutes) and remove an aliquot from the top layer, vial in a 2mL vial and relinquish to GC for analysis. Remove a second aliquot and save in the extra volume incubator. The remaining 9 mLs can now be disposed of by trained personnel.

**14.4 Wipe Extraction**

**14.4.1** Place entire sample into a labeled 16oz. wide-mouth jar.

**14.4.2** Add anhydrous granular sodium sulfate.

**14.4.3** Add 1ml of surrogate (See Table 2) appropriate to the final determinative procedure to the sample (the surrogate code to be used will be printed out on the preparation batch logbook sheets).

**14.4.4** Add appropriate spike (See Table2) to samples designated MS, MSD, MSB, MSBD. (The spike code to be used appears on the preparation batch logbook sheets.)

**14.4.5** For blank samples, approximately 30g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

**14.4.6** Add 100mls of hexane to the sample.

**14.4.7** Fold an 18.5 cm filter paper into quarters and place it in a powder funnel.

**14.4.8** Place this funnel in a labeled french square bottle with a napkin wrapped around the outside to prevent condensing water from entering the extract.

**14.4.9** Place the 16 oz. Glass sonication jar under the sonication horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch.

**14.4.10** Sonicate for 3 minutes at out put setting 10, pulsed mode, 50% duty cycle, using ¾ inch horn

**14.4.11** Pour off the solvent and transfer the wipe to the funnel. Rinse the sonicator jar with 10 – 20 mLs of hexane and transfer rinse also to the funnel.

**14.4.12** After sample has drained, rinse down the funnel with 20-30mLs of the extraction solvent being used

**14.4.13** Clean the sonicator horn between samples with DI water, acetone, and hexane, wiping the horns with paper towels after the DI water rinse.

**14.4.14** For the wipes concentration procedure follow 14.2.

## 15.0 CALCULATIONS N/A

## 16.0 METHOD PERFORMANCE

**16.1** Acceptable performance is monitored through the use of Method Detection Limit Studies, as well as, recoveries of surrogate and spike compounds.

**TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES**

**SUPERCEDES: Revision 8**

**17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES N/A**

**18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA**

**18.1** A job exception will be filed if any of the following occur.

**18.1.1** Holding time is exceeded.
**18.1.2** Insufficient sample volume.
**18.1.3** Any matrix problems that prevent the extraction from being completed.

**18.2** If at any time during the extraction, sample or extract is spilled, it must be determined if extra volume exists. If extra volume exists, the sample will immediately be prepped again. If there is no more volume, a comment must be included in the batch comments section, indicating at which point sample or extract was lost.

**19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA N/A**

**20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION**

**20.1** The following waste streams are produced when this method is carried out.

**20.1.1** Waste Hexane in vials. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations.).

**20.1.2** Waste Methylene Chloride. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal).

**20.1.3** Waste solid material from the extraction process. (Solid wastes are separated into 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste) are marked as "BC waste" and extra solid sample volumes (dry weights and other unextracted solid waste) are marked as "BE waste". When full, a

designated laboratory technician will transfer all of the lab generated solid waste into a 55-gallon drum.)

**20.1.4** Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. (Solid wastes are separated into 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste) are marked as "BC waste" and extra solid sample volumes (dry weights and other unextracted solid waste) are marked as "BE waste". When full, a designated laboratory technician will transfer all of the lab generated solid waste into a 55-gallon drum.).

**20.1.5** Assorted flammable solvent waste from various glassware rinses. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

**20.1.6** Methylene chloride waste from various glassware rinses. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

**20.1.7** Miscellaneous disposable glassware contaminated with solvents and sample residue. (All disposable glassware contaminated with solvent is air dried inside an operational fume hood then disposed in the recycling receptacle).

**20.2** All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

## 21 REFERENCE

**21.1** USEPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW-846, Third Edition; Revision 2, December 1996; Method 3550B.

## 22 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

**22.2** Table 1: Organic prep worksheet

**22.3** Table 2: Spike and Surrogate Reference Sheet

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**SUPERCEDES: Revision 8**

## 23.0 CHANGES FROM PREVIOUS REVISION

**23.1.** 14.1.10 had the client exception list revised for the use of 1:1 Methylene chloride/Acetone
**23.2.** Fixed numbering in Sections 14.2 and 14.3
**23.3.** Revised homogenization technique for medium level soils, Section 14.3.2
**23.4.** 18.2 was added – contingency for sample spillage
**23.5.** Added Table 2 and all references to it.

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**TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES**

**SUPERCEDES: Revision 8**

**Table 1**

STL Buffalo
Date: 08/28/2003
Time: 17:31:24

Organic Prep Log Book
(3550B) 8270 HIGH SOILS
A3B09714

Rept: AN0501

SURROGATE: A01
Expiration Date: 12/4/04
Prepared by: JMM
Spiked by: MP
Witnessed by: _____
1000.00 ul

MATRIX SPIKE: AS5
Expiration Date: 12/23/03
Prepared by: JMM
Spiked by: MP
Witnessed by: _____
1000.00 ul

MeCl2: V07E38
Acetone: _____
Hexane: _____
Na2SO4: [illegible]
Conc.H2SO4: _____

Date Ext/Initials: 8/28/03 ME
Cleanup Date/Initials: _____

Preconc Date/Initials: _____
Final Conc Date/Initials: 8/28/03 ME

SOLID EXTRACTIONS

| Job Number | Sample ID | BT ID | Samp Type | Vial # | Test | Protoc | Method | Surr Code | Spike Code | Sample Weight (g) | Clean Up | Final Volume (ml) | Dish Wght | Comb Wet | Comb Dry | D* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A03-8302 | A3830201 | P | FS | AS30015699 | 8270STAR | SW8463 | 8270 | A00001 | | 30.98 | | 1.0 | 1.27 | 5.34 | 4.56 | N |
| A03-8302 | A3830201MS | | MS | AS30015700 | 8270STAR | SW8463 | 8270 | A00001 | A00055 | 30.04 | | | | | | |
| A03-8302 | A3830201SD | | SD | AS30015701 | 8270STAR | SW8463 | 8270 | A00001 | A00055 | 30.15 | | | | | | |
| A3B09714 | A3B0971401 | | MSB | AS30015702 | 8270STAR | SW8463 | 8270 | A00001 | A00055 | 30.92 | | | | | | |
| A3B09714 | A3B0971402 | | MBLK | AS30015703 | 8270STAR | SW8463 | 8270 | A00001 | | 30.14 | | | | | | |

Comments: _____

D* = Decanted (Y/N)

9/1/03

**TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES**

**SUPERCEDES: Revision 8**

**Table 2**
**SPIKE AND SURROGATE REFERENCE SHEET**

**SURROGATES**

**A001: 8270 BN/AP SURROGATE**

| | |
|---|---|
| Nitrobenzene-d5 | 100.00 ng/µL |
| 2-Fluorobiphenyl | 100.00 ng/µL |
| p-Terphenyl | 100.00 ng/µL |
| Phenol-d5 | 150.00 ng/µL |
| 2-Fluorophenol | 150.00 ng/µL |
| 2,4,6-Tribromophenol | 150.00 ng/µL |

**A026: 625 SURROGATE**

| | |
|---|---|
| Nitrobenzene-d5 | 50.00 ng/µL |
| 2-Fluorobiphenyl | 50.00 ng/µL |
| p-Terphenyl | 50.00 ng/µL |
| Phenol-d5 | 50.00 ng/µL |
| 2-Fluorophenol | 50.00 ng/µL |
| 2,4,6-Tribromophenol | 50.00 ng/µL |

**A027: 8015B DRO SURROGATE**

| | |
|---|---|
| o-Terphenyl | 20.0 ng/µL |

**A028: CLP 3/90 SVOA SURROGATE**

| | |
|---|---|
| Nitrobenzene-d5 | 50.00 ng/µL |
| 2-Fluorobiphenyl | 50.00 ng/µL |
| p-Terphenyl | 50.00 ng/µL |
| 1,2-Dichlorobenzene | 50.00 ng/µL |
| Phenol-d5 | 75.00 ng/µL |
| 2-Fluorophenol | 75.00 ng/µL |
| 2,4,6-Tribromophenol | 75.00 ng/µL |
| 2-Chlorophenol-d4 | 75.00 ng/µL |

**A033: 8151 HERBICIDE SURROGATE**

| | |
|---|---|
| Dichlorophenyl Acetic Acid | 5.00 ng/µL |

**A035: PCB, PESTICIDE SURROGATE**

| | |
|---|---|
| Tetrachloro-m-xylene | 0.20 ng/µL |
| Decachlorobiphenyl | 0.20 ng/µL |

] - Only required for Screening PCBs. MMG 5/27/05

**A093: CLP PEST/PCB SURROGATE**

| | |
|---|---|
| Tetrachloro-m-xylene | 0.40 ng/µL |
| Decachlorobiphenyl | 0.40 ng/µL |

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 8

**A0148: 8270 LOW LEVEL SURROGATE**
Use 100.0µL of A0026

**A0151: 608, 8082 LOW LEVEL WATER SURROGATE**
Use 100.0 µL of A0035

**A0181 AND A0233: BASE ONLY 8270 SURROGATES**
Use 1000.0 µL of A0001

**A0277: BASE ONLY 8270 LOW LEVEL SURROGATE**
Use 200.0 µL of A0026

**SPIKES**

**A0047: 8151 HERBICIDE SPIKE**

| | |
|---|---|
| 2,4-D | 2.0 ng/µL |
| Dalapon | 2.0 ng/µL |
| Dinoseb | 2.0 ng/µL |
| Pentachlorophenol | 2.0 ng/µL |
| Picloram | 2.0 ng/µL |
| 2,4,5-TP (Silvex) | 2.0 ng/µL |
| 2,4,5-T | 2.0 ng/µL |
| 2,4-DB | 2.0 ng/µL |
| Dicamba | 2.0 ng/µL |
| Dinoseb | 2.0 ng/µL |
| Dichloroprop | 2.0 ng/µL |

**A0049: CLP PEST/PCB SPIKE**

| | |
|---|---|
| gamma-BHC (Lindane) | 0.5 ng/µL |
| Heptachlor | 0.5 ng/µL |
| Aldrin | 0.5 ng/µL |
| Dieldrin | 1.0 ng/µL |
| Endrin | 1.0 ng/µL |
| 4,4'-DDT | 1.0 ng/µL |

**A0051: 8081 PESTICIDE SPIKE**

| | |
|---|---|
| gamma-BHC (Lindane) | 1.0 ng/µL |
| alpha-BHC | 1.0 ng/µL |
| Heptachlor | 1.0 ng/µL |
| Aldrin | 1.0 ng/µL |
| Beta-BHC | 1.0 ng/µL |
| Dieldrin | 1.0 ng/µL |
| Endrin | 1.0 ng/µL |
| 4,4'-DDD | 1.0 ng/µL |

| | |
|---|---|
| 4,4'-DDT | 1.0 ng/µL |
| 4,4'-DDE | 1.0 ng/µL |
| Endosulfan I | 1.0 ng/µL |
| Endosulfan II | 1.0 ng/µL |
| Endrin Aldehyde | 1.0 ng/µL |
| Endosulfan Sulfate | 1.0 ng/µL |
| Heptachlor epoxide | 1.0 ng/µL |
| Methoxychlor | 1.0 ng/µL |
| Endrin Ketone | 1.0 ng/µL |

**A0055: 8270 BN/AP SPIKE**

| | |
|---|---|
| Phenol | 100.0 ng/µL |
| 2-Chlorophenol | 100.0 ng/µL |
| 1,4-Dichlorobenzene | 100.0 ng/µL |
| N-Nitroso-Di-n-propylamine | 100.0 ng/µL |
| 1,2,4-Trichlorobenzene | 100.0 ng/µL |
| 4-Chloro-3-methylphenol | 100.0 ng/µL |
| Acenaphthene | 100.0 ng/µL |
| 4-Nitrophenol | 100.0 ng/µL |
| 2,4-Dinitrotoluene | 100.0 ng/µL |
| Pentachlorophenol | 100.0 ng/µL |
| Pyrene | 100.0 ng/µL |

**A0056: 625 SPIKE**

Use 500.0 µL of A0193

**A0057: CLP 3/90 SVOA SPIKE**

| | |
|---|---|
| Phenol | 75.0 ng/µL |
| 2-Chlorophenol | 75.0 ng/µL |
| 1,4-Dichlorobenzene | 50.0 ng/µL |
| N-Nitroso-Di-n-propylamine | 50.0 ng/µL |
| 1,2,4-Trichlorobenzene | 50.0 ng/µL |
| 4-Chloro-3-methylphenol | 75.0 ng/µL |
| Acenaphthene | 50.0 ng/µL |
| 4-Nitrophenol | 75.0 ng/µL |
| 2,4-Dinitrotoluene | 50.0 ng/µL |
| Pentachlorophenol | 75.0 ng/µL |
| Pyrene | 50.0 ng/µL |

**A0060: CUSTOM CHLOROPYRIDINES SPIKE**

| | |
|---|---|
| 2-Chloropyridine | 100.0 ng/µL |
| 3-Chloropyridine | 100.0 ng/µL |
| 2,6-Dichloropyridine | 100.0 ng/µL |
| p-Fluoroaniline | 100.0 ng/µL |

**TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES**

**SUPERCEDES: Revision 8**

**A0061: CECOS CONSENT 1.6 BN/AP SPIKE**

| | |
|---|---|
| 1,4-Dichlorobenzene | 50.0 ng/µL |
| N,N'-Dimethylacetamide | 50.0 ng/µL |
| Methylaniline N.O.S. | 50.0 ng/µL |
| Pyridine | 50.0 ng/µL |

**A0062: 8270 TCLP SPIKE**

| | |
|---|---|
| 1,4-Dihlorobenzene | 100.0 ng/µL |
| 2,4-Dinitrotoluene | 100.0 ng/µL |
| Hexachlorobenzene | 100.0 ng/µL |
| Hexachlorobutadiene | 100.0 ng/µL |
| Hexachloroethane | 100.0 ng/µL |
| 2-Methylphenol | 100.0 ng/µL |
| 3-Methylphenol | 200.0 ng/µL |
| 4-Methylphenol | 200.0 ng/µL |
| Nitrobenzene | 100.0 ng/µL |
| Pentachlorophenol | 100.0 ng/µL |
| Pyridine | 100.0 ng/µL |
| 2,4,5-Trichlorophenol | 100.0 ng/µL |
| 2,4,6-Trichlorophenol | 100.0 ng/µL |

**A0095: DIESEL FUEL #2 DRO AND PETRO SPIKE**

| | |
|---|---|
| Diesel Fuel #2 | 1500.0 ng/µL |

**A0113: DECHLORANE PLUS**

| | |
|---|---|
| Dechlorane Plus | 1.0 ng/µL |

**A0143: 8082 PCB SPIKE (USE 100.0µL)**

| | |
|---|---|
| Aroclor 1254 | 50.0 ng/µL |

] Use for PCB Screening @ 5ng/µL
MMG 5/27/05

**A0147: 8270 LOW LEVEL SPIKE**

Use 100.0 µL of A0055

**A00152: 608 PESTICIDE AND PESTICIDE/PCB SPIKE**

Use 50.0 µL A0051

**A00153: 608 PCB SPIKE**

Use 100.0 µL of A0213

**A00184: DRO 10 COMPONENT SPIKE**

| | |
|---|---|
| Diesel Range Organics | 200.0 ng/µL |

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 8

A00193: 8270 FULL LIST SPIKE

| | |
|---|---|
| Acenaphthene | 100.0 ng/µL |
| Aniline | 100.0 ng/µL |
| Acenaphthylene | 100.0 ng/µL |
| Anthracene | 100.0 ng/µL |
| Benzo(a)anthracene | 100.0 ng/µL |
| Benzo(b)fluoranthene | 100.0 ng/µL |
| Benzo(k)fluoranthene | 100.0 ng/µL |
| Benzo(ghi)perylene | 100.0 ng/µL |
| Benzo(a)pyrene | 100.0 ng/µL |
| Benzoic Acid | 250.0 ng/µL |
| Benzyl alcohol | 100.0 ng/µL |
| Bis(2-chloroethoxy) methane | 100.0 ng/µL |
| Bis(2-chloroethyl) ether | 100.0 ng/µL |
| 2,2'-Oxybis(1-Chloropropane) | 100.0 ng/µL |
| Bis(2-ethylhexyl)phthalate | 100.0 ng/µL |
| 4-Bromophenyl phenyl ether | 100.0 ng/µL |
| Butyl benzyl phthalate | 100.0 ng/µL |
| 4-Chloroaniline | 100.0 ng/µL |
| 4-Chloro-3-methylphenol | 100.0 ng/µL |
| 2-Chloronaphthalene | 100.0 ng/µL |
| 2-Chlorophenol | 100.0 ng/µL |
| 4-Chlorophenyl phenyl ether | 100.0 ng/µL |
| Chrysene | 100.0 ng/µL |
| Dibenzo(a,h)anthracene | 100.0 ng/µL |
| Dibenzofuran | 100.0 ng/µL |
| Di-n-butyl phthalate | 100.0 ng/µL |
| 1,2-Dichlorobenzene | 100.0 ng/µL |
| 1,3-Dichlorobenzene | 100.0 ng/µL |
| 1,4-Dichlorobenzene | 100.0 ng/µL |
| 3,3'-Dichlorobenzidine | 100.0 ng/µL |
| 2,4'-Dichlorophenol | 100.0 ng/µL |
| Diethyl phthalate | 100.0 ng/µL |
| 2,4-Dimethylphenol | 100.0 ng/µL |
| Dimethyl phthalate | 100.0 ng/µL |
| 4,6-Dinitro-2-methylphenol | 100.0 ng/µL |
| 2,4-Dinitrophenol | 100.0 ng/µL |
| 2,4-Dinitrotoluene | 100.0 ng/µL |
| 2,6-Dinitrotoluene | 100.0 ng/µL |
| Di-n-octyl phthalate | 100.0 ng/µL |
| Fluoranthene | 100.0 ng/µL |
| Fluorene | 100.0 ng/µL |
| Hexachlorobenzene | 100.0 ng/µL |

| | |
|---|---|
| Hexachlorobutadiene | 100.0 ng/µL |
| Hexachlorocyclopentadiene | 100.0 ng/µL |
| Hexachloroethane | 100.0 ng/µL |
| Indeno(1,2,3-cd)pyrene | 100.0 ng/µL |
| Isophorone | 100.0 ng/µL |
| 2-Methylnaphthalene | 100.0 ng/µL |
| 2-Methylphenol | 100.0 ng/µL |
| 4-Methylphenol | 100.0 ng/µL |
| Naphthalene | 100.0 ng/µL |
| 2-Nitroaniline | 100.0 ng/µL |
| 3-Nitroaniline | 100.0 ng/µL |
| 4-Nitroaniline | 100.0 ng/µL |
| Nitrobenzene | 100.0 ng/µL |
| 2-Nitrophenol | 100.0 ng/µL |
| 4-Nitrophenol | 100.0 ng/µL |
| N-Nitrosodiphenylamine | 100.0 ng/µL |
| N-Nitroso-Di-n-prpoylamine | 100.0 ng/µL |
| Pentachlorophenol | 100.0 ng/µL |
| Phenanthrene | 100.0 ng/µL |
| Phenol | 100.0 ng/µL |
| Pyrene | 100.0 ng/µL |
| 1,2,4-Trichlorobenzene | 100.0 ng/µL |
| 2,4,5-Trichlorophenol | 100.0 ng/µL |
| 2,4,6-Trichlorophenol | 100.0 ng/µL |

**A0213: 8082 PCB SPIKE**

| | |
|---|---|
| Aroclor 1242 | 5.0 ng/µL |

**A0222: 8082 PCB SPIKE**

| | |
|---|---|
| Aroclor 1016 | 5.0 ng/µL |
| Aroclor 1260 | 5.0 ng/µL |

] Use Aroclor 1254 for PCB Screening mmk 5/27/05

**A0251: AFCEE 8081 PESTICIDE SPIKE**

| | |
|---|---|
| gamma-BHC (Lindane) | 1.0 ng/µL |
| alpha-BHC | 1.0 ng/µL |
| Heptachlor | 1.0 ng/µL |
| Aldrin | 1.0 ng/µL |
| Beta-BHC | 1.0 ng/µL |
| Dieldrin | 1.0 ng/µL |
| Endrin | 1.0 ng/µL |
| 4,4'-DDD | 1.0 ng/µL |
| 4,4'-DDT | 1.0 ng/µL |
| 4,4'-DDE | 1.0 ng/µL |

| | |
|---|---|
| Endosulfan I | 1.0 ng/µL |
| Endosulfan II | 1.0 ng/µL |
| Endrin Aldehyde | 1.0 ng/µL |
| Endosulfan Sulfate | 1.0 ng/µL |
| Heptachlor epoxide | 1.0 ng/µL |
| Methoxychlor | 1.0 ng/µL |
| Endrin Ketone | 1.0 ng/µL |
| alpha-Chlordane | 1.0 ng/µL |
| gamma-Chlordane | 1.0 ng/µL |

**A0234: 8270 1,4-DIOXANE ONLY SPIKE**

| | |
|---|---|
| 1,4-Dioxane | 100.0 ng/µL |

**A0251: 8082 LOW LEVEL WATER PCB SPIKE (USE 100.0µL)**

| | |
|---|---|
| Aroclor 1254 | 5.0 ng/µL |

**A0281: 8082 LOW LEVEL WATER PCB SPIKE**

Use 100.0 µL of A0222

# STL Buffalo
## LABORATORY STANDARD OPERATING PROCEDURES

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**TITLE: ANALYSIS OF PCBs – METHOD – 8082**

**SUPERCEDES: Revision 3**

| REVIEWED & APPROVED BY: | SIGNATURE | DATE |
|---|---|---|
| Verl D. Preston, Quality Manager | Verl D. Preston | 9/29/03 |
| Chris Oprandi, Laboratory Director | for C.O. | 9/29/03 |
| Gary Rudz, Supervisor | | 9/29/03 |



## 1.0 IDENTIFICATION OF TEST METHOD

1.1 EPA Method 8082A

## 2.0 APPLICABLE MATRIX

2.1 Applicable matrix is soil, water, wipes and waste (oil) samples.

## 3.0 REPORTING LIMIT

3.1 The routine reporting limits are:

0.5ug/L for water samples
16.67ug/Kg for soil samples (100% Dry)
1mg/Kg for oil/waste samples
1 ug/wipe for wipe sample

## 4.0 SCOPE AND APPLICATION

4.1 This method is used to quantify polychlorinated biphenyls (PCBs) in extracts from aqueous, soil, sludge or oil matrices by direct injection techniques into a capillary column equipped gas chromatograph. An electron capture detector (ECD) is employed for identification and quantification. This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and the integration of gas chromatograms.

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| Compound | CAS No. |
|---|---|
| Aroclor 1016 | 12674-11-2 |
| Aroclor 1221 | 11104-28-2 |
| Aroclor 1232 | 11141-16-5 |
| Aroclor 1242 | 53469-21-9 |
| Aroclor 1248 | 12672-29-6 |
| Aroclor 1254 | 11097-69-1 |
| Aroclor 1260 | 11096-82-5 |

## 5.0 SUMMARY OF THE TEST METHOD

5.1 Wastewater samples, approximately 1 liter of sample is extracted with Methylene chloride using a separatory funnel (SOP ASP-3510B-80) or a continuous liquid liquid extractor (SOP No. ASP-3520B-85). Soil samples are extracted using approximately 30g of soil/solid sample using sonication (SOP No. ASP-3550B-90) The extract is then exchanged to hexane and concentrated to 10 ml or less. The final extract is then separated by gas chromatography and detected by an electron capture detector.

Florisil column cleanup procedures and sulfur removal procedures may be utilized to mitigate any interferences that may be encountered during analysis. Although these procedures may eliminate several interferences, contamination of the sample may come from a variety of sources, including solvents, reagents, glassware and any of the hardware used in sample processing. For this reason, reagent and solvent blanks should be analyzed to insure their purity.

## 6.0 DEFINITIONS

6.1 Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual.

## 7.0 INTERFERENCES

7.1 Method interferences can be minimized by proper glassware cleaning methods, instrument maintenance, and the use of high purity reagents and solvents.

7.2 Sulfuric acid (ASP-3665A-98) and copper cleanup Method 3660 are part of the extraction procedure for all PCB samples.

7.3 Gel Permeation Cleanup (ASP-3640A-96) and Florisil Cartridge Cleanup (ASP-3620A-95) may be also used on samples when specified by project or historical results warrant further cleanup.

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**8.0 SAFETY**

8.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document. Safety glasses with side shields are to be worn in all laboratory areas and appropriate Personal Protective Equipment should be worn when working with chemicals and/or samples. Avoid direct contact with chemicals and/or samples since many chemicals can be absorbed through the skin and the samples may contain hazardous constituents. Wash your hands thoroughly with soap and water after contact with any chemical or sample. Always proceed as if a chemical, sample, and/or procedure is hazardous unless you have specific information indicating that it is not.

Sample handling procedures should be performed in an operational fume hood.

The analyst should be wearing; (at a minimum)
- Proper laboratory dress
- Safety Glasses
- Gloves
- Lab Coat

8.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

8.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|---|---|---|---|
| Acetone | Flammable | 1000 ppm-TWA | Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. |
| Hexane | Flammable Irritant | 500 ppm-TWA | Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes. |
| Methylene Chloride | Carcinogen Irritant | 25 ppm-TWA 125 ppm-STEL | Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin. |
| Sulfuric Acid | Corrosive Oxidizer Dehydrator Poison Carcinogen | 1 Mg/M3-TWA | Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

8.4 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory

**8.4.1 Aroclors have been classified as a potential carcinogen. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure.**

8.5 All $^{63}$Ni (ECDs) shall be leak tested every six months.

## 9.0 EQUIPMENT AND SUPPLIES

9.1 Gas chromatograph suitable for on-column injection and all required materials, i.e., syringes, columns, gases, detector and a data processing system capable of measuring peak areas and heights.

9.1.1 Hewlett Packard 5890 gas chromatograph
9.1.2 Hewlett Packard 7673 Auto Sampler
9.1.3 Hewlett Packard 3392 and 3396A Integrators

9.1.4 Capillary columns as described in sections 5.3
9.1.5 Electron Capture Detector
9.1.6 PE Nelson Turbochrome data system
9.1.7 Carrier Gas Hydrogen
9.1.8 Make Up Gas - Argon/Methane
9.1.9 Syringes - various

## 10.0 REAGENTS AND STANDARDS

10.1 Reagents or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of determination.

10.2 Standards are stored in the GC Standard Incubator at 4±2°C in teflon-sealed amber containers in the dark.

10.3 All stock standard solutions are replaced before the expiration date. All other standard dilutions or working standards are discarded after six month (or at the stock standard expiration date, whichever comes first) or sooner if routine QC indicates a problem

10.3.1 Certified PCB Mixes (Aroclors 1016/1260, 1221, 1232, 1242, 1248, 1254)
10.3.2 Second Source PCB Mixes for all Aroclors (Different Manu. or Lot #) to verify consistant response of newly prepared calibration curve.
10.3.3 Acetone (pesticide grade)
10.3.4 Hexane (pesticide grade)

## 11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Aqueous samples are to be collected in a 1-liter amber glass jar and stored at 4±2°C. Organic preparation is to be performed within 7 days of collection.

11.2 Soil (solid) samples are to be collected in a 4 oz jar and stored at 4±2°C. Organic preparation is to be performed within 14 days of collection.

11.3 Analysis of the extracts for both waters and soils is to be performed within 40 days of preparation.

11.4 Holding times specified in project specific quality assurance plans may supersede the above listed method criteria.

11.5 Extracts are stored in Incubator #5 under refrigeration at 4±2°C.

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11.5.1 For CLP, AFCEE, and USACE samples, the extracts are maintained in secure storage incubator SC# 4

## 12.0 QUALITY CONTROL

12.1 A method blank must be prepared and analyzed with each batch (maximum 20 samples). The acceptance criterion is that the method blank must contain a concentration less than the reporting limit for all target analytes. If the blank fails this criterion, the entire prep batch must be re-extracted and analyzed.

12.1.1 For USACE, the method blank must contain concentrations < ½ the reporting limit.

12.2 Analysis of at least one matrix spike and one matrix spike duplicate per batch. Sample spike and duplicate recoveries should fall within the laboratory Quality Control limits that are updated annually based upon historical data. If the recoveries are not achieved, the data is still valid as long as the matrix spike blank is acceptable. The routine matrix spiking solution is an Aroclor 1016/1260 mixture prepared at 5.0ng/ul. During preparation, 1000ul of this solution is added to all quality control samples (MSB/MS/MSD). The resulting expected concentration for both aqueous and soil sample is 0.50ng/ul in the 10ml extract. This translates to final sample concentrations of: 5.0 ug/L for a 1-Liter Aqueous samples, and 166.7ug/Kg for 30.0grams of Soil sample at 100% dry.

12.2.1 Limited sample volume can allow for the analysis of a matrix spike blank duplicate instead of a MS and MSD pair.

12.3 A matrix spike blank must be prepared and analyzed with each batch. Spike recoveries should fall within the laboratory Quality Control limits that are updated annually based upon historical data.

## 13.0 CALIBRATION AND STANDARDIZATION

For PCB Screen for Hudson use Aroclor 1254

13.1 An Initial Calibration Curve (ICC) must be run for the Aroclor mix 1016/1260 and any Aroclor that is specified by a project.

For PCB's a three Point calibration is all that is required.

13.1.1 The curve will consist of a minimum of five concentration points ranging from 0.05ng/uL–2.5ng/uL. The concentration points will be prepared by diluting a certified Aroclor standard. Lower Levels may be prepared at the time of calibration by diluting existing higher levels. Preparation is performed by diluting a level by dilution factor of 10 to get a new level. The following table summarizes the concentration levels used and the associated reporting levels for water and soil samples.



MWG 5/27/05

| Calibration Standard Conc. | Report Level Water (1L) | Report Level Soil (30g/100% dry) |
|---|---|---|
| 0.05 ng/uL | 0.50 ug/L | 16.67 ug/kg |
| 0.10 ng/uL | 1.0 ug/L | 33.34 ug/kg |
| 0.25 ng/uL | 2.5 ug/L | 83.35 ug/kg |
| 0.50 ng/uL | 5.0 ug/L | 166.7 ug/kg |
| 1.0 ng/uL | 10.0 ug/L | 333.4 ug/kg |
| 2.5 ng/uL | 25.0 ug/L | 833.5 ug/kg |

13.1.2 For each Aroclor, select 3–5 unique chromatographic peaks that represent the major biphenyls present in the particular aroclor.

13.1.3 Aroclor 1016/1260 will be defined by a retention time window empirically determined for each analytical column as follows, for each Component:

13.1.3.1 Calculate 3X Standard deviation (in minutes) of 3 injections of Midpoint Standard (0.5ng) over a 72-hour period.

13.1.3.2 Any component with a 3X Standard deviation of <0.05ng minutes defaults to 0.05 minutes or 3.0 seconds at minimum. It should be noted that the primary means of identification are based upon pattern recognition. Matrix effects may shift actual retention times, but the primary pattern may remain intact.

13.1.3.3 Turbochrom calibration files retention time windows should be updated with these values for each component for each Aroclor component chosen.

13.1.4 The Percent Relative Standard Deviation for each individual aroclor must be ≤20%, or have a Correlation Coefficient "R" ≥0.995 (R squared ≥0.990) for the ICC to be acceptable.

13.1.4.1 The curve may be determined using a linear regression fit if a minimum of 5 points are used.

13.1.4.2 If a quadratic regression fit is required to obtain acceptable data, 6 or more calibration points must be employed.

13.1.4.3 If RSD ≤20% for each peak, then linearity of the detector can be assumed for all other Aroclors over the same analytical range.

13.2 A single point calibration must be run for all other Aroclors.

13.2.1 The Aroclor concentration should be near the midpoint concentration of the 1016/1260 curve (~0.5ng/ul on column).

13.2.2 For each Aroclor, five major chromatographic peaks are chosen that represent the key to the patterns present in each particular aroclor. This is important due

to the assessment of degraded patterns when determining identification. For Aroclor 1221, three peaks are used due to the lower number and response of the biphenyls present.

13.3 An Initial Calibration Curve must be run for the surrogate compounds Decachlorobiphenyl and Tetrachloro-meta-xylene, which is to be contained in the Ar1016/1260 calibration standards.

13.3.1 The ICC for the surrogates will require a minimum of five (5) concentration points. The concentrations will be made by serial dilutions of a certified standard. Levels are: 0.005ng, 0.010ng, 0.020ng, 0.030ng, 0.40ng, and 0.5ng.

13.3.2 The Percent Relative Standard Deviation for each surrogate must be ≤20%, or have a Correlation Coefficient "R" ≥0.995 (R squared ≥0.990) for the ICC to be acceptable. If RSD ≤20% for each peak, then linearity of the detector can be assumed. The curve may be determined linear if a minimum of 5 points are used, and quadratic if a minimum of 6 points are used.

## 14.0 PROCEDURE

14.1 Set up the Hewlett Packard Gas Chromatograph as a split injection dual column instrument.

14.1.1 Split injection instruments shall have different columns as to maximize the ability to confirm Aroclors present in the extracts in the most efficient manner.

Acceptable ICC's and single point calibrations are run for all Aroclors and surrogates. Both sides of a split injection instrument must be calibrated with the identical injections.

14.2 The response factor of the ICV must be ±15% D for each aroclor if quantification of that aroclor is to be made. The ICVs and CCVs may >15% biased high to confirm non-detects for any particular aroclor. ICVs and CCVs that are biased >15% low will require a rerun of a compliant standard, or a new calibration curve run.

14.2.1 An ICV will consist of a concentration point at or near the midrange of the curve, (generally 0.5ng on column). The concentration point will be prepared through serial dilutions of a certified Aroclor standard.

14.3 The retention time window determined as in 13.1.3 for each peak must be adjusted using the ICV on a daily basis to account for any shifts in the instrument's operating conditions.

14.3.1 The 1016/1260 mix contains all of the unique components (or congeners) of each individual Aroclor and if the ICV is acceptable for this, it can be understood that the ICV is acceptable for all Aroclors.

14.4 Continuing Calibration Verifications (CCVs) must bracket every 10 samples. Any other Aroclor (with an ICC) may be used along with the midpoint standard of the surrogate as a CCV pair.

14.4.1 The response factor for each peak in the CCV must be ±15% D. All data is acceptable as long as it is bracketed by an acceptable ICV and CCV, or CCV and CCV.

14.5 Each sample is injected into the gas chromatograph and its acceptable data is evaluated for Aroclor patterns and surrogate recovery. Spike recovery is also evaluated in spiked samples.

## 15.0 CALCULATIONS

15.1 Analyzing, confirming and calculating Aroclors.

15.1.1 Retention time windows, pattern recognition and the experience of the analyst will be employed in the identification process. If a chromatographic peak falls outside the established retention time window but an Aroclor pattern is discernible, positive identification shall be made. Turbochrom's compare function can be used to overlay AR 1016/1260 mix against unknown samples. If any Aroclors are present, corresponding congener peaks will be observed. The retention times of the congeners in other Aroclors will match the congener peaks in 1016/1260, but in different response ratios depending on the Aroclor.

15.1.2 The presence of multiple Aroclors will be noted in the summary report.

15.1.3 Single Aroclors shall be confirmed by a clearly recognizable pattern or based on historical data from a specific site.

15.1.4 Calculating the ng amount of an Aroclor in soil and aqueous matrices. This is actually an average of the concentration of the individual peaks.

15.1.4.1 Calculating the ng amount of Aroclor in each peak.

$$\frac{\text{Area of the peak (a)}}{\text{Calibration factor of peak (a)}} = \text{ng amount of peak (a)}$$

or the ng amount from the curve equation

15.1.4.2 Calculating the ng amount of Aroclor in the sample

$$\frac{\text{Peak(n)ng + peak(n+1)ng + peak(n+2)ng +}}{\text{Total Peaks}} = \text{ng amount of sample}$$

15.1.4.3 Converting ng amount to ug/Kg, ug/L and ug/wipe

$$\text{ug/Kg} = \frac{\text{(ng) x (final volume in ml) x (dilution factor)}}{\text{(injection vol. in ul) x (sample wt.) x (\% Dry)}} \times 1000$$

For Oils % dry = 100

$$\text{ug/L} = \frac{\text{(ng) x (final volume in ml) x (dilution factor)}}{\text{(injection volume in ul) x (sample volume in L)}}$$

$$\text{ug/wipe} = \frac{\text{(ng) x (final volume in ml) x (dilution factor)}}{\text{(injection vol. in ul) x (sample wt.) x (\% Dry)}}$$

For wipes, sample weight = 1, % Dry = 100

## 16.0 METHOD PERFORMANCE

16.1 An initial demonstration of capability is performed for either aqueous and soil matrices per analyst and compared to the method criteria.

16.2 MDL studies are performed annually on a matrix and instrument-specific basis.

## 17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 Calib. Check: A calibration check standard must be analyzed after every 10 sample measurements (including quality control). The percent difference between the check standard CF and the calibration standard average CF must be less than 15%. If the acceptance criteria are met, continue. If the acceptance criteria are not met, reanalyze. If reanalysis meets the acceptance criteria, continue. If reanalysis does not meet the acceptance criteria the system must be corrected before samples may be analyzed.

17.2 MBLK: A laboratory method blank must be analyzed with every set of 20 samples at a minimum of 1 per batch. Acceptance criteria are less than the report limit. If the acceptance criteria are met, the QC sample indicates no contamination due to the preparation procedure and is considered acceptable. If analyte is measured above the reporting limit, reanalyze. If reanalysis is acceptable, continue. If reanalysis again indicates contamination the sample results are not useable for drinking water samples. Results for other sample matrices may be used if they are greater than 10 times the blank contamination.

17.2.1 For USACE, the method blank must not contain compounds at levels > ½ the report level.

17.3 MSB: A matrix spike blank (lab fortified blank) must be analyzed with every batch of 20 samples or a minimum of 1 per day. The routine matrix spiking solution is an Aroclor 1016/1260 mixture prepared at 5.0ng/ul. During preparation, 1000ul of this solution is added to all quality control samples (MSB/MS/MSD). The resulting expected concentration for both aqueous and soil sample is 0.50ng/ul in the 10ml extract. This translates to final sample concentrations of: 5.0 ug/L for a 1-Liter Aqueous samples, and 166.7ug/Kg for 30.0grams of Soil sample at 100% dry. Statistical in-house acceptance limits are updated annually and are maintained in the laboratory LIMS system. If the required recovery limits are met, the QC sample indicates control of the preparation procedure and is considered acceptable. If the recovery limits are not met, reanalyze. If reanalysis yields acceptable recovery, continue. If the recovery limits are again not met the batch results are not useable unless the control sample recovery is high and the sample concentrations are below the reportable limit.

17.4 MS: A matrix spike sample must be set for one in every batch of 20 samples. Statistical in-house acceptance limits are updated annually and are maintained in the laboratory LIMS system. If the acceptance criteria are met, no adverse matrix effects are indicated. If acceptance criteria are not met, reanalyze. If reanalysis yields acceptable recovery, continue. If the recovery limits are again not met dilute the MS sample and reanalyze. To minimize bias, samples for matrix spike analysis shall be chosen at random. All analytes in the spike solution shall be measured unless they are not of interest in the spiked sample.

17.5 MSD: Along with every matrix spike sample, a duplicate MS must also be set. This sample is the matrix spike duplicate (MSD). Acceptance criteria are <30% RPD. If the acceptance criteria are met, continue. If the acceptance criteria are not met, reanalyze. If reanalysis yields acceptable recovery, continue. If the recovery limits are again not met report the data yielding the best recovery noting the duplicate failure.

## 18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 Blank contamination and recoveries outside this range may lead to: Re-extraction if within holding time and volume available, noting recoveries in case narrative, or flagging values as estimated. The spike results, sample matrix, and reported positives in the prep batch are also to be considered. The Project Manager will be notified with a job exception, and acceptability may be determined by citing historical, sample, and method results on a case by case basis

## 19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 When QC results, unknown positives, or sample matrix present the analyst with questionable data, the spike results, sample matrix, and reported positives in the prep batch are all to be considered. Acceptability may be determined by citing historical, sample, and method results on a case by case basis. The project manager shall be notified of any method anomalies, and can then contact the client as to specific instructions on the usability of the data and any further actions

## 20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

20.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic waste generated in the lab.
- Solvent waste generated by the extraction
- Expired primary and working PCB standards.
- Vials containing sample extracts.

20.3 All solvent waste generated by the extraction is to be disposed of in a labeled "C" waste container.

20.4 All acidified aqueous waste is to be disposed of into a labeled "A" waste container

## 21.0 REFERENCES

21.1 Method 8082 U.S. Environmental Protection Agency, Office of Solid Waste and Energy Response, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," 3rd edition, SW-486, update III, Dec. 1996.

## 22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Job Summary Sheet and Data Review Checklist

## 23.0 CHANGES FROM PREVIOUS REVISION

23.1 Laboratory Director change – signature updated.

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23.2 Section 8.0: Added specific safety information developed by STL Corporate EH&S
23.3 Section 11.2: Included soil preservation and holding time information.
23.4 Section 11.5.1: Included special storage requirements for CLP, AFCEE and USACE
23.5 Section 12.1.1: Added USACE method blank acceptance criteria
23.6 Section 12.2: Included spike solution preparation and correlated spike concentrations to final concentrations in samples
23.7 Section 13.1.1: Developed table to correlate calibration concentrations to final concentrations in samples
23.8 Section 13.1.4: Expanded information regarding regression fit used for calibration.
23.9 Section 14.3: Made reference to section 13.1.3 for development of the retention time window.
23.10 Section 17.2.1: Added USACE method blank acceptance criteria
23.11 Section 17.4: Removed reference to default MS acceptance limits (70-130%) and replaced with requirement for development of statistical in-house acceptance limits.
23.12 Section 17.5: Removed reference to default MSB acceptance limits (70-130%) and replaced with requirement for development of statistical in-house acceptance limits.
23.13 Section 20.0: Added specific waste management information developed by STL Corporate EH&S.
23.14 Section 22.1: Added Job Summary Sheet and Stat Review Checklist as attachment

# STL Buffalo
## LABORATORY STANDARD OPERATING PROCEDURES

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**22.1 Job Summary Sheet and Data Review Checklist**

Method: _____ Client: _____ **Job # A03 -**

Level: 1 2 3 4 No. Samples _____ Tat DUE /

Inst.: Ch. A / B *Matrix: W / S / Oil / Wipe* **RUSH ☐**

CCV Criteria - Date: Comments:

Ch: A / B / /2003

Ch: A / B / /2003

Ch: A / B / /2003

| | | |
|---|---|---|
| CU TREATED: | Y N | DL CODE |
| ACID TREAT | Y N | 002 / 008 |
| DILUTIONS: | Y N | Matr. / Pos. |

**BATCH REVIEW:**

Blanks Clean? Y / N Surr. In Limits Y / N

MSB Rec. In Limits? Y / N

MSBD Rec. In Limits? Y / N RPD Y / N

**SURROGATE Review** List Samples: =>

TCX Out / DCB In _____ DCB Out / TCX In _____ BOTH Out _____ Diluted Out _____

MS Rec. In Limits? Y / N / DL NA

MSD Rec. In Limits? Y / N / DL RPD Y / N / DL

Comments: _____

☐ No Positives Above RL

☐ Heavy Matrix Effects

| PROCESSING | DATE | ANALYST(S) |
|---|---|---|
| COMPARED | | / |
| COMMENTS ENTERED | | |
| REPORTING CHANNEL | A / B | |
| TX0'D & MOVED | | |
| AIMS ENTERED: | | |
| WORKSHEETS PRINTED: | | |
| REVIEWED & CLOSED: | | |
| RUNLOG COPIED (L4) | | |
| CLIENT FORMS PRINTED (L4) | | |
| STANDARD SUMMARY (L4) | | |

Manual Integrations Needed for Samples? ( Y / N )

| | |
|---|---|
| BA | Baseline Adjustment |
| NP | Negative Peaks |
| CE | Co-Elution |
| PS | Peak Splitting |
| ME | Matrix Effects |
| OT | Other |

Review of Manual Integrations Y / NA

Initial & Date _____ ____/____