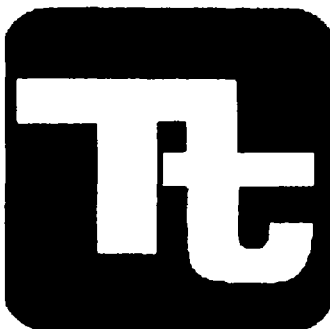




**SUPERFUND TECHNICAL ASSESSMENT AND RESPONSE TEAM (START)  
CONTRACT FOR EMERGENCY RESPONSE ACTIVITIES  
IN REGION 5**

**FINAL  
FIELD SAMPLING AND ANALYSIS PLAN FOR  
ILLINOIS CENTRAL SPRING EMERGENCY RESPONSE SITE  
MONROE COUNTY, INDIANA**

**Prepared for  
U.S. Environmental Protection Agency  
Region 5  
Chicago, Illinois**



Technical Direction Document No. : S05-0105-013  
Contract No. : 68-W-00-129  
Date Prepared : 03 Apr 02  
Prepared by : Tetra Tech EM Inc.  
Tetra Tech Project Manager : Jeffrey Lifka  
Telephone No. : (312) 946-6491  
EPA On-Scene Coordinator : Kenneth Theisen  
Telephone No. : (312) 886-1959

**Tetra Tech EM Inc.**



**Tetra Tech EM Inc.**

200 E. Randolph Drive, Suite 4700 ♦ Chicago, IL 60601 ♦ (312) 856-8700 ♦ FAX (312) 938-0118

03 Apr 02

Mr. Kenneth Theisen  
On-Scene Coordinator  
Emergency Response Branch (SE-5J)  
U.S. Environmental Protection Agency Region 5  
77 West Jackson Boulevard  
Chicago, IL 60604-3507

**Subject: Final Field Sampling and Analysis Plan for  
Illinois Central Spring  
Emergency Response Site  
Bloomington, Monroe County, Indiana  
Technical Direction Document No. S05-0105-013  
Tetra Tech Contract No. 68-W-00-129**

Dear Mr. Theisen:

At your request, the Tetra Tech EM Inc. (Tetra Tech) Superfund Technical Assessment and Response Team (START) is submitting its Final Field Sampling and Analysis Plan to the U.S. Environmental Protection Agency (EPA). The Field Sampling and Analysis Plan discusses all relevant sampling and analysis activities for the two-week sampling event occurring from 08 Apr 02 through 19 Apr 02 at the above-referenced site in Monroe County, Indiana. Comments received from EPA and Tetra Tech's subcontracted laboratory for the project, Air Toxics Ltd., of Folsom, California, on the draft sampling and analysis plan have been incorporated into the final version. The approval page of the Final Field Sampling and Analysis Plan will be signed.

If you have any questions about this submittal, please call me at (312) 946-6491.

Sincerely,

A handwritten signature in black ink, appearing to read 'Jeffrey Lifka', written in a cursive style.

Jeffrey Lifka  
Tetra Tech START Project Manager

Enclosure

cc: Lorraine Kosik, U.S. EPA START Project Officer (letter only)  
Thomas Kouris, Tetra Tech START Program Manager (letter only)  
Dede Dodge, Air Toxics Ltd. Project Manager

**APPROVAL**

**FINAL  
FIELD SAMPLING AND ANALYSIS PLAN FOR  
ILLINOIS CENTRAL SPRING EMERGENCY RESPONSE SITE  
MONROE COUNTY, INDIANA**

**REVISION 1**

**03 APR 02**


**Prepared by Tetra Tech EM Inc.**

  
\_\_\_\_\_  
Tetra Tech EM Inc. Project Manager


4/8/02  
Date

  
\_\_\_\_\_  
Tetra Tech EM Inc. Quality Assurance Officer

4/9/02  
Date

  
\_\_\_\_\_  
U.S. Environmental Protection Agency Region 5  
On-Scene Coordinator

4-16-02  
Date

  
\_\_\_\_\_  
Air Toxics Ltd. Project Manager

4/8/02  
Date

  
\_\_\_\_\_  
Air Toxics Ltd. Quality Assurance Manager

4/8/02  
Date

**APPROVAL**  
**FINAL**  
**FIELD SAMPLING AND ANALYSIS PLAN FOR**  
**ILLINOIS CENTRAL SPRING EMERGENCY RESPONSE SITE**  
**MONROE COUNTY, INDIANA**

**REVISION 1**

**03 APR 02**

**Prepared by Tetra Tech EM Inc.**

\_\_\_\_\_  
Tetra Tech EM Inc. Project Manager

\_\_\_\_\_  
Date

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Tetra Tech EM Inc. Quality Assurance Officer

\_\_\_\_\_  
Date

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U.S. Environmental Protection Agency Region 5  
On-Scene Coordinator

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Date

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Air Toxics Ltd. Project Manager

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Date

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Air Toxics Ltd. Quality Assurance Manager

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Date

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TETRA TECH EM INC. FIELD STANDARD OPERATING PROCEDURES  
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## LIST OF ACRONYMS, ABBREVIATIONS, AND SYMBOLS

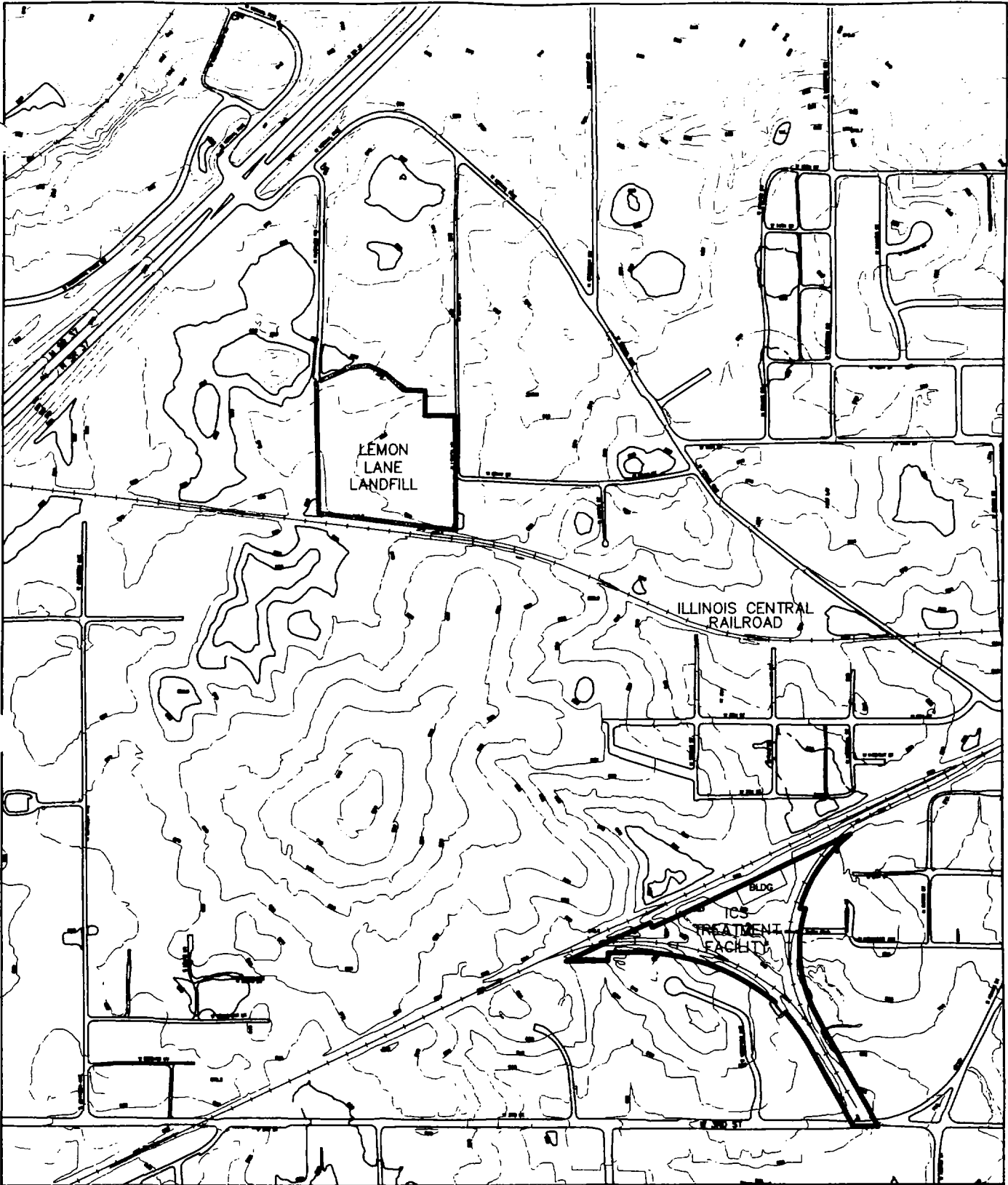
°C	Degrees Celsius
%R	Percent recovery
Air Toxics	Air Toxics Ltd.
CBS	Columbia Broadcasting System
Earth Tech	Earth Tech, Inc.
EPA	U.S. Environmental Protection Agency
LCS	Laboratory control spike
LCSD	Laboratory control spike duplicate
MS	Matrix spike
MSD	Matrix spike duplicate
OSC	On-Scene Coordinator
PCB	Polychlorinated biphenyl
ppm	Parts per million
PRQL	Project-required quantitation limit
QA	Quality assurance
QC	Quality control
RPD	Relative percent difference
SAP	Sampling and analysis plan
SOP	Standard operating procedure
START	Superfund Technical Assessment and Response Team
Tetra Tech	Tetra Tech EM Inc.
TDD	Technical Direction Document
Viacom	Viacom, Inc.
Westinghouse	Westinghouse Electric Corporation

## SECTION 1 PROJECT DESCRIPTION

This field sampling and analysis plan (SAP) has been prepared for the Illinois Central Spring (ICS) emergency response site located in Bloomington, Indiana, at the request of the U.S. Environmental Protection Agency (EPA) for Technical Direction Document (TDD) No. S05-0105-013 under the Superfund Technical Assessment and Response Team (START) Contract No. 68-W-00-129 for Region 5. Tetra Tech EM Inc. (Tetra Tech) has prepared this field SAP to describe sample collection and analysis activities to be conducted by Tetra Tech and its subcontracted laboratory, Air Toxics, Ltd. (Air Toxics), of Folsom, California. In 1985, EPA, the State of Indiana, Monroe County, the City of Bloomington, and Westinghouse Electric Corporation (Westinghouse [later known as Columbia Broadcasting System (CBS) and currently known as Viacom, Inc. (Viacom)]) signed a Consent Decree. Under the terms of the Consent Decree, Viacom is required to remediate six sites in the Bloomington, Indiana, area containing polychlorinated biphenyls (PCB) following a detailed program. The six sites are Neal's Landfill, Neal's Dump, Lemon Lane Landfill, Bennett's Dump, Winston Thomas Wastewater Treatment Facility, and Anderson Road Landfill. The ICS site is located about 0.5 mile southeast of one of the above-referenced sites, Lemon Lane Landfill, which is the primary source of groundwater discharge flowing from the spring (see Figure 1-1). A spring treatment facility was constructed by EPA near ICS and began operation in May 2000, capturing the spring water for treatment of PCBs before being discharged back to the ICS channel downstream of the spring treatment facility.

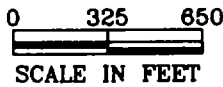
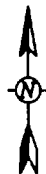
As requested by the EPA On-Scene Coordinator (OSC), Mr. Kenneth Theisen, this field SAP has been prepared for sample collection and analysis at the ICS emergency response site. The data obtained from this activity will be used to assist EPA in conducting an assessment of possible PCB air emissions from the spring water at the site and determining if there is a health risk to the ICS treatment facility operators and nearby residents of the treatment facility. The field SAP was prepared in accordance with EPA requirements outlined in (1) "EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations" (EPA 1994b) and (2) "Region 5 Superfund Model Mini Quality Assurance Project Plan" (EPA 1997).

C:\G\9000\75013-Illinois Central Spring\ Fig 1-1 Site Location.dwg 03/25/2002 mark.kennedy CH



**LEGEND**

- AREAS OF INTEREST
- RAILROAD
- ROAD
- CONTOUR LINES



ILLINOIS CENTRAL SPRING TREATMENT FACILITY  
AND LEMON LANE LANDFILL SITE  
BLOOMINGTON, INDIANA

**FIGURE 1-1**  
SITE LOCATION MAP

 Tetra Tech EM Inc.

SOURCE: MODIFIED FROM EARTH TECH MAP

This field SAP consists of the following 14 sections:

- Section 1, Project Description
- Section 2, Project Organization and Responsibilities
- Section 3, Quality Assurance (QA) Objectives for Analytical Data
- Section 4, Sampling Procedures
- Section 5, Custody Procedures
- Section 6, Calibration Procedures
- Section 7, Analytical Procedures
- Section 8, Data Reduction, Validation, and Reporting
- Section 9, Internal Quality Control (QC) Checks
- Section 10, Performance and System Audits
- Section 11, Preventive Maintenance Procedures
- Section 12, Data Assessment Procedures
- Section 13, Corrective Actions; and
- Section 14, QA Reports to Management.

References used to prepare the field SAP are provided at the end of the field SAP. The site background for the Lemon Lane Landfill (Tetra Tech 2000) and its associated ICS emergency response site, a summary of sampling activities, and the proposed project schedule are described below.

## **1.1 SITE HISTORY AND BACKGROUND**

The Lemon Lane Landfill is a 10-acre site that accepted both municipal and industrial wastes from 1933 to 1966. Between 1958 and 1966, Westinghouse, now Viacom, deposited large quantities of electrical capacitors filled with oil containing PCBs in the landfill. The landfill is situated on karst terrain, and PCB contamination has migrated off site to nearby springs and to Clear Creek, causing downstream contamination of sediment and aquatic life. ICS, which is located approximately 0.5 mile southeast of the landfill, is the source of the PCBs in Clear Creek. The source control remedial action for ICS and Lemon Lane Landfill was completed in May and December 2000, respectively.

Since the early 1980s, numerous geophysical, hydrogeologic, and contaminant characterization investigations have been conducted by Viacom, EPA, and the State of Indiana. Viacom and EPA conducted an investigation in 1996 to delineate PCB hot spots in the landfill. Based on the results of that investigation, the selected remedial action for source control at the site was completed and consisted of (1) PCB hot spot soil excavation, consolidation, and off-site disposal; (2) a Resource Conservation and Recovery Act (RCRA) Subtitle C-compliant cap; (3) drainage controls; (4) institutional controls; (5) long-term monitoring; and (6) supplemental investigations and studies. EPA constructed the ICS spring treatment facility to capture spring water downstream of Lemon Lane Landfill for treatment of PCBs before discharging the treated water back to the ICS channel downstream of the treatment facility.

## **1.2 SUMMARY OF SAMPLING AND ANALYSIS ACTIVITIES**

As part of its TDD for the ICS site, Tetra Tech, in coordination with the EPA OSC and Earth Tech, Inc. (Earth Tech—the treatment plant operations contractor), will obtain high-volume perimeter air samples and low-volume personnel monitoring air samples for a two-week period at the ICS site. Up to 60 perimeter and 14 personnel air samples will be obtained from the site.

Tetra Tech's Field Sampling Leader in consultation with the EPA OSC selected the perimeter air sampling locations at the site. The locations include one air sampler unit being placed upwind, one unit downwind, and two (one unit is for collection of duplicate samples) units near the fenceline adjacent to the condominiums northeast of the ICS site. The perimeter air samples will be collected each day over a 24-hour period during the two-week sampling event. The high-volume sampling units that will be used for perimeter air sampling are the General Motor Works GPS-1 polyurethane foam (PUF) samplers. These units will sample over 200 cubic meters of air during the 24-hour period.

Earth Tech personnel monitoring will consist of the treatment plant operator wearing a low-flow portable pump with an air hose and PUF cartridge attached near the operator's breathing zone. These samples will be collected over the course of 8 hours and are intended to represent the normal work day conditions of the treatment plant operator.

Sample collection and analysis is being performed by Tetra Tech for EPA to assess whether there is a human health risk associated with PCB air emissions exposure to the treatment plant operators and nearby residential areas.

All PUF samples will be packaged each day after removal from the PUF sampling units and personnel pumps and shipped for overnight delivery to Air Toxics in Folsom, California. The PUF cartridges will be wrapped in aluminum foil, sealed in plastic bags, and placed in coolers with blue ice packets.

The high-volume PUF samples collected from the ICS site will be analyzed for PCBs in accordance with EPA Method TO-4A. The low-volume PUF samples collected during personnel monitoring will be analyzed for PCBs in accordance with EPA Method TO-10A. Air Toxics will also analyze associated QC samples in accordance with each method. Table 1-1 presents a sampling and analysis summary.

Detection limits of the analytical methods selected will be less than or equal to the detection of the analytical methods selected by EPA and the project-required quantitation limits (PRQL). The PRQL for PCBs in air is 1 microgram. The project-specific objectives for accuracy, precision, and completeness will be achieved in accordance with the appropriate organics standard operating procedures (SOP).

**TABLE 1-1  
SAMPLING AND ANALYSIS SUMMARY**

<b>Matrix</b>	<b>Location</b>	<b>No. of Samples</b>	<b>No. of LCS/LCSD Pairs<sup>a</sup></b>	<b>No. of Field Duplicates</b>	<b>Total No. of Samples</b>	<b>Analytical Parameter</b>	<b>Analytical Methods</b>
Air	Perimeter	60	3	12	75	PCBs	EPA Method TO-4A; High Volume
	Personnel monitor	14	1	NA	15	PCBs	EPA Method TO-10A; Low Volume

Notes:

LCS/LCSD = Laboratory control spike/laboratory control spike duplicate

<sup>a</sup> Tetra Tech will have Air Toxics analyze LCS/LCSD samples for QC analysis. LCS/LCSD samples are typically analyzed at a frequency of one for each group of 20 or fewer investigative samples.

NA Not applicable; treatment plant operator cannot wear two personnel air sampling pumps simultaneously over an 8-hour period.

### **1.3 PROJECT SCHEDULE**

Air sampling at the ICS site is scheduled to begin on April 8, 2002, and is to be completed on April 19, 2002. It is anticipated that 6 to 8 weeks will be required for sample analysis, data validation, data evaluation, and preparation of a final data evaluation report.

## SECTION 2

### PROJECT ORGANIZATION AND RESPONSIBILITIES

Under the direction of the EPA OSC, Tetra Tech has overall responsibility for sampling and analysis necessary to assist EPA in conducting an assessment of possible PCB air emissions from the ICS site and determining if any human health risk exists to the treatment plant operators and nearby residents of the ICS site. Tetra Tech will also provide all project management. The project organization and responsibilities of various key management, QA, field, and laboratory personnel are discussed below. Support personnel roles are not discussed; however, all personnel will be required to adhere to all provisions of this field SAP.

#### 2.1 PROJECT ORGANIZATION

The project organization is illustrated in Figure 2-1. Personnel responsibilities are discussed below.

#### 2.2 MANAGEMENT RESPONSIBILITIES

The responsibilities of the EPA OSC, Tetra Tech program manager, and Tetra Tech project manager are discussed below.

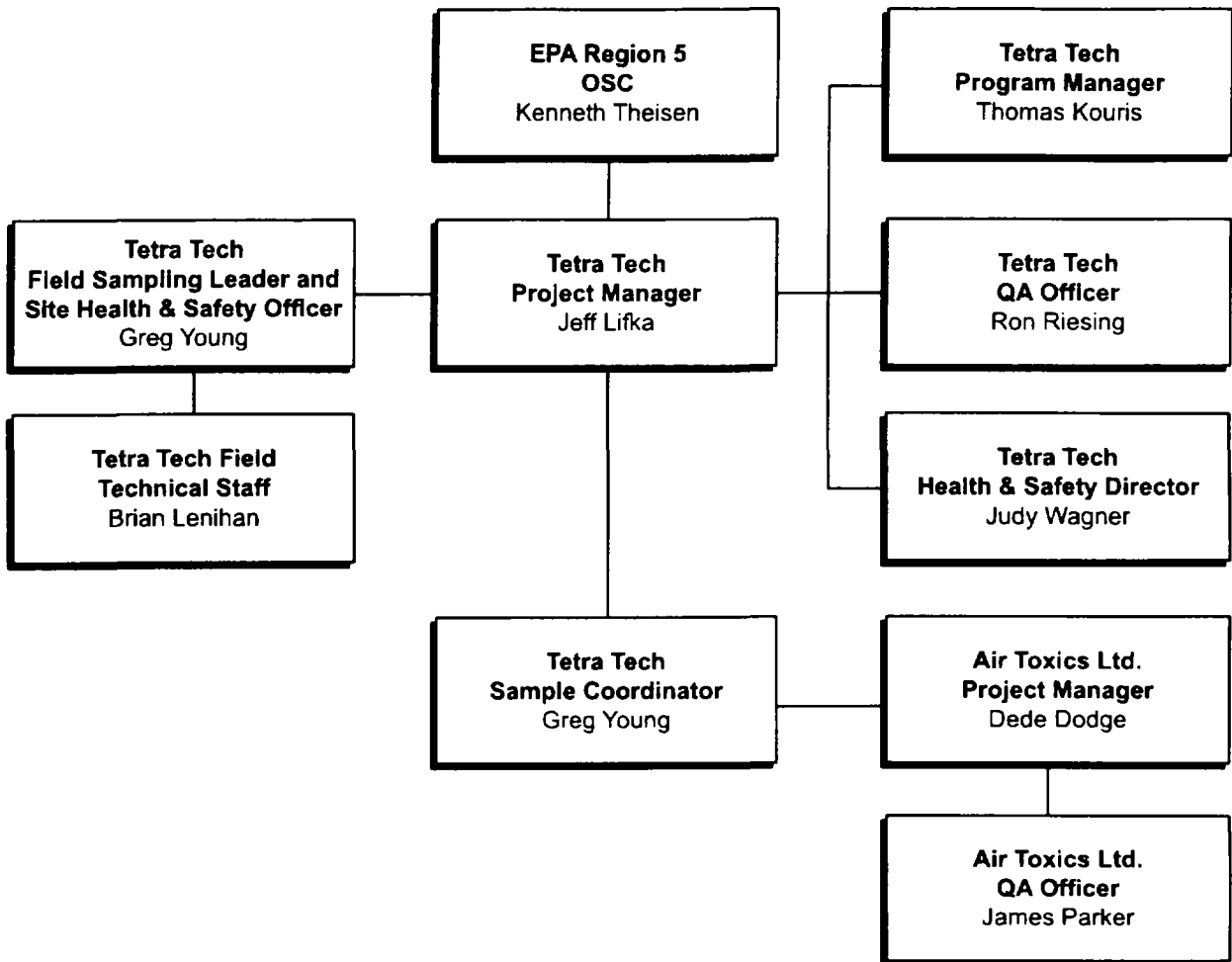
##### **EPA On-Scene Coordinator**

The EPA OSC, Kenneth Theisen, has the overall responsibility for all phases of the sampling and analysis activities.

##### **Tetra Tech Program Manager**

The Tetra Tech program manager, Thomas Kouris, has overall responsibility for ensuring that the project meets EPA objectives and Tetra Tech quality standards. In addition, the program manager is responsible

**FIGURE 2-1**  
**PROJECT ORGANIZATION CHART**



for technical QC and project oversight and will provide the Tetra Tech project manager with access to corporate management.

### **Tetra Tech Project Manager**

The Tetra Tech project manager, Jeffrey Lifka, is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The project manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The project manager will report directly to the EPA OSC and will provide the major point of contact and control for matters concerning the project. The project manager's duties and responsibilities are as follows:

- Define project objectives and develop a detailed work plan schedule
- Establish project policies and procedures to address specific needs of the project as a whole and objectives of each project task
- Acquire and apply technical and corporate resources as needed to ensure that project performance is within budget and schedule constraints
- Orient field sampling leader and technical staff concerning the project's special considerations
- Monitor and direct the field sampling leader
- Develop and meet ongoing project and task staffing requirements, including mechanisms to review and evaluate each task product
- Review the work performed under each task to ensure its quality, responsiveness, and timeliness
- Review and analyze overall task performance with respect to planned requirements and authorizations
- Approve all external reports (deliverables) before their submission to EPA Region 5
- Ultimately be responsible for the preparation and quality of interim and final reports
- Represent the project team at meetings

## **2.3 QUALITY ASSURANCE RESPONSIBILITIES**

The responsibilities of the Tetra Tech QA reviewer are discussed below.

### **Tetra Tech Quality Assurance Officer**

The Tetra Tech QA officer, Ron Riesing, will remain independent of direct project involvement and day-to-day operations and will have direct access to corporate executive personnel as necessary to resolve any dispute. This individual is responsible for auditing the implementation of the QA program in conformance with Tetra Tech's Region 5 START requirements, Tetra Tech policies, and EPA requirements. The Tetra Tech QA officer's specific functions and duties are as follows:

- Conduct audits of various phases of field operations
- Review and approve external reports (deliverables), including field SAPs, before their submission to EPA Region 5
- Provide QA technical assistance to project personnel
- Report on the adequacy, status, and effectiveness of the QA program on a regular basis to the Tetra Tech program manager and project manager

## **2.4 FIELD RESPONSIBILITIES**

The responsibilities of the Tetra Tech field sampling leader, health and safety director, site health and safety officer, and field technical staff are described below.

### **Tetra Tech Field Sampling Leader**

The Tetra Tech project manager will be supported by the Tetra Tech field sampling leader, Greg Young. The field sampling leader is responsible for leading and coordinating day-to-day field activities and reporting directly to the Tetra Tech project manager. Specific field sampling leader responsibilities are as follows:

- Provide day-to-day coordination with the project manager on technical issues in specific areas of expertise
- Develop and implement the field SAP and ensure project schedule compliance
- Ensure that all Tetra Tech field personnel follow the site health and safety plans
- Coordinate and manage field personnel
- Implement QC procedures for technical data provided by field personnel, including field measurement data
- Identify problems at the field technical staff level and discuss resolutions with the project manager, implement and document corrective action procedures, and provide communication between the technical field staff and upper management

#### **Tetra Tech Health and Safety Director**

The Tetra Tech health and safety director, Judy Wagner, will interact directly with the Tetra Tech project manager and will be responsible for providing technical coordination for the health and safety program.

Additional health and safety director responsibilities are as follows:

- Act in an advisory capacity to the site health and safety officer
- Act as a liaison with officers and representatives of EPA on matters relating to health and safety
- Maintain up-to-date records of health and safety plan-related documentation and health and safety program participants

#### **Tetra Tech Site Health and Safety Officer**

The Tetra Tech site health and safety officer, Greg Young, will report directly to the Tetra Tech project manager and will be responsible for the field implementation and enforcement of the health and safety plans. Additional site health and safety officer responsibilities are as follows:

- Ensure that the “Approval and Sign-Off Form” is signed by all personnel who are to perform field work
- Ensure that field personnel complete a “Daily Site Log” before leaving the site

### **Tetra Tech Field Technical Staff**

The Tetra Tech field sampling leader will be supported by a Tetra Tech field technical staff member, Brian Lenihan. The field technical staff member will have the following responsibilities:

- Conduct specific sampling tasks outlined in the field SAP
- Adhere to field SAP requirements
- Analyze data and prepare various task reports and support materials

## **2.5 LABORATORY RESPONSIBILITIES**

Analyses and data validation of the samples requires coordination between Tetra Tech, EPA, and Tetra Tech’s subcontracted laboratory. Specifically, Tetra Tech will schedule analyses with Air Toxics. Tetra Tech will send the samples to the laboratory for analysis. Sample data will then be sent to Tetra Tech for data validation. Validated data will be reviewed and then signed by the EPA OSC. The signed review will then be sent to Tetra Tech for use in preparing a sample evaluation report. The roles and responsibilities of the Tetra Tech sample coordinator, laboratory project manager, laboratory QA officer, laboratory sample custodian, and laboratory technical personnel are described below.

### **Tetra Tech Sample Coordinator**

The Tetra Tech sample coordinator, Greg Young, will report directly to the Tetra Tech project manager and will be responsible for coordinating sample processing with Earth Tech’s treatment plant operator, Martin Lytle, and laboratory analysis of field samples with Air Toxics. The sample coordinator will have the following additional responsibilities:

- Ensure that chain-of-custody procedures are appropriately followed
- Coordinate receipt and management of laboratory analytical results

### **Laboratory Project Manager**

The laboratory project manager, Ms. Dede Dodge, will report directly to Tetra Tech and will have the following responsibilities:

- Ensure that all laboratory resources are available on an as-required basis
- Coordinate and schedule laboratory sample analyses
- Supervise in-house chain-of-custody
- Oversee data review
- Oversee preparation of analytical reports
- Review final analytical reports
- Approve final analytical reports prior to submission to Tetra Tech

### **Laboratory Quality Assurance Officer**

The laboratory QA officer, Mr. James Parker, has the overall responsibility for data after the data leave the laboratory. The laboratory QA officer will be independent of laboratory technical personnel involved with sample analysis but will communicate data issues to them through the laboratory project manager.

In addition, the laboratory QA officer will have the following responsibilities:

- Oversee laboratory QA
- Oversee QA/QC documentation
- Conduct detailed data review
- Determine whether to implement laboratory corrective actions, if required

- Define appropriate laboratory QA procedures
- Prepare laboratory SOPs

### **Laboratory Sample Custodian**

The laboratory sample custodian for the laboratory will report to the laboratory project manager. The laboratory sample custodian will have the following responsibilities:

- Receive and inspect incoming sample containers
- Record the condition of incoming sample containers
- Sign appropriate documents
- Verify that chain-of-custody records are complete and correct
- Notify the laboratory project manager of sample receipt and inspection
- Assign each sample a unique identification number and customer number, and enter each number into the sample receiving log
- Initiate transfer of samples to appropriate laboratory sections with the help of the laboratory project manager
- Control and monitor access and storage of samples and extracts

### **Laboratory Technical Personnel**

Laboratory technical personnel from the laboratory will be responsible for sample analysis and identification of corrective actions in accordance with this field SAP. These personnel will report directly to the laboratory project manager.

## SECTION 3

### QUALITY ASSURANCE OBJECTIVES FOR ANALYTICAL DATA

The overall QA objectives for analytical data are to develop and implement procedures for field sampling, sample chain-of-custody, laboratory analysis, and reporting that will provide valid and complete data required for the field investigation to assist EPA in conducting a PCB air emissions risk assessment. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, internal QC audits, data reporting, preventive maintenance for field equipment, data assessment, and corrective action are described in subsequent sections of this field SAP. This section discusses specific QA objectives for precision, accuracy, completeness, representativeness, comparability, level of QC effort, and PRQLs.

#### 3.1           PRECISION

Precision is a measure of the degree to which two or more field or laboratory measurements are in agreement. Field sampling precision will be assessed by collecting field duplicate samples. Precision for laboratory sample analysis will be assessed by having Air Toxics analyze laboratory control spike and laboratory control spike duplicate (LCS/LCSD) samples. Matrix spike and matrix spike duplicate (MS/MSD) samples are not typically collected for EPA Methods TO-4A and TO-10A; therefore, Air Toxics will analyze LCS/LCSD samples. LCS/LCSD samples will be analyzed at a frequency of one per group of 20 or fewer investigative samples for each sample parameter. Because field duplicate and LCS/LCSD samples will be analyzed as separate sample volumes in separate containers, these sample results can be used to evaluate the precision of the analytical procedures. The number of LCS/LCSD samples to be analyzed during this field investigation is presented in Table 1-1. Precision will be assessed by determining the relative percent difference (RPD) of LCS and LCSD sample results (see Section 12) in accordance with Air Toxics SOP No. 26 for PCB analysis (see Attachment 2).

### **3.2 ACCURACY**

Accuracy is the degree of agreement between an observed value and an accepted reference value. Field accuracy will be assessed through adherence to all sample handling, preservation, and holding times specified in Section 4. Accuracy of laboratory sample analysis will be assessed by determining the percent recovery (%R) of LCS and LCSD samples (see Section 12) in accordance with Air Toxics SOP No. 26 for PCB analysis (see Attachment 2).

### **3.3 COMPLETENESS**

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under normal conditions. It is expected the laboratories will provide 95 percent or more data that meet the QC acceptance criteria. If 95 percent of the laboratory data meet these criteria, the data will be considered complete. If laboratory data is found to be incomplete, Tetra Tech will inform the EPA OSC. If directed by the EPA OSC, Tetra Tech will perform additional sampling necessary to meet the QC acceptance criteria for completeness.

### **3.4 REPRESENTATIVENESS**

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter dependent on the proper design of the sampling program and proper laboratory protocols. The oversight sample network for the field investigation was designed to provide data representative of site conditions. During development of the sample network, past waste disposal practices, existing analytical data, the physical site setting, and site processes were considered. Representativeness of field data will be achieved by ensuring that the sampling program is followed and that proper sampling techniques are used. Representativeness in the laboratory will be achieved by ensuring that proper laboratory analytical procedures are used and sample holding times are not exceeded.

### **3.5 COMPARABILITY**

Comparability expresses the confidence with which one data set can be compared to another. Comparability is also dependent on similar QA objectives. Comparability of field data will be achieved by ensuring that the sampling program is followed and that proper sampling techniques are used. Comparability of laboratory data will be achieved by ensuring that proper analytical procedures are followed.

### **3.6 LEVEL OF QUALITY CONTROL EFFORT**

As described in Sections 3.1 and 3.2, LCS/LCSD samples will be analyzed to assess data quality. Duplicate samples will also be collected and used to assess field and analytical precision. In addition, Tetra Tech will not collect field blank or trip blank samples as part of this investigation. The specific procedures to be used for analyzing LCS/LCSD samples are described below.

LCS/LCSD samples are typically analyzed at a frequency of one per group of 20 or fewer investigative samples for each sample matrix. For each matrix and parameter analyzed, one additional sample container will be analyzed for LCS analysis and one additional sample container will be analyzed for LCSD analysis. LCS/LCSD samples will be analyzed using the same procedures used to analyze the field samples, except that they will be clearly designated for LCS/LCSD analysis.

### **3.7 PROJECT-REQUIRED QUANTITATION LIMIT**

The PRQL for PCBs in air is 1 microgram. The PRQL for PCBs in air is specified in Air Toxics SOP No. 26 for PCB analysis (see Attachment 2).

## SECTION 4 SAMPLING PROCEDURES

This section describes the sample collection, sample handling, and sample packaging and shipping procedures that will be used during the air sampling activities. Sampling will be used to assist EPA in conducting a PCB air emissions risk assessment.

### 4.1 SAMPLE COLLECTION PROCEDURES

Tetra Tech will collect up to 60 high-volume perimeter air samples using PUF samplers and up to 10 low-volume personnel monitoring air samples from the ICS site using a portable pump and PUF cartridges. Table 1-1 summarizes the number of samples to be collected and the number of QC samples to be submitted for analysis. Sampling locations have been selected by the Tetra Tech project manager based on discussions with the EPA OSC. Tetra Tech will collect the perimeter and personnel monitoring samples in accordance with the procedures described below.

**Perimeter.** For high-volume samples for PCB analysis, Tetra Tech will calibrate the PUF samplers and load the samplers with the TO-4A PUF cartridges during each day of the sampling period. The PUF samplers will operate for 24-hour periods per sample.

**Personnel.** For low-volume personnel monitoring samples for PCB analysis, Earth Tech personnel will wear a portable pump with an air hose and TO-10A PUF cartridge attached near the breathing zone. Personnel samples will be collected during each work day over an 8-hour period.

Field QC samples will consist of field duplicate samples. MS/MSD samples are not typically collected for EPA Methods TO-4A and TO-10A; therefore Air Toxics will analyze LCS/LCSD samples at a frequency of one per group of 20 or fewer investigative samples for each sample matrix and parameter. For each matrix and parameter analyzed, one additional sample container will be analyzed for LCS analysis and one additional sample container will be analyzed for LCSD analysis. LCS/LCSD samples will be analyzed using the same procedures used to analyze the field samples, except that they will be clearly designated for LCS/LCSD analysis.

## **4.2 SAMPLE HANDLING PROCEDURES**

Tetra Tech will prepare the PUF sample cartridges for shipment and complete all necessary paperwork. Sample containers, sample preservation and preparation, and sample numbering procedures are discussed below.

### **4.2.1 Sample Containers**

Contaminant-free PUF cartridges to be used for samples undergoing organic analyses will be prepared in accordance with the procedures specified in EPA's "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" (EPA 1992). Specifications for the PUF cartridges will be verified by Tetra Tech by checking the supplier's certified statement and analytical results for each container lot. These activities will be documented on a continuing basis, and the documents will be maintained in the project evidence file. PUF sample cartridge requirements are summarized in Table 4-1.

### **4.2.2 Sample Preservation and Preparation**

Sample preservation for the PUF cartridges includes storage at 4°C ( $\pm 2^\circ\text{C}$ ) until analysis. Samples will be wrapped in aluminum foil, sealed in plastic bags, and shipped to Air Toxics by overnight express carrier on the day of collection. Sample preservation and holding times are summarized in Table 4-1.

### **4.2.3 Sample Numbering**

A sample numbering system will be used to identify each sample for chemical analysis. Each number will include three identifiers: project identification, sample type, and sample identifier. A list of the sample identification numbers will be maintained in the logbook by the field leader. The three identifiers are described below.

### **Project Identification**

A three-letter project identification number will be used to identify the site where each sample was collected. "ICS" will be used for Illinois Central Spring.

### **Sample Type**

Each sample collected will be further identified by letters corresponding to the sample type. The code for the high-volume perimeter air samples will be "HV" and low-volume personnel air samples will be "LV".

### **Sample Identifier**

A sample identifier will follow the sample type. For each high-volume perimeter air sample, a one-digit number will be used to indicate the sample collection location, followed by a two-digit number to indicate the particular day in the sampling period. For each low-volume personnel air sample, a two-digit number will be used to indicate the particular day in the sampling period.

Examples of low- and high-volume sample numbers are listed below.

- ICS-LV-03 = Illinois Central Spring, low-volume personnel air monitoring sample collected during the third day of the sampling period
- ICS-HV4-10 = Illinois Central Spring, high-volume perimeter air sample collected from PUF sampler location no. 4 during the tenth day of the sampling period.

**TABLE 4-1**  
**SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES**

Analytical Parameter	No. of Containers Per Sample	Container Type	Preservation Method	Contractual Holding Time
<b>Perimeter and Personnel Air Samples</b>				
High-Volume PCBs	One	PUF TO-4 cartridge, 102 mm quartz fiber filter	Store at 4°C (± 2°C) until analysis	7 days to extraction; 40 days extract holding time
Low-Volume PCBs	One	PUF TO-10 cartridge, 32 mm quartz fiber filter	Store at 4°C (± 2°C) until analysis	7 days to extraction; 40 days extract holding time

Notes:

- °C = Degree celsius
- PCB = Polychlorinated biphenyl
- PUF = Polyurethane foam
- TO = Toxic organics

Sampling activities will be documented in a bound logbook using a ballpoint pen in accordance with Tetra Tech's field SOP No. 024 (see Attachment 1). The time of collection, sample number, sampling location, field observations, sampler's name, and analyses will be recorded in the logbook for each sample. Each page of the logbook will be dated, numbered, and signed by Tetra Tech personnel. Field data records will be maintained in accordance with National Enforcement Investigation Center policies and procedures (EPA 1985).

#### **4.3 SAMPLE PACKAGING AND SHIPPING PROCEDURES**

All environmental samples collected for chemical analysis will be shipped on the day of collection using an overnight delivery service. PUF sample cartridges will be wrapped in aluminum foil and placed in plastic bags in case the containers break during shipment. The sample containers will then be placed in coolers filled with a cushioning packing material. Blue ice packets supplied by the laboratory and stored in the ICS treatment facility freezer will then be placed in the coolers to maintain the temperature of the samples at 4 °C or lower during transport. Laboratory paperwork for the samples will be placed in a sealed plastic bag and taped to the inside of the cooler lid. The cooler lid will then be taped closed and chain-of-custody seals placed on the cooler lid. All air samples will be packaged and shipped as low-concentration samples (EPA 1988). All shipping containers will be labeled as required by the U.S. Department of Transportation. Sample packaging and shipping procedures are discussed in detail in Tetra Tech field SOP No. 019 (see Attachment 1).

After packaging, the samples will be shipped by overnight carrier to Tetra Tech's subcontracted laboratory, Air Toxics, of Folsom, California. Tetra Tech field technical staff and the sample coordinator from the Tetra Tech Chicago office will coordinate shipments with the laboratory for the planned analyses.

## SECTION 5 CUSTODY PROCEDURES

Proper chain-of-custody for samples is necessary for the admissibility of environmental data as evidence in a court of law. A sample or evidence file is considered under an individual's custody if any of the following conditions are met:

- The item is in the actual possession of the individual.
- The item is in the view of the individual after being in actual possession of the individual.
- The item was in the actual physical possession of an individual but is locked up to prevent tampering.
- The item is in a designated and identified secure area.

All chain-of-custody procedures will conform with the EPA Region 5 sample custody and chain-of-custody protocols described in "National Enforcement Investigation Center (NEIC) Policies and Procedures" (EPA 1985). This document divides chain-of-custody procedures into three categories: sample collection, laboratory analysis, and final evidence file. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area. Field chain-of-custody, laboratory chain-of-custody, and final evidence file chain-of-custody procedures are discussed below.

### 5.1 FIELD CHAIN-OF-CUSTODY PROCEDURES

Sample packaging and shipment procedures will help ensure that samples are shipped in accordance with all EPA-mandated chain-of-custody procedures. Protocols for specific sample numbering designations are discussed in Section 4. Field procedures, field logbooks and documentation, and transfer of custody and shipment procedures to maintain field chain-of-custody requirements are discussed below.

### **5.1.1 Field Procedures**

The following procedures must be followed to maintain chain-of-custody during sample collection in the field:

- The Tetra Tech field sampling leader is personally responsible for the care and custody of the samples until they are transferred or properly dispatched to the laboratory. As few people as possible will handle the samples.
- All bottles will be tagged with sample numbers and sampling location designations. Sample tags and labels must be affixed to each bottle shipped to the laboratory. QC samples, which receive an entirely separate sample identification number, will be noted under the sample description on the sample tag.
- Sample tags must be completed for each sample using waterproof ink unless prohibited by weather conditions. If waterproof ink is not used, a logbook notation should explain why. For example, the logbook could explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.
- Prior to shipping, the chain-of-custody forms, air bills, and all other relevant documents will be completed. Chain-of-custody forms will be sealed in plastic bags and taped to the inside of the cooler lid. An inert packing material will also be placed in the cooler for cushioning.

### **5.1.2 Field Logbooks and Documentation**

Field logbooks are used to record field activities. Logbook entries will describe field activities in as much detail as possible. Field logbooks are bound, field survey books or notebooks assigned to field personnel and stored in Tetra Tech's Chicago office document control room when not in use. Each logbook will be identified by a project-specific document number. The title page of each logbook will contain the following information:

- Person to whom the logbook is assigned
- Logbook number
- Project name

- Project start date
- Project end date

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

Measurements made and samples collected will be recorded. All entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out using a single strike mark. Whenever a sample is collected or a measurement is made, the time of sampling will be noted, along with a sample description, depth from which the sample was collected, and volume and number of containers used to hold the sample. The number of photographs taken of each sampling location, if any, will also be noted. All equipment used to take measurements will be identified, along with the date of calibration.

### **5.1.3 Transfer of Custody and Shipment Procedures**

EPA-mandated procedures for the transfer of sample custody are summarized below.

- Samples must be accompanied by a properly completed chain-of-custody form. The sample numbers and location designations must be listed on the chain-of-custody form. When transferring possession of samples, the individuals relinquishing and receiving the samples must sign, date, and note the time of transfer on the chain-of-custody record. This record documents the custody transfer of samples from the sampler to another party (for example, to a mobile laboratory, to the permanent laboratory, or to or from a secure storage area).
- Samples must be properly packaged on ice at 4 °C for shipment and dispatched to the appropriate laboratory for analysis with a signed custody record enclosed in each sample box or cooler. Shipping containers must be locked or secured with strapping tape. EPA custody seals must be used to seal each sample box or cooler for shipment to the laboratory. The preferred procedure includes attaching a custody seal to the front right or left side of the cooler and attaching the other seal on the back side of the cooler on the opposite corner or center of the cooler. Custody seals must be covered with clear plastic tape. The cooler must be strapped shut with strapping tape in at least two locations.

- All shipments must be accompanied by a chain-of-custody record identifying the contents. Colored copies of the chain-of-custody record will be delivered to the appropriate entities as required by the chain-of-custody form.
- If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading must be retained as part of the permanent documentation. If sent by mail, the sample package must be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.
- Samples will be shipped to the laboratory by overnight carrier within 2 days of collection.

Tetra Tech SOPs No. 018 and 019 (see Attachment 1) provide additional information concerning custody and shipment procedures.

## **5.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES**

Chain-of-custody procedures for the laboratory are described in Air Toxics SOP No. 50 for sample receiving, login, and tracking of samples (see Attachment 2).

## **5.3 FINAL EVIDENCE FILE CHAIN-OF-CUSTODY PROCEDURES**

Tetra Tech is the custodian of the evidence file and will maintain the contents of the evidence files for this field investigation, including all relevant records, reports, field logbooks, field notebooks, documents, photographs, drawings, field and data deliverables, correspondence, laboratory data, chain-of-custody documents, and data reviews. All evidence files will be kept in a secured, limited access area under custody of the Tetra Tech project manager. The final evidence file will be submitted to EPA with all other project files when Tetra Tech receives notification from EPA to close out the project TDD.

**SECTION 6**  
**CALIBRATION PROCEDURES**

The calibration of laboratory instruments will be consistent with Air Toxics SOP No. 26 for PCB analysis (see Attachment 2).

**SECTION 7**  
**ANALYTICAL PROCEDURES**

Up to 60 high-volume perimeter air samples and 10 personnel monitoring air samples from the ICS site will be analyzed for PCBs in accordance with Air Toxics SOP No. 26 for PCB analysis using EPA Methods TO-4A and TO-10A (see Attachment 2). All analytical and QC requirements specified in these methods will be adhered to by the laboratory.

**SECTION 8**  
**DATA REDUCTION, VALIDATION, AND REPORTING**

This section discusses the data reduction, validation, and reporting procedures to be used.

**8.1 DATA REDUCTION**

Data reduction will be performed in accordance with the requirements of Air Toxics SOP No. 26 for PCB analysis (see Attachment 2).

**8.2 DATA VALIDATION**

Tetra Tech will be responsible for all laboratory data validation. Laboratory data will be validated in accordance with procedures presented in "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review" (EPA 1994a).

**8.3 DATA REPORTING**

Data reporting will be performed in accordance with the requirements of Air Toxics SOP No. 26 for PCB analysis (see Attachment 2).

**SECTION 9**  
**INTERNAL QUALITY CONTROL CHECK**

The internal QC check of the laboratory performing the air sample analyses will be performed in accordance with the requirements of Air Toxics SOP No. 26 for PCB analysis (see Attachment 2).

## SECTION 10 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the procedures described in the field SAP. Audits of field and laboratory activities include two independent parts: internal and external audits. These types of audits for field and laboratory activities are discussed below.

### 10.1 FIELD AUDITS

Internal audits of field activities, including sampling and measurement, may be conducted by the Tetra Tech QA manager, or his designee. The audits would include examination of field sampling records, field instrument operating records, sample collection procedures, handling and packaging procedures, QA procedures, and chain-of-custody procedures. These audits may be conducted at the onset of the project to verify that all established procedures are followed. Follow-up audits may be conducted to correct deficiencies and to verify that QA procedures are maintained throughout the project. The audits will involve review of field measurement records, instrumentation calibration records, and sample documentation.

External audits may be conducted by EPA Region 5. External audits may be conducted at any time during the field investigation activities and may or may not be announced.

### 10.2 LABORATORY AUDITS

Air Toxics has received accreditation from the National Environmental Laboratory Accreditation Conference (NELAC) and is certified by the State of California Department of Health Services, New York Department of Health, Utah Department of Health, Arizona Department of Health, and Louisiana Department of Environmental Quality. Air Toxics is validated by the U.S. Army Corps of Engineers and serves as the QA laboratory for the New England District. In addition, Air Toxics (1) has been audited and approved for air analysis by the U.S. Navy Facilities Engineering Service Center, and (2) is a participant in the National Institute of Occupational Safety Hazards (NIOSH) PAT proficiency program.

External laboratory audits may be conducted by Tetra Tech and/or EPA Region 5. External audits may be conducted at any time during the laboratory activities and may or may not be announced.

**SECTION 11**  
**PREVENTIVE MAINTENANCE PROCEDURES**

Laboratory equipment will be maintained in accordance with the manufacturer's specifications and in such a way that the QC requirements of the laboratory SOP No. 26 for PCB analysis (see Attachment 2) will be met for all analyses performed.

**SECTION 12**  
**DATA ASSESSMENT PROCEDURES**

All analytical data will be evaluated for precision, accuracy and completeness. The acceptability of the analytical precision and accuracy will be determined by comparing them to the control limits recommended in the most current Air Toxics SOP No. 26 for PCB analysis (see Attachment 2). Data determined to be insufficiently precise or accurate will be subject to the corrective actions prescribed by the appropriate analytical method. The QC samples used to determine precision and accuracy are described in Section 3. Specific equations used to calculate precision, accuracy, and completeness are presented below.

**12.1           PRECISION**

The precision of laboratory analysis will be assessed by comparing the analytical results with LCS/LCSD results for organic analysis. The RPD will be calculated for each pair of duplicate analyses using the following equation:

$$RPD = \frac{S - D}{(S + D)/2} \times 100 \qquad (12-1)$$

where:

S = First sample value (original or LCS value)

D = Second sample value (duplicate or LCSD value)

## 12.2 ACCURACY

Accuracy of laboratory results will be assessed using method blank, LCS/LCSD, and equipment blank analytical results. The %R of LCS samples will be calculated using the following equation where:

$$\%R = \frac{A - B}{C} \times 100 \quad (12-2)$$

where:

A = Analyte concentration determined experimentally from spiked sample

B = Analyte concentration determined by a separate analysis of the unspiked sample

C = Amount of the spike added

## 12.3 COMPLETENESS

The completeness of laboratory analytical results will be calculated using the following equation.

$$\text{Completeness (\%)} = \frac{\text{Number of valid measurements}}{\text{Number of measurements planned}} \times 100 \quad (12-3)$$

## **SECTION 13 CORRECTIVE ACTIONS**

Corrective actions may be required to address analytical and equipment problems and noncompliance problems. Problems may occur during sampling and sample handling, sample preparation, laboratory instrument analysis, and data review. All problems will be corrected using a formal corrective action program that will be determined and implemented at the time the problem is identified. Corrective actions related to field procedures, laboratory analysis and data validation and assessment are discussed below.

### **13.1 FIELD PROCEDURES CORRECTIVE ACTIONS**

Corrective action for field procedures, including sample handling and packaging, and documentation, may be required. The project manager is responsible for controlling, tracking, and implementing the identified changes. Reports on all changes will be distributed to all affected parties, including the EPA OSC. The OSC will be notified before minor or major program changes are made in the field. Approval of the OSC will be sought prior to making any significant field changes.

### **13.2 LABORATORY ANALYSIS CORRECTIVE ACTIONS**

Specific analytical corrective actions are described in the most recent version of Air Toxics SOP No. 26 for PCB analysis (see Attachment 2) and will be adhered to by the laboratory.

### **13.3 DATA VALIDATION AND ASSESSMENT CORRECTIVE ACTIONS**

EPA Region 5 and Tetra Tech may request corrective action for any nonconformance identified during the data validation process. For minor problems, the laboratory may be contacted directly. Corrective actions may include the following:

- Reanalyzing the samples if holding time criteria permit

- Resampling and reanalyzing
- Evaluating and amending sampling procedures and/or evaluating and amending analytical procedures
- Accepting the data and acknowledging the level of uncertainty

The EPA OSC or his designee will issue a nonconformance report for each nonconformance situation. If resampling is deemed necessary because of laboratory problems, the EPA OSC must identify the necessary approach, including cost recovery from the laboratory for the additional sampling.

Tetra Tech may identify the need for corrective action during assessment of analytical data received from the laboratory. The Tetra Tech data assessor will report all corrective action requirements to the Tetra Tech project manager. After consultation with the EPA OSC, the Tetra Tech project manager will be responsible for approving and implementing the corrective actions. Corrective actions may include resampling by the field team or reanalysis by the laboratory. All corrective actions will be documented by the Tetra Tech project manager and reported to the Tetra Tech QA manager.

**SECTION 14**  
**QUALITY ASSURANCE REPORTS TO MANAGEMENT**

A QA/QC summary of all data quality information collected during the field investigation will be submitted to EPA as part of the sample evaluation report that Tetra Tech will prepare. The QA/QC summary will include the following information:

- Status of the project
- Changes in the field SAP
- Summary of QA/QC programs
- Results of technical systems and performance evaluation audits
- Significant QA/QC problems, recommended solutions, and corrective action results and their impact on the project
- Data quality assessment in terms of precision, accuracy, representativeness, completeness, comparability, and method detection limits
- Indication of whether QA objectives have been met
- Limitations to the use of measurement data
- Changes in key personnel

## REFERENCES

- Earth Tech, Inc. (Earth Tech). 2002. Bloomington, Indiana, AutoCAD Drawing. March 22.
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- EPA. 1994a. "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review." Office of Solid Waste and Emergency Response (OSWER). Washington, D.C. OSWER Publication 9240.1-05. February.
- EPA. 1994b. "Requirements for Quality Assurance Project Plans for Environmental Data Operations." QA/R-5. August.
- EPA. 1997. "Region 5 Superfund Model Mini Quality Assurance Project Plan." Revision 1 December 4.

**ATTACHMENT 1**

**TETRA TECH EM INC.  
FIELD STANDARD OPERATING PROCEDURES**

- SOP No. 018, "Sample Custody" (27 Sheets)
- SOP No. 019, "Packaging and Shipping Samples" (15 Sheets)
- SOP No. 024, "Recording Notes in the Field Logbook" (7 Sheets)

**SOP APPROVAL FORM**

**PRC ENVIRONMENTAL MANAGEMENT, INC.™**

**STANDARD OPERATING PROCEDURE**

**SAMPLE CUSTODY**

**SOP NO. 018**

**REVISION NO. 2**

Approved by:

Daniel Ashenberg  
Quality Assurance Officer

2/2/93  
Date

Date of Original Issue: 03/31/91

Title: Sample Custody

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## 1.0 BACKGROUND

In any sampling and analytical program, the integrity of a sample must be documented from its point of collection to its final disposition. The documentation of the possession and handling of samples is referred to as "chain of custody." The components of this chain, such as sample custody seals, traffic reports, field logbooks, chain-of-custody records, and sample identification tags, and the procedures for their use are described below.

### 1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for sample custody. It has been prepared in accordance with the U.S. Environmental Protection Agency (EPA)/National Enforcement Investigation Center (NEIC) User's Guide to the EPA Contract Laboratory Program (CLP). Sample custody and documentation procedures described in this SOP should be followed throughout all sample collection activities unless the procedures are revised by EPA. All revisions must be documented in the field logbook.

### 1.2 SCOPE

This SOP applies to sample custody and the activities associated with it.

### 1.3 DEFINITIONS

None.

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#### **1.4 REFERENCES**

U.S. Environmental Protection Agency, 1988, User's Guide to the EPA Contract Laboratory Program. EPA Office of Emergency and Remedial Response, Washington, D.C.

#### **1.5 REQUIREMENTS AND RESOURCES**

Numerous sample identification documents are used to control sample disposition and to maintain a chain-of-custody record for all samples collected. These documents include sample container labels, identification tags, custody seals, chain-of-custody records, and traffic report forms.

### **2.0 PROCEDURES**

The following subsections present detailed instructions for completing chain-of-custody documents, including sample container labels, sample identification tags, custody seals, chain-of-custody records, and traffic reports.

#### **2.1 SAMPLE CONTAINER LABELS**

The sample container label is an adhesive label with designated areas to indicate the station location, date and time of sample collection, analysis requested, and preservative added. Before placing the sample material in the appropriate container, the sampler should complete the label with waterproof ink and affix the label to the container. When necessary, the sample label should be protected from water and solvents with clear tape.

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## **2.2 SAMPLE IDENTIFICATION TAGS**

Sample identification tags (Figures 1 and 1A) provided by EPA are used to maintain control of laboratory samples. The PRC field team leader should distribute the tags to field team members. The EPA serial numbers should be recorded in the field notebook. Sample tags should be attached to all sample containers used. Unused tags should be returned to the field team leader. Each tag should be completed as follows:

- Space No. 1 should be completed with the case number provided by the CLP laboratory.
- Space No. 2 should be completed with the appropriate sample location number.
- Spaces No. 3A and 3B should be completed with the time and date of sample collection, respectively.
- Space No. 4 should designate whether the sample is grab or composite.
- Space No. 5 should be completed with the sampling location.
- Space No. 6 should contain the signature(s) of the sampler(s).
- Space No. 7 should designate whether a preservative has been added to the sample. If a preservative has been added to the sample, the name of the preservative should be written in the space marked "Remarks."
- Space No. 8 should designate all appropriate analyses to be performed by the laboratory.
- The remarks section should include the traffic report sample number and bottle lot number.

FIGURE 1

SAMPLE TAG



☆ GPO 808-467

Project Code	1-	Station Location 5	Station No.	2	Temperature (Signature)	Month/Day/Year	3B	Time	3A	Designator	Grid	Comp.	Grid		
<b>Preservative:</b> Yes <input type="checkbox"/> , No <input type="checkbox"/>															
<b>ANALYSES</b> 8															
BOD Solids (MS) (MS) (MS)      Anions															
COD, TOC, Nutrients															
Phenolics															
Mercury															
Metals															
Cyanide															
Oil and Grease															
Organics GC/MS															
Priority Pollutants															
Volatile Organics															
Pesticides															
Mutagenicity															
Bacteriology															
Remarks:															
Log No.      Lab Sample No.															
<b>3- 158232</b>															


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FIGURE 1A

SAMPLE TAG

Project Code Lab 5 3/0, CD1 21. SM10.0		Station No. MW 26 55-11(6)	Month/Day/Year 5-28-93	Time 1007	Background Cont. <input checked="" type="checkbox"/>
Station Location Monitoring well 112L Split Spoon # 11		Sampler (Signature) <i>Jane Doe</i>			
Tag No. H0502		Lab Sample No.			
Preservative: Yes <input type="checkbox"/> No <input checked="" type="checkbox"/>					
<b>ANALYSES</b>					
BOD	Asions				
Solids	(P&S) (TDS) (SS)				
COO, TOC, Nutrients					
Phenolics					
Mercury					
Metals					
Cyanide					
Oil and Grease					
Organics GC/MS					X
Priority Pollutants					X
Volatile Organics					X
Pesticides					X
Mutagenicity					
Bacteriology					
Remarks: CASE 1746 TR # ME0637 Bottle Lot # 63120					



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

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### 2.3 CUSTODY SEALS

A custody seal is an adhesive label. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been packed for shipping. When samples are shipped to an EPA regional CLP laboratory, they must be placed in containers sealed with EPA custody seals. Custody seals differ among EPA regions; three examples are shown on Figure 2. Some custody seals are serially numbered; others are unnumbered and only provide evidence that the sample has not been tampered with. Two seals must be signed and placed on each shipping container or cooler: one at the front and one at the back as shown on Figure 3. Clear tape should be placed over the seals to help ensure that they are not accidentally broken during shipment.

### 2.4 CHAIN-OF-CUSTODY RECORDS



All sample shipments should be accompanied by a chain-of-custody record (see Figures 4 and 4A) that identifies their contents. The original record should accompany the shipment, and the yellow copy should be given to the PRC field team leader. Because field sample data may be the object of litigation, the custody of the samples must be documented from collection through laboratory analysis. A chain-of-custody record accompanies the samples to identify each transfer of custody. Individuals relinquishing and receiving samples should sign, date, and note the time of transfer on the record. This record should be used to document sample custody transfer from the sampler to another PRC team member, to a shipper, or to the regional or CLP laboratory. All field sampling personnel should sign the form as field samplers. The first individual to relinquish custody must sign the chain-of-custody record as a field sampler.


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FIGURE 2  
CUSTODY SEALS

ENVIRONMENTAL PROTECTION AGENCY	SAMPLE NO.	DATE	EPA Form 7500-2 (10/71)
	SIGNATURE		
	PRINT NAME AND TITLE (Inspector, Analyst or Technician)		

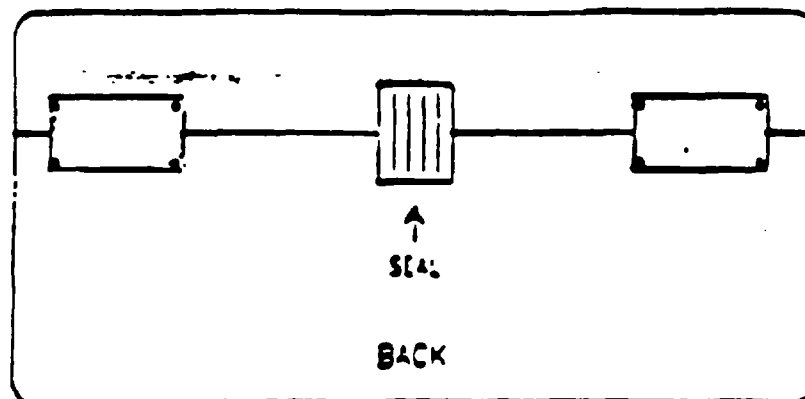
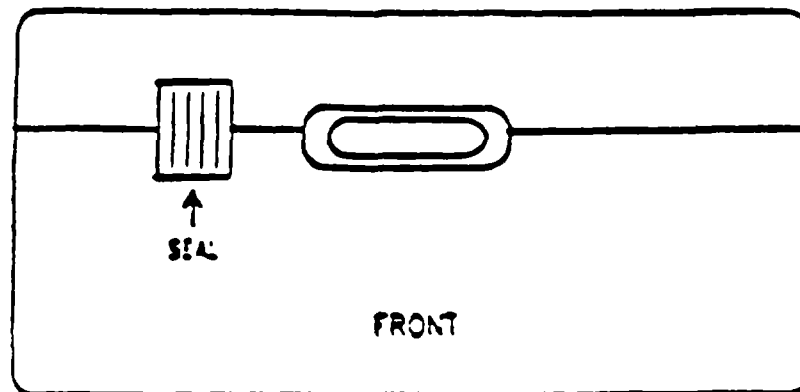
CUSTODY SEAL			CUSTODY SEAL
Signature			Date
Date			Signature

 UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICIAL SAMPLE SEAL	SAMPLE NO.	DATE	DATE	EPA Form 7500-2 (10/71)
	SIGNATURE			
	PRINT NAME AND TITLE (Inspector, Analyst or Technician)			

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**FIGURE 3**  
**PLACEMENT OF SECURITY SEALS ON**  
**SHIPPING CONTAINERS AND COOLERS**





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FIGURE 4A  
CHAIN-OF-CUSTODY RECORD

PROJ. NO. 12-2456				PROJECT NAME # 0123				NO. OF CONTAINERS	Total Metals Chemical				REMARKS
SAMPLERS: <i>John Samples</i>									ITR	TAG #			
STA. NO.	DATE	TIME	1	2	STATION LOCATION	1	2						
001	9/26	8:00	X		LOC-ST-010	2	X	X			ME1701	5-102501	
												5-102502	
002	9/26	11:00	X		LOC-ST-011	2	X	X			ME1702	5-102503	
												5-102504	
003	9/26	14:00	X		LOC-ST-012	2	X	X			ME1703	5-102505	
												5-102506	
004	9/26	17:00	X		LOC-ST-013	2	X	X			ME1704	5-102507	
												5-102508	
005	9/26	18:30	X		LOC-ST-014	2	X	X			ME1705	5-102509	
												5-102510	
Retrieved by: <i>John Samples</i>		Date / Time 9/26 18:00		Received by: <i>Federal Express</i>		Retrieved by: _____		Date / Time		Received by: _____			
Retrieved by: _____		Date / Time		Received by: _____		Retrieved by: _____		Date / Time		Received by: _____			
Retrieved by: _____		Date / Time		Received for Laboratory by: <i>Mary Sunshine</i>		Date / Time 9/27 10:00		Remarks CASE # 1000 Fed. Ex. # 122456789 Custody Seals # 81122 ; 81123					

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Samples should be packaged properly for shipment (as detailed in SOP No. 019) and should be dispatched to the appropriate laboratory for analysis. A separate chain-of-custody record should accompany each shipment.

Whenever samples are split with another party, it should be noted in the remarks section of the chain-of-custody record.

The procedure for completing the chain-of-custody record is as follows:

1. Space 1 should list the case number provided by the laboratory.
2. Space 2 should list the initials of the site name.
3. Space 3 should contain the signature(s) of the sampler(s).
4. Column 4 should list the sampling location number for each sample shipped.
5. Columns 5A and 5B should list the date and time each sample was taken.
6. Columns 6A and 6B should indicate whether each sample shipped was a composite or grab sample.
7. Column 7 should list the sampling location for each sample.
8. Column 8 should list the total number of sample containers for each sample shipped.
9. Columns 9A through 9F should indicate the type of sample shipped.
10. Column 10 should list the traffic report sample number and the sample identification tag number for each sample container shipped.
11. Space 11 should contain the signature of the sample collector.
12. Spaces 12A and 12B should list the date and time the samples were relinquished by the sample collector.

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13. Space 13 should list the carrier's name and the airbill number.
14. The sample cooler should be properly packed for shipping.
15. The completed original, white copy of the chain-of-custody record, should be shipped inside the cooler, as detailed in SOP No. 019. The yellow copy should be retained by the PRC field team leader and should be placed in the project file.

If sent by mail, the package should be registered and a return receipt should be requested. If sent by common carrier, a bill of lading should be used. Air freight shipments should be sent collect. Freight bills, postal service receipts, and bills of lading should be retained as part of the chain-of-custody documentation (see Figure 5). The carrier will provide the forms and instructions for filling them out.

## 2.5 TRAFFIC REPORTS

Different traffic report forms are used for each of the major types of assay under the CLP. These include organic, inorganic, dioxin, and special analytical service (SAS).

### 2.5.1 Organic and Inorganic Traffic Reports

For samples shipped to CLP laboratories for organic and inorganic analysis, field investigation personnel should use Organic Traffic Report forms (see Figures 6 and 6A) and Inorganic Traffic Report forms (see Figures 7 and 7A) provided by EPA.

Traffic reports are preprinted forms provided by EPA's Sample Management Office (SMO). These forms are part of EPA's sample tracking system and are used to trace shipment of samples for CLP laboratory analysis.

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FIGURE 5

AIRBILL

6725343811

29 928 6725343811

1246-2717-6

1303-299-1101

**PRC ENVIRONMENTAL MGMT INC**  
1099 10TH STE 1960  
DENVER CO 80202

SHIPPER'S CERTIFICATION FOR RESTRICTED ARTICLES/DANGEROUS GOODS

6725343811

TRANSPORT DETAILS	THE SHIPPER'S OTHER THE LABELING PROVIDED FOR	NEEDS RECEIPT	OTHER AIRCRAFT ONLY	SELF-IDENTIFIABLE
APPROX OF SIGNATURE	APPROX OF SIGNATURE	SHIPMENT TYPE	RESTRICTIVE	RESTRICTIVE

IF ACCEPTABLE FOR PASSENGER AIRCRAFT THE SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN OR INCIDENT TO RESEARCH MEDICAL DIAGNOSIS OR TREATMENT

I HEREBY DECLARE THAT THE CONTENTS OF THIS COMBINATION ARE FULLY AND ACCURATELY DESCRIBED ABOVE BY PROPER SHIPPING NAME AND ARE CLASSIFIED, PACKED, MARKED, AND LABELED, AND ARE IN ALL RESPECTS IN PROPER CONDITION FOR TRANSPORT BY AIR ACCORDING TO THE APPLICABLE INTERNATIONAL AND NATIONAL GOVERNMENT REGULATIONS.

NAME AND TITLE OF SHIPPER: \_\_\_\_\_ PLACE AND DATE: \_\_\_\_\_

EMERGENCY TELEPHONE NUMBER: \_\_\_\_\_ SIGNATURE OF SHIPPER: \_\_\_\_\_ SEE WARNING

ORIGIN COPY



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FIGURE 6A  
ORGANIC TRAFFIC REPORT

EPA Organic Traffic Report (For CLP Use Only)										Class Number	Lab No. of specimens	
1. Type of Activity (Check one) <input type="checkbox"/> Air <input type="checkbox"/> Land <input type="checkbox"/> Water <input type="checkbox"/> Other			2. Region Number I		3. Name of Company XYZ Co.		4. Date Shipped / Arrival Number 11-4-90 / 1234567890		5. Sample Description (Enter in Column 4)			
6. Site Name Drum Site			7. Site Address Anytown, MA		8. Lab to CLP Lab 100 Main St. Tiny Town, ME 05555		9. Shipping Method Fed Ex		10. Sample Description (Enter in Column 4) 1. Surface Water 2. Ground Water 3. Leachate 4. Sludge 5. Soil/Sediment 6. Oil (OIL) 7. Waste (WAS) 8. Other (OAS) (Specify)			
CLP Site No.	No. of Specimens	Type of Sample	No. of Specimens			No. of Specimens	No. of Specimens	No. of Specimens	No. of Specimens	No. of Specimens	No. of Specimens	No. of Specimens
			1	2	3							
AB123	2	L	X	X	X							MAZ 221
AB124	2	L	X	X	X							MAZ 22
AB125	2	L	X	X	X							MAZ 223
AB126	2	L	X	X	X							MAZ 224
AB127	2	L	X	X	X							MAZ 225
AB128	2	L	X	X	X							MAZ 226
AB129	2	L	X	X	X							MAZ 227
AB130	2	L	X	X	X							MAZ 228
AB131	2	L	X	X	X							MAZ 229
AB132	2	L	X	X	X							MAZ 230
AB133	2	L	X	X	X							MAZ 231
AB134	2	L	X	X	X							MAZ 232
AB135	2	L	X	X	X	MS/MSD						MAZ 233
AB136	2	L	X	X	X							MAZ 234
AB137	2	L	X	X	X							MAZ 235
AB138	2	L	X	X	X							MAZ 236
<b>SHIPPING COMPLETE</b>												
No more to ship under this org no.												

MS - MSD Only MS/MSD - Lab Only for MSD in MS/MSD MS/MSD - Lab Only



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FIGURE 7A  
INORGANIC TRAFFIC REPORT

EPA United States Environmental Protection Agency Central Laboratory Program Region Management Office PO Box 910 Alexandria, VA 22304 703 557-7000 FTS 557-7000										Inorganic Traffic Report (For CLP Use Only)		Case Number 10101	SM No. if applicable	
1. Type of Activity (Check one) <input type="checkbox"/> SW <input type="checkbox"/> WPLC <input type="checkbox"/> PA <input checked="" type="checkbox"/> B <input type="checkbox"/> ST <input type="checkbox"/> Other (Specify)				2. Reporting Number X		3. Sampling Co. Acme Co.		4. Date Shipped 11-4-88		Actual Number 0787659321		5. Sample Description (Enter in Column A)		
6. Sample Program Drum Site				7. Sample Name Joan Sampler		8. Ship To Analytical Lab 100 Center Ave Anytown, CA 94568		9. Ship Method Fed Ex		10. Remarks Double volume required for metals ephalothalate separate sample. Ship medium and high concentration samples in glass jars. See manual for additional instructions.		11. Sample Matrix 1. Surface Water 2. Ground Water 3. Leachate 4. Process 5. Soil/Sediment 6. Oil (SAS) 7. Waste (SAS) 8. Other (SAS) (Specify)		
12. Site Name Green City, OR				13. Site ID 05		14. Attn Attn A. Metal								
CLP Sample Number (From Manual)	SM Sample Type (Part 9)	SM Container Type (Part 9)	15. Analysis		SM Special Handling	SM Status Location	SM Date/Time of Sample Collection	SM Contaminant Code Sample Number						
			Metals	Organic										
MTZ 900	1	L	X	X		LOC-1	11-4/0700	JA 321						
MTZ 901	1	L	X	X		LOC-2	11-4/0730	JA 322						
MTZ 902	1	L	X	X		LOC-3	11-4/0800	JA 323						
MTZ 903	1	L	X	X		LOC-4	11-4/0830	JA 324						
MTZ 904	1	L	X	X		LOC-5	11-4/0900	JA 325						
MTZ 905	1	L	X	X		LOC-6	11-4/0930	JA 326						
MTZ 906	1	L	X	X		LOC-7	11-4/0945	JA 327						
MTZ 907	1	L	X	X		LOC-8	11-4/1000	JA 328						
MTZ 908	1	L	X	X		LOC-9	11-4/1030	JA 329						
MTZ 909	1	L	X	X		LOC-10	11-4/1100	JA 330						
MTZ 910	1	L	X	X		LOC-11	11-4/1130	JA 331						
MTZ 911	1	L	X	X		LOC-12	11-4/1200	JA 332						
MTZ 912	1	L	X	X		LOC-13	11-4/1215	JA 333						
MTZ 913	1	L	X	X		LOC-14	11-4/1245	JA 334						
MTZ 914	1	L	X	X		LOC-15	11-4/1300	JA 335						
MTZ 915	1	L	X	X		LOC-16	11-4/1330	JA 336						
MTZ 916	1	L	X	X		LOC-17	11-4/1400	JA 337						
MTZ 917	1	L	X	X		LOC-18	11-4/1430	JA 338						
MTZ 918	1	L	X	X		LOC-19	11-4/1500	JA 339						
MTZ 920	1	L	X	X	MS/dup	LOC-20	11-4/1530	JA 340						

EPA Form 700-1 (8-88) Replaces EPA Form 700-1 which may be used. Green - OHS Copy Pink - Region Copy White - Lab Copy for Return to OHS Yellow - Lab Copy

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The CLP generates unique sample numbers for each organic and inorganic sample. The unique sample numbers are printed on adhesive labels. The sampler is responsible for assigning this critical sample number correctly and transcribing it accurately onto the traffic report. The labels should be attached to each sample container prior to shipment. Organic sample number labels have the format XX123 and have 10 labels per strip: four for extractables, two for volatile organic analyses (VOA), and four extra blanks. Inorganic sample number labels have the format MXX123 and have seven labels per strip: two for total metals, two for cyanide, and three extra blanks (see Figure 8). The unique sample number must be used only once. Unused labels should be destroyed to prevent duplication of sample numbers.

Organic and inorganic traffic reports should be completed as follows:

- The spaces indicated by case number and Special Analytical Service (SAS) number (if applicable) in the top right corner should be completed with the appropriate numbers.
- Box 1 should indicate the type of sampling activity performed, the site name, the city, the state, and the site spill identification number.
- Box 2 should indicate the EPA region number, the name of the sampling company, and the name of the sampler.
- Box 3 should indicate the name of the sample custodian or CLP contact the sample is being shipped to, the name of the CLP laboratory, and its full address.
- Box 4 should indicate the date shipped, the carrier (abbreviated), and the airbill number.
- The CLP sample number from the printed sample labels should be listed in the far left column.

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**FIGURE 8**  
**SAMPLE NUMBER LABELS**

<b>INORGANIC SAMPLE NUMBER LABELS</b>		<b>ORGANIC SAMPLE NUMBER LABELS</b>	
MCK 00	- Total Metals	CC 20	- Extractable
MCK 00	- Total Metals	CC 20	- Extractable
MCK 00	- Cyanide	CC 20	- Extractable
MCK 00	- Cyanide	CC 20	- Extractable
MCK 00		CC 20	- VOA
MCK 00		CC 20	- VOA
MCK 00		CC 20	
MCK 00		CC 20	
MCK 00		CC 20	
MCK 00		CC 20	

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- Column A should indicate the appropriate sample description code from box 5. Blanks should be listed as item 3, "leachate." The word "blank" should be written in column D, the special handling section. Item 6, "oil," and item 7, "waste," should be used for Routine Analytical Service (RAS) PLUS SAS projects only. Do not ship oily samples or waste without making prior arrangements with the SMO.
- Column B should indicate the concentration of the sample shipped. For organic samples, low- or medium-concentration samples should be labeled "L," and high-concentration samples should be labeled "H." For inorganic samples, low-concentration samples should be labeled "L," medium-concentration samples should be labeled "M," and high-concentration samples should be labeled "H." Do not ship high concentrated samples without making prior arrangements with the SMO.
- Column C should indicate the appropriate analytical fractions requested under RAS for each sample.
- Column D should specify any special handling instructions for each sample. Blank samples should be identified in this space. When shipping RAS PLUS SAS samples, the sampler may code SAS parameters in the blank space and enter the codes in this column.
- Column E should indicate the sampling location.
- Column F should indicate the date and time of sample collection.
- Column G should indicate the corresponding CLP sample number for organic or inorganic analysis.

The original, white copy of the traffic report should be sent to SMO, P.O. Box 818, Alexandria, Virginia 22313. The phone number of the SMO is 703/557-2490. The pink copy should be retained by the PRC field team leader. The other white copy and the yellow copy should be sent with the shipment to the laboratory. The address of the laboratory will be provided by SMO.

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### **2.5.2 Dioxin Shipment Record**

The CLP Dioxin Shipment Record, a four-part, carbonless form, is used to document samples for the dioxin program (see Figures 9 and 9A). This form must be used for any dioxin samples. The dioxin shipment record provides documentation in each shipment of dioxin samples.

The form is similar to the traffic reports described above. To provide a permanent record of each sample collected, the sampler should record the appropriate case number and batch or shipment number on each shipment record form. The sampler should record the type of sampling activity, regional information, shipping information, and analysis laboratory. For each sample, the sampler should record the sample matrix and its description, such as soil or sediment field sample or solvent rinsate, by checking the appropriate box following each sample number.

After completing the dioxin shipment record, the sampler should send the bottom two copies to the laboratory with the sample shipment. Following sample shipment, the sampler should send the top copy to the SMO and retain the remaining copy as a file copy.

### **2.5.3 Special Analytical Service (SAS) Packing List**

For samples requiring special analytical services, samplers should use the SAS Packing List, a four-part carbonless form (see Figures 10 and 10A). The packing list provides space for up to 20 samples on one form. These samples should be numbered with the SAS number provided by the SMO. The SAS number, such as 2000E, should be followed by a hyphen and a progressive numerical designation starting with one, such as 2000E-1, 2000E-2, 2000E-3, and so on. If the sampling activity continues for several days and requires more than one list, sample numbers should not be repeated. EPA's regional office will verify with SMO that the packing list is appropriate for use in the situation.



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FIGURE 9A  
CLP DIOXIN SHIPMENT FORM

USEPA Central Laboratory Program  
Residue Management Office  
P.O. Box 618 Annapolis, Virginia 22023  
PFS 8-657-6488 FAX 8-657-6489

CASE NO: 3000	BATCH NO: 0
SAS NO: N/A	

CLP DIOXIN SHIPMENT RECORD

Type of Activity (see 101) Substrate: PAH, PCB, DIOXIN, PCDD, PCDF, CHLORINATED BIPHENYL, OTHER	Region Number: IV	Site No: Dioxin Lab
Sampling Context: Drum Site	Sampling Contact: John Digger	100 Oak Run
City, State: Rustyville, Pa 1524	Sampling Date: 11/1/92	Treatment, PAH, PCB, DIOXIN, PCDD, PCDF, CHLORINATED BIPHENYL
Sampling Date: 11/1/92	Center: Fed. Ex.	Date Shipped: 11/2/92
	AMS# No: 122156309	

Sample Instructions: 1) Ship all samples in leak-free, leak-tight containers with sample labels affixed to outside of case.  
2) Use TCE or Isopar M for sample containers for dioxin samples.  
3) Sample Volumes Required: 200 µL for PCBs; 1 mL per sample in glass for PCBs; 2 Liters per sample in other glass. Send one 4 Liter sample per batch of samples destined for lab use.  
4) Samples to ship will be analyzed at Lab as a split sample (SS). If the sample requires analysis prior to shipping, the container must include a separate sample labeled with a unique sample number.

CLP Sample ID	SAMPLE DESCRIPTION						SAMPLE LOCATION for case label	ANALYSIS
	SOIL OR SEDIMENT	AQUEOUS	GROUP RESIDUE (FORM BULK)	OTHER (SAS ONLY)	SAMPLE NO. (SAS ONLY)	DUPLICATE (SAS ONLY)		
DD011201	X						D01-1	
DD011202	X						D01-2	
DD011203	X						D01-3	
DD011204	X						D02-1	
DD011205	X						D02-1A	
DD011206			X				D02-2	
DD011207	X						D02-3	
DD011208	X				X		D02-1	
DD011209	X						D02-2	
DD011210	X						D02-3	
DD011211	X						D02-4	
DD011212	X				X		D04-1	
DD011213	X						D04-2	
DD011214	X						D05-1	
DD011215	X						D05-2	
DD011216	X						D06-1	
DD011217	X						D06-2	
DD011218	X						D06-3	
DD011219	X						D07-1	
DD011220	X						D09-2	
DD011221	X						D08-1	
DD011222		X			X		D09-1	
DD011223		X			X		D09-2	
DD011224		X					D09-3	

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FIGURE 10

SPECIAL ANALYTICAL SERVICE PACKING LIST

U.S. ENVIRONMENTAL PROTECTION AGENCY  
CLP Sample Management Office  
P.O. Box 818 - Alexandria, Virginia 22313  
Phone: 703/557-2490 - FTS/557-2490

SAS Number
------------

SPECIAL ANALYTICAL SERVICE  
PACKING LIST

Sampling Office	Sampling Date(s)	Ship To	For Lab Use Only
Sampling Contact	Date Shipped		Date Samples Rec'd
(name)	Site Name/Code	Attn	Received By
(phone)			

Sample Numbers	Sample Description La., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

For Lab Use Only

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FIGURE 10A  
SPECIAL ANALYTICAL SERVICE PACKING LIST

U.S. ENVIRONMENTAL PROTECTION AGENCY  
CLP Sample Management Office  
P.O. Box 818 - Alexandria, Virginia 22313  
Phone: 703/557-2490 - FTS/557-2490

SAS Number  
1000 - A

SPECIAL ANALYTICAL SERVICE  
PACKING LIST

Sampling Office <u>Region I</u>	Sampling Date(s) <u>11/2 - 11/4/88</u>	Ship To <u>SAS LAB</u>	For Lab Use Only
Sampling Contact <u>Joe Samoky</u> (name)	Date Shipped <u>11/4/88</u>	<u>100 Main Street</u>	Date Samples Rec'd _____
<u>147/555-1234</u> (phone)	Site Name/Code <u>#01</u>	<u>Anytown, CO 98765</u>	Received By: _____
		Attn <u>Jim Smith</u>	

Sample Numbers	Sample Description Ln., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1. <u>1000A-01</u>	<u>LOW CONC. WATER - 2,4-D; 2,4,5-TP</u>	_____
2. <u>1000A-02</u>	"	_____
3. <u>1000A-03</u>	"	_____
4. <u>1000A-04</u>	"	_____
5. <u>1000A-05</u>	"	_____
6. <u>1000A-06</u>	"	_____
7. _____	_____	_____
8. _____	_____	_____
9. _____	_____	_____
10. _____	_____	_____
11. _____	_____	_____
12. _____	_____	_____
13. _____	_____	_____
14. _____	_____	_____
15. _____	_____	_____
16. _____	_____	_____
17. _____	_____	_____
18. _____	_____	_____
19. _____	_____	_____
20. _____	_____	_____

For Lab Use Only

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The sampler should complete the list by recording the SAS number, site name, location, sampling date, shipment date, analysis laboratory, sampling office (the organization that did the sampling), sampler names, sampler telephone number, individual SAS sample numbers, and sample description. The description must include the sample matrix, concentration (if applicable), and analyses to be done. After completing the list, the sampler should send the bottom two copies to the laboratory with the sample shipment. Following sample shipment, the sampler should send the top copy to the SMO and should retain the second copy as a file copy.

**SOP APPROVAL FORM**

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

**PACKAGING AND SHIPPING SAMPLES**

**SOP NO. 019**

**REVISION NO. 5**

Last Reviewed: January 2000

*K. Miesing*

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Quality Assurance Approved

*January 28, 2000*

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Date

## 1.0 BACKGROUND

In any sampling program, the integrity of a sample must be ensured from its point of collection to its final disposition. Procedures for classifying, packaging, and shipping samples are described below. Steps in the procedures should be followed to ensure sample integrity and to protect the welfare of persons involved in shipping and receiving samples. When hazardous substances and dangerous goods are sent by common carrier, their packaging, labeling, and shipping are regulated by the U.S. Department of Transportation (DOT) Hazardous Materials Regulations (HMR, *Code of Federal Regulations*, Title 49 [49 CFR] Parts 106 through 180) and the International Air Transportation Association (IATA) Dangerous Goods Regulations (DGR).

### 1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for packaging and shipping samples. It has been prepared in accordance with the U.S. Environmental Protection Agency (EPA) "Sampler's Guide to the Contract Laboratory Program (CLP)," the DGR, and the HMR. Sample packaging and shipping procedures described in this SOP should be followed for all sample packaging and shipping. Deviations from the procedures in this SOP must be documented in a field logbook. This SOP assumes that samples are already collected in the appropriate sample jars and that the sample jars are labeled and tagged appropriately.

### 1.2 SCOPE

This SOP applies to sample classification, packaging, and shipping.

### 1.3 DEFINITIONS

**Custody seal:** A custody seal is a tape-like seal. Placement of the custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been packaged for shipping.

**Dangerous goods:** Dangerous goods are articles or substances that can pose a significant risk to health, safety, or property when transported by air; they are classified as defined in Section 3 of the DGR (IATA 1999).

**Environmental samples:** Environmental samples include drinking water, most groundwater and ambient surface water, soil, sediment, treated municipal and industrial wastewater effluent, and biological specimens. Environmental samples typically contain low concentrations of contaminants and when handled require only limited precautionary procedures.

**Hazardous Materials Regulations:** The HMR are DOT regulations for the shipment of hazardous materials by air, water, and land; they are located in 49 CFR 106 through 180.

**Hazardous samples:** Hazardous samples include dangerous goods and hazardous substances. Hazardous samples shipped by air should be packaged and labeled in accordance with procedures specified by the DGR; ground shipments should be packaged and labeled in accordance with the HMR.

**Hazardous substance:** A hazardous substance is any material, including its mixtures and solutions, that is listed in Appendix A of 49 CFR 172.101 and its quantity, in one package, equals or exceeds the reportable quantity (RQ) listed in the appendix.

**IATA Dangerous Goods Regulations:** The DGR are regulations that govern the international transport of dangerous goods by air. The DGR are based on the International Civil Aviation Organization (ICAO) Technical Instructions. The DGR contain all of the requirements of the ICAO Technical Instructions and are more restrictive in some instances.

**Nonhazardous samples:** Nonhazardous samples are those samples that do not meet the definition of a hazardous sample and **do not** need to be packaged and shipped in accordance with the DGR or HMR.

**Overpack:** An enclosure used by a single shipper to contain one or more packages and to form one handling unit (IATA 1999). For example, a cardboard box may be used to contain three fiberboard boxes to make handling easier and to save on shipping costs.

#### 1.4 REFERENCES

U.S. Department of Transportation, Transport Canada, and the Secretariat of Communications and Transportation of Mexico (DOT and others). 1996. "1996 North American Emergency Response Guidebook."

International Air Transport Association (IATA). 1997. "Guidelines for Instructors of Dangerous Courses."

IATA. 1999. "Dangerous Goods Regulations." 40th Edition.

U.S. Environmental Protection Agency. 1996. "Sampler's Guide to the Contract Laboratory Program." Office of Solid Waste and Emergency Response. Washington, DC. EPA/540/R-96/032. On-Line Address: <http://www.epa.gov/oerrpage/superfund/programs/clp/guidance.htm#sample>

#### 1.5 REQUIREMENTS AND RESOURCES

The procedures for packaging and shipping **nonhazardous** samples require the following:

- Coolers
- Ice
- Vermiculite, bubble wrap, or similar cushioning material
- Chain-of-custody forms and seals
- Airbills
- Resealable plastic bags for sample jars and ice
- Tape (strapping and clear)

The procedures for packaging and shipping **hazardous** samples require the following:

- Ice
- Vermiculite or other non-combustible, absorbent packing material
- Chain-of-custody forms and seals
- Appropriate dangerous goods airbills and emergency response information to attach to the airbill

- Resealable plastic bags for sample jars and ice
- Tape (strapping and clear)
- Appropriate shipping containers as specified in the DGR
- Labels that apply to the shipment such as hazard labels, address labels, "Cargo Aircraft Only" labels, and package orientation labels (up arrows)

## 2.0 PROCEDURES

The following procedures apply to packaging and shipping nonhazardous and hazardous samples.

### 2.1 SAMPLE CLASSIFICATION

Prior to sample shipment, it must be determined whether the sample is subject to the DGR. Samples subject to these regulations shall be referred to as hazardous samples. If the hazardous sample is to be shipped by air, then the DGR should be followed. Any airline, including FedEx, belonging to IATA must follow the DGR. As a result, FedEx **may not** accept a shipment that is packaged and labeled in accordance with the HMR (although in most cases, the packaging and labeling would be the same for either set of regulations). The HMR states that a hazardous material may be transported by aircraft in accordance with the ICAO Technical Instruction (49 CFR 171.11) upon which the DGR is based. Therefore, the use of the DGR for samples to be shipped by air complies with the HMR, but not vice versa.

Most environmental samples are not hazardous samples and do not need to be packaged in accordance with any regulations. Hazardous samples are those samples that can be classified as specified in Section 3 of the DGR, can be found in the List of Dangerous Goods in the DGR in bold type, are considered a hazardous substance (see definition), or are mentioned in "Section 2 - Limitations" of the DGR for countries of transport or airlines (such as FedEx). The hazard classifications specified in the DGR (and the HMR) are as follows:

#### Class 1 - Explosives

Division 1.1 - Articles and substances having a mass explosion hazard

- Division 1.2 - Articles and substances having a projection hazard but not a mass explosion hazard
- Division 1.3 - Articles and substances having a fire hazard, a minor blast hazard and/or a minor projection hazard but not a mass explosion hazard
- Division 1.4 - Articles and substances presenting no significant hazard
- Division 1.5 - Very sensitive substances mass explosion hazard
- Division 1.6 - Extremely insensitive articles which do not have a mass explosion hazard

**Class 2 - Gases**

- Division 2.1 - Flammable gas
- Division 2.2 - Non-flammable, non-toxic gas
- Division 2.3 - Toxic gas

**Class 3 - Flammable Liquids**

**Class 4 - Flammable Solids; Substances Liable to Spontaneous Combustion; Substances, which, in Contact with Water, Emit Flammable Gases**

- Division 4.1 - Flammable solids.
- Division 4.2 - Substances liable to spontaneous combustion.
- Division 4.3 - Substances, which, in contact with water, emit flammable gases.

**Class 5 - Oxidizing Substances and Organic Peroxide**

- Division 5.1 - Oxidizers.
- Division 5.2 - Organic peroxides.

**Class 6 - Toxic and Infectious Substances**

- Division 6.1 - Toxic substances.
- Division 6.2 - Infectious substances.

**Class 7 - Radioactive Material**

**Class 8 - Corrosives**

**Class 9 - Miscellaneous Dangerous Goods**

The criteria for each of the first eight classes are very specific and are outlined in Section 3 of the DGR and 49 CFR 173 of the HMR. Some classes and divisions are further divided into packing groups based on their level of danger. Packing group I indicates a great danger, packing group II indicates a medium danger, and packing group III indicates a minor danger. Class 2, gases, includes any compressed gas being

shipped and any noncompressed gas that is either flammable or toxic. A compressed gas is defined as having a pressure over 40 pounds per square inch (psi) absolute (25 psi gauge). Most air samples and empty cylinders that did not contain a flammable or toxic gas are exempt from the regulations. An empty hydrogen cylinder, as in a flame ionization detector (FID), is considered a dangerous good unless it is properly purged with nitrogen in accordance with the HMR. A landfill gas sample is usually considered a flammable gas because it may contain a high percentage of methane. Class 3, flammable liquids, are based on the boiling point and flash point of a substance. Most class 3 samples include solvents, oil, gas, or paint-related material collected from drums, tanks, or pits. Division 6.1, toxic substances, is based on oral toxicity ( $LD_{50}$  [lethal dose that kills 50 percent of the test animals]), dermal toxicity ( $LD_{50}$  values), and inhalation toxicity ( $LC_{50}$  [lethal concentration that kills 50 percent of the test animals] values). Division 6.1 substances include pesticides and cyanide. Class 7, radioactive material, is defined as any article or substance with a specific activity greater than 70 kiloBecquerels (kBq/kg) (0.002 [microCuries per gram [ $\mu$ Ci/g)]). If the specific activity exceeds this level, the sample should be shipped in accordance with Section 10 of the DGR. Class 8, corrosives, are based on the rate at which a substance destroys skin tissue or corrodes steel; they are not based on pH. Class 8 materials include the concentrated acids used to preserve water samples. Preserved water samples are not considered class 8 substances and should be packaged as nonhazardous samples. Class 9, miscellaneous dangerous goods, are substances that present a danger but are not covered by any other hazard class. Examples of class 9 substances include asbestos, polychlorinated biphenyls (PCB), and dry ice.

Unlike the DGR, the HMR includes combustible liquids in hazard class 3. The definition of a combustible liquid is specified in 49 CFR 173.120 of the HMR. The HMR has an additional class, ORM-D, that is not specified in the DGR. "ORM-D material" refers to a material such as a consumer commodity, that although otherwise subject to the HMR, presents a limited hazard during transport due to its form, quantity, and packaging. It must be a material for which exceptions are provided in the table of 49 CFR 172.101. The DGR lists consumer commodities as a class 9 material.

In most instances, the hazard of a material sampled is unknown because no laboratory testing has been conducted. A determination as to the suspected hazard of the sample must be made using knowledge of the site, field observations, field tests, and other available information.

According to 40 CFR 261.4(d) and (e), samples transported to a laboratory for testing or treatability studies, including samples of hazardous wastes, are **not** hazardous wastes. FedEx will not accept a shipment of hazardous waste.

## 2.2 PACKAGING NONHAZARDOUS SAMPLES

Nonhazardous samples, after being appropriately containerized, labeled, and tagged, should be packaged in the following manner. Note that these are general instructions; samplers should be aware of any client-specific requirements concerning the placement of custody seals or other packaging provisions.

1. Place the sample in a resealable plastic bag.
2. Place the bagged sample in a cooler and pack it to prevent breakage.
3. Prevent breakage of bottles during shipment by either wrapping the sample container in bubble wrap, or lining the cooler with a noncombustible material such as vermiculite. Vermiculite is especially recommended because it will absorb any free liquids inside the cooler. It is recommended that the cooler be lined with a large plastic garbage bag before samples, ice, and absorbent packing material are placed in the cooler.
4. Add a sufficient quantity of ice to the cooler to cool samples to 4 °C. Ice should be double bagged in resealable plastic bags to prevent the melted ice from leaking out. As an option, a temperature blank (a sample bottle filled with distilled water) can be included with the cooler.
5. Seal the completed chain-of-custody forms in a plastic bag and tape the plastic bag to the inside of the cooler lid.
6. Tape any instructions for returning the cooler to the inside of the lid.
7. Close the lid of the cooler and tape it shut by wrapping strapping tape around both ends and hinges of the cooler at least once. Tape shut any drain plugs on the cooler.
8. Place two signed custody seals on the cooler, ensuring that each one covers the cooler lid and side of the cooler. Place clear plastic tape over the custody seals.
9. Place address labels on the outside of the cooler.
10. Ship samples overnight by a commercial carrier such as FedEx.

### 2.3 PACKAGING HAZARDOUS SAMPLES

The procedures for packaging hazardous samples are summarized below. Note that according to the DGR, all spellings must be exactly as they appear in the List of Dangerous Goods, and only approved abbreviations are acceptable. The corresponding HMR regulations are provided in parentheses following any DGR referrals. The HMR must be followed only if shipping hazardous samples by ground transport.

1. Determine the proper shipping name for the material to be shipped. All proper shipping names are listed in column B of the List of Dangerous Goods table in Section 4 of the DGR (or column 2 of the Hazardous Materials Table in 49 CFR 172.101). In most instances, a generic name based on the hazard class of the material is appropriate. For example, a sample of an oily liquid collected from a drum with a high photoionization detector (PID) reading should be packaged as a flammable liquid. The proper shipping name chosen for this sample would be "flammable liquid, n.o.s." The abbreviation "n.o.s." stands for "not otherwise specified" and is used for generic shipping names. Typically, a specific name, such as acetone, should be inserted in parentheses after most n.o.s. descriptions. However, a technical name is not required when shipping a sample for testing purposes and the components are not known. If shipping a hazardous substance (see definition), then the letters "RQ" must appear in front of the proper shipping name.
2. Determine the United Nations (UN) identification number, class or division, subsidiary risk if any, required hazard labels, packing group, and either passenger aircraft or cargo aircraft packing instructions based on the quantity of material being shipped in one package. This information is provided in the List of Dangerous Goods (or Hazardous Materials Table in 49 CFR 172.101) under the appropriate proper shipping name. A "Y" in front of a packing instruction indicates a limited quantity packing instruction. If shipping dry ice or a limited quantity of a material, then UN specification shipping containers do not need to be used.
3. Determine the proper packaging required for shipping the samples. Except for limited quantity shipments and dry ice, these are UN specification packages that have been tested to meet the packing group of the material being shipped. Specific testing requirements of the packages is listed in Section 6 of the DGR (or 49 CFR 178 of the HMR). All UN packages are stamped with the appropriate UN specification marking. Prior planning is required to have the appropriate packages on hand during a sampling event where hazardous samples are anticipated. Most samples can be shipped in either a 4G fiberboard box, a 1A2 steel drum, or a 1H2 plastic drum. Drums can be purchased in 5- and 20-gallon sizes and are ideal for shipping multiple hazardous samples. When FedEx is used to ship samples containing PCBs, the samples must be shipped in an inner metal packaging (paint can) inside a 1A2 outer steel drum. This method of packaging PCB samples is in accordance with FedEx variation FX-06, listed in Section 2 of the DGR.

4. Place each sample jar in a separate resealable plastic bag. Some UN specification packagings contain the sample jar and plastic bag to be used when shipping the sample.
5. Place each sealed bag inside the approved UN specification container (or other appropriate container if a limited quantity or dry ice) and pack with enough noncombustible, absorbent, cushioning material (such as vermiculite) to prevent breakage and to absorb liquid.
6. Place chain-of-custody forms in a resealable plastic bag and either attach it to the inside lid of the container or place it on top inside the container. Place instructions for returning the container to the shipper on the inside lid of the container as appropriate. Close and seal the shipping container in the manner appropriate for the type of container being used.
7. Label and mark each package appropriately. All irrelevant markings and labels need to be removed or obliterated. All outer packagings must be marked with proper shipping name, UN identification number, and name and address of the shipper and the recipient. For carbon dioxide, solid (dry ice), the net weight of the dry ice within the package needs to be marked on the outer package. For limited quantity shipments, the words "limited quantity" or "LTD. QTY." must be marked on the outer package. Affix the appropriate hazard label to the outer package. If the material being shipped contains a subsidiary hazard, then a subsidiary hazard label must also be affixed to the outer package. The subsidiary hazard label is identical to the primary hazard label except that the class or division number is not present. It is acceptable to obliterate the class or division marking on a primary hazard label and use it as the subsidiary hazard label. If using cargo aircraft only packing instructions, then the "Cargo Aircraft Only" label must be used. Package orientation labels (up arrows) must be placed on opposite sides of the outer package. Figure 1 depicts a properly marked and labeled package.
8. If using an overpack (see definition), mark and label the overpack and each outer packaging within the overpack as described in step 7. In addition, the statement "INNER PACKAGES COMPLY WITH PRESCRIBED SPECIFICATIONS" must be marked on the overpack.
9. Attach custody seals, and fill out the appropriate shipping papers as described in Section 2.4.

## **2.4 SHIPPING PAPERS FOR HAZARDOUS SAMPLES**

A "Shippers Declaration for Dangerous Goods" and "Air Waybill" must be completed for each shipment of hazardous samples. FedEx supplies a Dangerous Goods Airbill to its customers; the airbill combines both

the declaration and the waybill. An example of a completed Dangerous Goods Airbill is depicted in Figure

2. A shipper's declaration must contain the following:

- Name and address of shipper and recipient
- Air waybill number (not applicable to the HMR)
- Page \_\_\_ of \_\_\_
- Deletion of either "Passenger and Cargo Aircraft" or "Cargo Aircraft Only," whichever does not apply
- Airport or city of departure
- Airport or city of destination
- Deletion of either "Non-Radioactive" or "Radioactive," which ever does not apply
- The nature and quantity of dangerous goods. This includes the following information in the following order (obtained from the List of Dangerous Goods in the DGR): proper shipping name, class or division number, UN identification number, packing group number, subsidiary risk, quantity in liters or kilograms (kg), type of packaging used, packing instructions, authorizations, and additional handling information. Authorizations include the words "limited quantity" or "LTD. QTY." if shipping a limited quantity, any special provision numbers listed in the List of Dangerous Goods in the DGR, and the variation "USG-14" when a technical name is required after the proper shipping name but not entered because it is unknown.
- Signature for the certification statement
- Name and title of signatory
- Place and date of signing certification
- A 24-hour emergency response telephone number for use in the event of an incident involving the dangerous good
- Emergency response information attached to the shipper's declaration. This information can be in the form of a material safety data sheet or the applicable North American Emergency Response Guidebook (NAERG; DOT 1996) pages. Figure 3 depicts the appropriate NAERG emergency response information for "Flammable liquids, n.o.s." as an example.

Note that dry ice does not require an attached shipper's declaration. However, the air waybill must include the following on it: "Dry ice, 9, UN1845, \_\_\_\_ x \_\_\_\_ kg." The blanks must include the number of packages and the quantity in kg in each package. If using FedEx to ship dry ice, the air waybill includes a box specifically for dry ice. Simply check the appropriate box and enter in the number of packages and quantity in each package.

The HMR requirements for shipping papers are located in 49 CFR 172 Subpart C.

### 3.0 POTENTIAL PROBLEMS

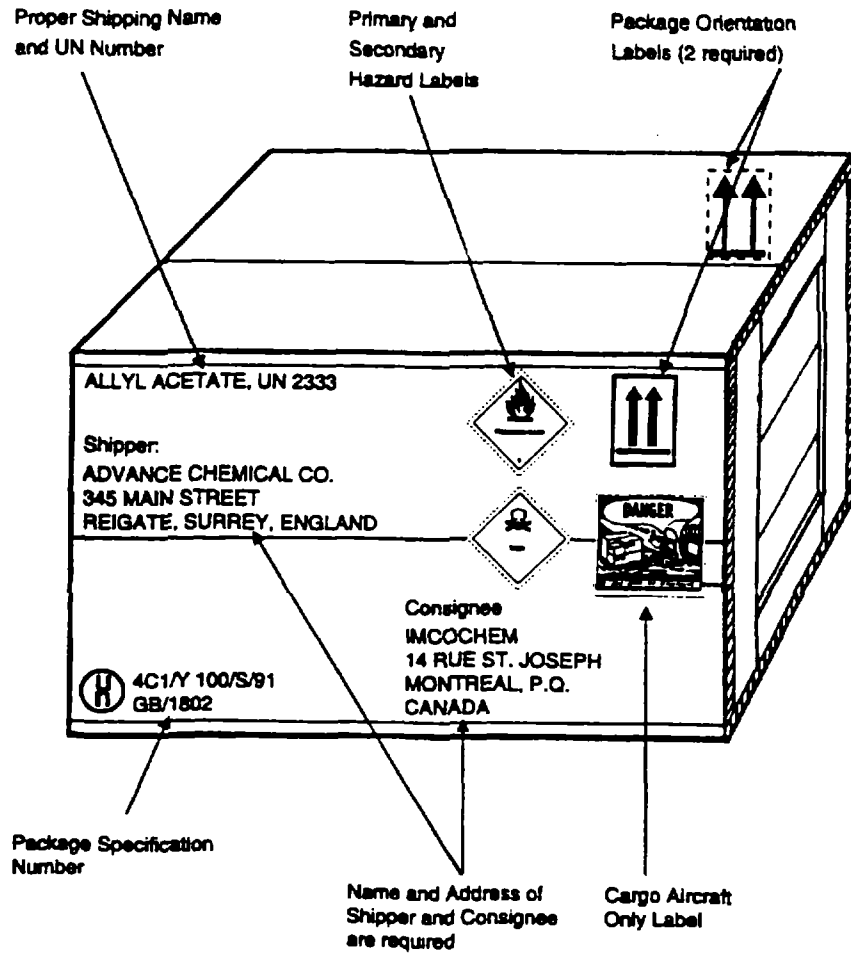
The following potential problems may occur during sample shipment:

- **Leaking package.** If a package leaks, the carrier may open the package, return the package, and if a dangerous good, inform the Federal Aviation Administration (FAA), which can result in fines.
- **Improper labeling and marking of package.** If mistakes are made in labeling and marking the package, the carrier will most likely notice the mistakes and return the package to the shipper, thus delaying sample shipment.
- **Improper, misspelled, or missing information on the shipper's declaration.** The carrier will most likely notice this as well and return the package to the shipper.

Contact FedEx with questions about dangerous goods shipments by calling 1-800-463-3339 and asking for a dangerous goods expert.

FIGURE 1

EXAMPLE OF A CORRECTLY MARKED AND LABELED DANGEROUS GOODS PACKAGE



Source: International Air Transport Association (IATA). 1997.

FIGURE 2

EXAMPLE OF A DANGEROUS GOODS AIRBILL

**FedEx Dangerous Goods Airbill** Sender's Copy *The World On Time.*

13729449 **RETAIN THIS COPY FOR YOUR RECORD**

From: Please print and print name of Sender's Office  
 Date: **FILL IN** Sender's Office: **1788-8014-4**  
 Sender's Name: **FILL IN** Phone: **(312) 856 8700**

Company: **TETRA TECH EM INC**

Address: **200 E RANDOLPH ST STE 4700**

City: **CHICAGO** State: **IL** ZIP: **60601**

Your nearest FedEx Reference Number (if known) **FILL IN**

To: **FILL IN**

Recipient's Name: **FILL IN** Phone: **FILL IN**

Company: **FILL IN**

Address: **FILL IN**

City: **FILL IN** State: **FILL IN** ZIP: **FILL IN**

For HOLD at FedEx Location check here  
 Hold Warehouse  Hold Station  
 Hold Office  Hold Home

For WEEKEND Delivery check here  
 Saturday Delivery  Sunday Delivery

Express Package Service Packages up to 65 lb. **FILL IN**

Express Freight Service Packages over 65 lb. **FILL IN**

Page 1 of 1 Pages Two completed and signed copies of this Declaration must be handed to the operator.

**TRANSPORT DETAILS**  
 This document is subject to the conditions prescribed for this service. **Chicago**  
 Point of Origin: **Chicago**  
 Point of Destination: **City sending sample to**

**WARNING**  
 Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a co-ordinator, a forwarder or an IATA cargo agent.

Shipment type: **NON-RADIOACTIVE / RADIOACTIVE**

DANGEROUS GOODS IDENTIFICATION					Quantity and Type of Packaging	Packing Instructions	Authorization
Proper Shipping Name	Class or Division	UN or I.D. No.	Packing Group	Subsidiary Risk			
Flammable liquid, n.o.s.	3	VN 1993	III	-	4 glass jars in a 2A2 steel drum Net Quantity = 4L	309	A3 USG-14

Additional Handling Information: **NAERG # 128 Attached.**

Prepared for AIR TRANSPORT according to (Customer MUST check one)  
 ICAO  ICAO / IATA

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name and are classified, packaged, marked, and labeled/retarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

Emergency Telephone Number (required for U.S. Origin or Destination Shipments): **FILL IN**

Name/Title of Signatory: **ME, Environmental Scientist**  
 Place and Date: **200 E Randolph, Chicago, IL 01/28/00**  
 Signature: **ME**

Tracking Number: **813350883058 0204**

Signature Release Unavailable

IF ACCEPTABLE FOR PASSENGER AIRCRAFT THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS OR TREATMENT.

FIGURE 3

NAERG EMERGENCY RESPONSE INFORMATION  
FOR FLAMMABLE LIQUIDS, N.O.S.

**GUIDE 128-129** **HAZARD INFORMATION** **GUIDE 128-129**

**POTENTIAL HAZARDS**

**SPILLS OR EXPLOSIONS**

- **FLAMMABLE** Will be easily ignited by open sparks or flames.
- Vapor may form explosive mixtures with air.
- Vapor may be heavier than air and collect in low areas.
- Most vapors are heavier than air. They will spread along ground and collect in low areas.
- Vapor may be heavier than air and collect in low areas.
- Some may irritate (Pneumonitis and related or aggravated by the high relative humidity of the air).
- Some vapors may be irritating to the eyes.
- Many vapors are heavier than water.
- Substances may be harmful if inhaled.

**HEALTH**

- May be irritating to the eyes.
- May be irritating to the respiratory system.
- May be irritating to the skin.
- Runoff of the liquid or vapor may be irritating.

**PUBLIC SAFETY**

- Call Emergency Response Telephone Number on Shipping Paper first if Shipping Paper not available or no answer, refer to appropriate telephone number listed on the inside back cover.
- Avoid ignition sources (e.g., open flames, electrical equipment).
- Keep away from hot surfaces.
- Do not breathe vapor.
- Do not get liquid on skin or clothing.

**PROTECTIVE CLOTHING**

- Wear protective gloves and clothing when handling.
- Wear eye protection when handling.

**EVACUATION**

- Consider evacuation if necessary.
- Evacuate if necessary.
- Evacuate if necessary.

**EMERGENCY RESPONSE**

**CAUTION:** All these products have a very low flash point. Use of water spray when fighting fires may be ineffective.

**Spill Free**

- Do not use water spray to regularize.
- Large Spills
- Wear eye protection.
- Do not use water spray.
- Move containers from fire area if safe to do so.

**Fire Fighting Tanks or Car-Trailer Loads**

- Fight fire from upwind.
- Do not use water spray to regularize.
- Do not use water spray to regularize.
- Do not use water spray to regularize.

**SPILL ON LEAK**

- Eliminate all ignition sources.
- Do not use water spray to regularize.
- Do not use water spray to regularize.
- Do not use water spray to regularize.

**Large Spills**

- Do not use water spray to regularize.
- Do not use water spray to regularize.
- Do not use water spray to regularize.

**FIRST AID**

- Move victim to fresh air.
- Do not use water spray to regularize.
- Do not use water spray to regularize.

Source: DOT and others. 1996.

**SOP APPROVAL FORM**

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

**RECORDING OF NOTES IN FIELD LOGBOOK**

**SOP NO. 024**

**REVISION NO. 1**

Last Reviewed: November 1999

*K. Miesing*

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Quality Assurance Approved

*May 18, 1993*

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Date

## **1.0 BACKGROUND**

The field logbook should contain detailed records of all the field activities, interviews of people, and observations of conditions at a site. Entries should be described in as much detail as possible, so that personnel can accurately reconstruct the activities and events which have taken place during field assignments. Field logbooks are considered accountable documents in enforcement proceedings and may be subject to review. Therefore, the entries in the logbook must be accurate, detailed, and reflect the importance of the field events.

### **1.1 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide guidance to ensure that logbook documentation for any field activity is correct, complete, and adequate. Logbooks are used for identifying, locating, labeling, and tracking samples. A logbook should document any deviations from the project approach, work plans, quality assurance project plans, health and safety plans, sampling plans, and any changes in project personnel. They also serve as documentation of any photographs taken during the course of the project. In addition, the data recorded in the logbook may assist in the interpretation of analytical results. A complete and accurate logbook also aids in maintaining good quality control. Quality control is enhanced by the proper documentation of all observations, activities, and decisions.

### **1.2 SCOPE**

This SOP establishes the general requirements and procedures for recording notes in the field logbook.

### **1.3 DEFINITIONS**

None

### **1.4 REFERENCES**

Compton, R.R. 1985. *Geology in the Field*. John Wiley and Sons. New York, N.Y.

## **1.5 REQUIREMENTS AND RESOURCES**

The following items are required for field notation:

- Field logbooks
- Ballpoint pens with permanent ink
- 6-inch ruler (optional)

Field logbooks should be bound (sewn) with water resistant and acid-proof covers; they should have preprinted lines and wide columns. They should be approximately 7 1/2 by 4 1/2 inches or 8 1/2 by 11 inches in size. Loose-leaf sheets are not acceptable for field notes. If notes are taken on loose paper, they must be transcribed as soon as possible into a regular field logbook by the same person who took the notes.

Logbooks can be obtained through the Document Control Administrator (DCA) for each office. The DCA will have assigned each logbook an identification number. The DCA will make sure the pages in the logbooks are preprinted with consecutive numbers or are consecutively numbered by hand. If the numbers are written by hand, then numbers should be circled so that they are not confused with data.

## **2.0 PROCEDURES**

The following subsections provide general guidelines and formatting requirements for field logbooks and detailed procedures for completing field logbooks.

### **2.1 GENERAL GUIDELINES**

- A separate field logbook must be maintained for each project. If a site consists of multiple subsites, designate a separate logbook for each subsite. For special tasks, such as periodic well water-level measurements, data from multiple subsites may be entered into one logbook which contains only one type of information.
- All logbooks must be bound and contain consecutively numbered pages.
- No pages can be removed from the logbook for any purpose.

- All field activities, meetings, photographs, and names of personnel must be recorded in the site logbook.
- All logbooks pertaining to a site or subsite should be assigned a serial number based on the date the logbook is issued to the project manager. The first logbook should be assigned number 1, the next logbook issued assigned number 2, and so on. The project manager is to maintain a record of all logbooks issued under the project.
- All information must be entered with a ballpoint pen with waterproof ink. Do not use pens with "wet ink," because the ink may wash out if the paper gets wet. Pencils are not permissible for field notes because information can be erased. The entries should be written dark enough so that the logbook can be easily photocopied.
- Do not enter information in the logbook that is not related to the project. The language used in the logbook should be factual and objective.
- Begin a new page for each day's notes.
- Write notes on every line of the logbook. If a subject changes and an additional blank space is necessary to make the new subject title stand out, skip one line before beginning the new subject. Do not skip any pages or parts of pages unless a day's activity ends in the middle of a page.
- Draw a diagonal line on any blank spaces of four lines or more to prevent unauthorized entries.

## **2.2 LOGBOOK FORMAT**

The layout and organization of each field logbook should be consistent with other field logbooks.

Guidelines for the cover, spine, and internal pagination are discussed below.

### **2.2.1 FORMAT OF FIELD LOGBOOK COVER AND SPINE**

Write the following information in clear capital letters on the front cover of each logbook.

- Logbook identification number (assigned by the DCA)
- The serial number of the logbook (assigned by the project manager)
- Name of the site, city, and state

- Name of subsite if applicable
- Type of activity
- Beginning and ending dates of activities entered into the logbook
- "Tetra Tech EM Inc." City and State
- "REWARD IF FOUND"

Some of the information listed above, such as the list of activities and ending dates, should be entered after the entire logbook has been filled or after it has been decided that the remaining blank pages in the logbook will not be filled.

The spine of the logbook should contain an abbreviated version of the information on the cover. For example: "1, Col. Ave., Hastings, 5/88 - 8/88."

### **2.2.2 First Page of the Field Logbook**

Spaces are usually provided on the inside front cover (or the opening page in some logbooks), for the company name ("Tetra Tech EM Inc."), address, and telephone number. If preprinted spaces for this information are not provided in the logbook, write the information on the first available page.

### **2.3 ENTERING INFORMATION IN THE LOGBOOK**

Enter the following information at the beginning of each day or whenever warranted during the course of a day:

- Date
- Starting time
- Specific location
- General weather conditions and approximate temperature

- Names of personnel present at the site. Note the affiliation(s) and designation(s) of all personnel.
- Equipment calibration and equipment models used.
- Changes in instructions or activities at the site.
- Levels of personal protective clothing and equipment.
- A general title of the first task undertaken (for example, well installation at MW-11, decon at borehole BH-11, groundwater sampling at MW-11).
- Provide an approximate scale for all diagrams. If this can't be done, write "not to scale" on the diagram. Indicate the north direction on all maps and cross-sections. Label features on each diagram.
- Corrections should be made by drawing a single line through the entry being corrected. Initial and date any corrections made in the logbook.
- The person recording notes is to initial each page after the last entry. No information will be entered in the area following these initials.
- At the end of the day, the person recording notes is to sign and date the bottom of the last page. Indicate the end of the work day by writing "Left site at (time)." A diagonal line will be drawn across any blank space to the bottom of the page.

The following information should be recorded in the logbook after taking a photograph:

- Time, date, location, direction, and if appropriate, weather conditions
- Description of the subject photographed and the reason for taking the picture
- Sequential number of the photograph and the film roll number (if applicable)
- Name of the photographer

The following information should be entered into the logbook when taking samples:

- Location description
- Names of samplers
- Collection time
- Designation of samples as a grab or composite sample
- Type of sample (water, sediment, soil gas, etc.)

- On-site measurement data (pH, temperature, specific conductivity)
- Field observations (odors, colors, weather, etc.)
- Preliminary sample description
- Type of preservative used
- Instrument readings

#### **2.4 PRECAUTIONS**

Custody of field logbooks must be maintained at all times. Field personnel must keep the logbooks in a secure place (locked car, trailer, or field office) when the logbook is not in personal possession. Logbooks are official project documents and must be treated as such.

**ATTACHMENT 2**

**AIR TOXICS LTD.  
LABORATORY STANDARD OPERATING PROCEDURES**

- SOP No. 26, "Analysis of Pesticides and PCBs Collected on PUF Cartridges; EPA Methods TO-4A/TO-10A and Modified EPA SW-846 Methods 8081A and 8082" (19 Sheets)
- SOP No. 50, "Receiving, Login, and Tracking of Samples" (14 Sheets)

Control Copy #: \_\_\_\_\_

@ Air Toxics Limited  
STANDARD OPERATING PROCEDURE

**ANALYSIS OF PESTICIDES AND PCBs COLLECTED ON PUF  
CARTRIDGES**

**EPA METHODS TO-4A/TO-10A AND MODIFIED EPA SW-846  
METHODS 8081A AND 8082**

SOP #26

Revision Date: 05/25/00  
Revision #: 3  
Reason for Revision: External Use

Updated By: \_\_\_\_\_

Signature	Print	Title	Date
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Technical Review: \_\_\_\_\_

Signature	Print	Title	Date
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QA Review: \_\_\_\_\_

Signature	Print	Title	Date
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Laboratory Director: \_\_\_\_\_

Linda L. Freeman			Date
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**1.0 SCOPE AND APPLICATION**

To provide a procedural guide for the application of EPA Methods TO-4/TO-10A (High Volume Sampling) to the analysis of pesticides and Aroclor PCBs in ambient air collected on PUF (polyurethane foam) cartridges.

**2.0 METHOD SUMMARY**

**2.1 Description**

Adsorbent PUF cartridges are cleaned using solvents and vacuum dried. Cartridges are sent to the field wrapped tightly in aluminum foil to prevent degradation by UV light. Following sampling, the filters and cartridges are subjected to soxhlet extraction with methylene chloride, the solvent is switched to hexane, and the extract concentrated. Analysis is performed using a GC/ECD (Electron Capture Detector). The target compound list and detection limits are noted in *Appendix A*.

**2.2 Deviations**

Modifications to EPA Method TO-4A used to carry out the analyses of air samples are listed in Table 1.

Table 1. Summary of Method Modifications (*Revised, see attached fax dated Mar 28, '02*)

Requirement	TO-4A/TO-10A	ATL Modifications
Extraction solvent	10% diethylether in hexane	Methylene chloride, exchanging to hexane during the condensation step
Target list	Table 1. TO-4A method	ATL target list is a subset of Table 1
Detector	Allows use of ECD, FPD/NPD, Nitrogen Phosphorus detector, HPLC/UV, MS	Use ECD detection
Detection Limit (DL)	Dependent on method of detection; ECD section suggests capability of 0.001 µg to 0.050 µg.	DL for single component analytes generally 0.10 µg; DL for multicomponent analytes 1.0 µg
Extract cleanup	Clean-up options listed in section 12.2	Use sulfuric acid cleanup for PCB only analysis.
Glassware cleaning	Cleaning series consisting of water/detergent, deionized water (DI H <sub>2</sub> O), acetone, hexane, DI H <sub>2</sub> O, oven at 500°C	Cleaning consists of rinsing with last solvent, water/detergent, DI H <sub>2</sub> O, air dry, triple rinse with methylene chloride. (Once solvent is exchanged to hexane, all glassware used is triple rinsed with hexane.)
Filter cleaning	Bake at 400°C for 5 hours	No cleaning performed; extract one from lot and certify as clean

Table 1. Summary of Method Modifications (continued) *(Revised; see attached fax dated Mar 25)*

Requirement	TO-4A/TO-10A	ATL Modifications
PUF cleaning	Acetone for 16 hrs	Methylene chloride - four times for four hours each
Media certification	Extract one cartridge; criteria is <10 ng/cartridge for single analyte; <100 ng total/cartridge for multicomponent	Condense final solvent extract from cleaning procedure; target analytes must be less than the Detection Limit
Media hold time	30 days from certification	After 90 days, re-certify media
Reagent Blank	Set up extraction system without filter/PUF; reflux with solvent	No reagent blank extracted. Reagent lots certified as acceptable prior to use.
Condensation	Use KD, Snyder column, and water bath.	Use flat bottom flask, Snyder column, and hot plate.
Field Blank and Field Spike	(Actually a trip blank and trip spike) required for each sampling episode. No air is drawn through either cartridge and media remains in sealed container. "Field" spike is spiked at the laboratory prior to shipment.	Client can request these QC samples at the time of shipment.
Blank criteria	No target hits in reagent blank, field blank, or lab blank >10 ng for single analyte; >100 ng for multicomponent analytes.	No target hits for the Lab Blank above the Detection Limit (DL)
Initial Calibration	3 point curve; low point near analytical DL; %RSD < 20%	5 point curve; low point is the detection limit; %RSD < 20%
Initial Calibration Verification (ICV)	Second source standard analyzed after ICAL; recovery 85-115%	Recovery limits are 80-120%.
LCS requirement	No LCS requirement	LCS is extracted with each batch; recovery limits are based on laboratory control limits.
PCB Quantitation	Requires a minimum of 5 peaks	Use 4 peaks for quantitation.
Surrogate recovery criteria	60-120%	Use laboratory control limits.
Identification	Confirm all results with secondary column.	Confirmation performed for all pesticides; generally not used for PCBs.
Method Detection Limit	No guidance provided	Extract 7 low-level spikes following 40 CFR Part 136B.

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AIRTOXICS LTD

TEL:916 985 1020

P. 002/002

Approved:

Revision: Initials,

Reviewed: Date Table 1. Summary of Method Modifications

SL 3/29/02

Te Proj. Mgr.

KLW/MS  
4/13/02

Te QA Office

Requirement	TO-4A/TO-10A	ATL Modifications
Target list	Table 1 TO-4A Method	ATL target list is a subset of Table 1
Detector	Allows use of ECD, FPD/NPD, Nitrogen Phosphorus detector, HPLC/UV, MS	Use ECD detector
Reporting Limit (RL)	Dependent on method of detection; ECD section suggests capability of 0.001 µg to 0.050 µg.	RL for single component analytes generally 0.10 µg; RL for multicomponent analytes 1.0 µg
Reagent Blank	Set up extraction system without filter/PUF; reflux with solvent	No Reagent Blank extracted. Reagent lots certified as acceptable prior to use.
Field Blank and Field Spike	(Actually a Trip Blank and Trip Spike) required for each sampling episode. No air is drawn through either cartridge and media remains in sealed container. "Field" Spike is spiked at the laboratory prior to shipment.	Client can request these special QC samples at the time of shipment.
Blank criteria	No target hits in the Reagent Blank, Field Blank, or Lab Blank >10 ng for single analyte; >100 ng for multicomponent analytes.	No target hits in the Lab Blank Above the Reporting Limit (RL)
Initial Calibration	3 point curve; low point near analytical RL; % RSD < 20%	3 point curve; low point is the Reporting Limit; % RSD < 20 %
Initial Calibration Verification (ICV)	Second source standard analyzed after ICAL; recovery 85-115 %	Recovery limits are 80-120 %.
LCS requirement	No LCS requirement	An LCS is extracted with each batch; Recovery limits are 65-125 %R
PCB Quantitation	Requires a minimum of 5 peaks	Use 4 peaks for quantitation.
Identification	Confirm all results with secondary column.	Confirmation performed for all Pesticides; generally not used for PCBs.
Method Detection Limit	No guidance provided	Extract at least 7 low-level spikes following 40 CFR Part 136B.

KLW 4-1-02  
EPA OSC

### **3.0 HEALTH AND SAFETY**

Normal laboratory safety precautions must be used when extracting samples, preparing standards from neat materials and analyzing samples. Appropriate eye wear, gloves and lab coat should be worn when handling any chemical used in this method. All manipulation of standards, solvents, and acid and basic solutions should be done with the utmost care in the hood. MSDS for each chemical should be consulted for specific dangers and precautions. The procedures described in this SOP are designed for skilled chemists trained in the safe operation of GC/ECD and sample handling.

### **4.0 SAMPLE PRESERVATION, HANDLING, AND STORAGE**

Samples are shipped to the laboratory in 4°C ice chests. Upon receipt, the cartridges are stored in a refrigerator (4°C) until extraction. Extraction must be performed within one week of sample collection. Sample extracts are stored in a separate refrigerator (4°C) until analysis. Analysis must occur within 40 days of extraction.

### **5.0 INTERFERENCES AND POTENTIAL PROBLEMS**

A common interference in the pesticide procedure is from phthalate esters. Phthalate esters are introduced into the system through contaminated glassware and/or when the extraction solvent contacts plastic materials. All glassware is thoroughly cleaned to minimize phthalate contamination and there is limited use of plastic materials during the extraction procedure.

Carryover from a high level sample can also create false positive results. The system is demonstrated to be clean by analysis of a hexane blank and any sample suspected of carryover contamination is reanalyzed to verify results. Scientist judgment should always be used to determine when reanalysis is necessary but a typical guideline would be whenever the sample concentration exceeds 5 times the upper calibration limit.

### **6.0 EQUIPMENT AND MATERIALS**

#### **6.1 List of Equipment**

- GC/dual ECD system equipped with a temperature programmable oven suitable for splitless injection
- Capillary column - 30m X 0.32mm with 0.50 µm film thickness. (RTX-CL Pesticides; Primary)
- Capillary column - 30m X 0.32mm with 0.25 µm film thickness. (RTX-CL Pesticides II; Secondary)

- HP Chemstation and Target Thru-put System Chromatography Software
- HP 7673 Automatic Liquid Injection System

## 7.0 REAGENTS AND SUPPLIES

- TO-4 Glass Cartridges (Graseby/General Metal Works, Village of Cleves, OH)
- QMA quartz microfiber filters 10.16 cm (Whatman)
- PUF plugs (Graseby/General Metal Works, Village of Cleves, OH)
- Aluminum foil
- Soxhlet extractors capable of extracting filters and adsorbent cartridges, 1.0 L flask and condenser
- Glass funnels
- Filter paper #41
- Zymark Automated Liquid Concentrator equipped with 1 mL end point
- Polyethylene gloves for handling filters and cartridges
- Vacuum oven
- Methylene Chloride - Reagent Grade
- DI Water – Barnstead e-pure (for rinsing glassware only)
- Hexane - Reagent Grade
- Methanol - Reagent Grade
- Sodium Sulfate, anhydrous
- Vortex Supermixer II (Labcraft)

## 8.0 PROCEDURES

### 8.1 Preparation of Sampling Media

PUF inserts are generally 3-inch cylindrical plugs. The PUF material is initially cleaned by soxhlet extraction with methylene chloride for a minimum of 18 hours at approximately 4 cycles per hour. The extracted foam is dried for two to four hours at approximately 120°C in a vacuum oven. To prepare the media for shipment, the PUF is placed into the glass-sampling cartridge using polyester gloves. The cartridge is wrapped with aluminum foil followed by bubble wrap and then placed into a sealed plastic bag. To certify the batch, the final (from the third six hour period) solvent rinse is solvent exchanged to hexane and blown down to 1.0 mL and submitted for analysis. The batch is certified if all compounds are less than the required detection limit. The PUF inserts that are not immediately being used in assembly of cartridges are placed in a glass jar or wrapped in aluminum foil and kept at ambient temperature until they are needed. The jar is labeled with the above batch ID. PUF inserts that are not used within 3 months of preparation will be recertified as clean prior to use.

## 8.2 Sample Extraction

### 8.2.1 *Glassware Preparation*

All glassware used in the extraction is prepared as follows: wash with warm soapy water, triple rinse with tap water, triple rinse with de-ionized water, then set to dry. All glassware must be completely dry prior to use. Triple rinse with methylene chloride. If glassware is not dry, a triple methanol rinse followed by a triple methylene chloride rinse may be used. The turbo vap vials must also be triple rinsed with hexane.

### 8.2.2 *Funnels*

Set up a funnel for each sample lined with #41 filter paper in the fume hood using ring stands. Add a small amount of sodium sulfate and triple rinse with hexane.

### 8.2.3 *Extraction Procedure*

- Fill each round bottom flask with approximately 700 mL methylene chloride.
- Add several methylene chloride rinsed PTFE boiling chips to each flask and cover with foil.
- Triple rinse each soxhlet extraction body with methylene chloride, place on flat bottom flask.
- Unwrap each sample and place the sample cartridge and its quartz filter into labeled extraction soxhlets. Include a blank and spike.
- Bring chiller to set point temperature of 10°C.
- Add 500 µL of surrogate spiking solution to each soxhlet, including the blank and spike. The surrogate spiking solution is stored at -18±5°C and expires in 6 months.
- Add 500 µl of appropriate spiking solution to the spike sample (LCS). For the standard pesticide list, the spike is prepared from a purchased mix from Restek or equivalent.
- Carefully place the condenser on each soxhlet.
- Turn on each heating mantle to a set point of 5.5. The orange light flashing indicates the mantle is heating.
- Extract samples for a minimum of 18 hours, but not more than 24 hours, maintaining a chiller temperature of less than 15°C.

#### 8.2.4 Concentration

- Triple rinse each Snyder column with methylene chloride into the sample's respective flat bottom flask. Place the column on each flask.
- Place the samples on hot plates using a temperature setting between 5 and 6. Samples will begin to boil and columns will chatter.
- Allow to boil until sample volume is NEAR dry. Do not allow the samples to go to dryness. This will cause poor recoveries and loss of sample.
- Add approximately 100 mL of Hexane through the Synder column and return to heating mantle. Again, allow to boil to near dryness and add another 100 mL Hexane through the Synder column. Repeat for a third addition.
- Remove sample from mantle, let cool, and rinse Synder column with hexane into round bottom flask. Remove Synder column and pour sample through a funnel lined with #41 filter paper and sodium sulfate, collecting sample extract into turbo vap vial. Rinse round bottom flask two more times with Hexane through funnel. After all solvent has drained out of the funnel, triple rinse with Hexane into the turbo vap vial.

#### 8.2.5 Nitrogen Evaporation

- Evaporate filtered sample to just below 1.0 mL using nitrogen in the turbo vap.
- Remove sample from turbo vap and adjust the volume to exactly 1.0 mL using a clean Pasteur pipette and clean hexane.
- Vortex the sample for five seconds.
- Using a clean Pasteur pipette, transfer the 1.0 mL sample to a labeled coarse-threaded screw top vial and cap each sample.
- Store vials in the refrigerator and fill out all appropriate documents.

#### 8.2.6 PCB Cleanup Procedures

If the target list includes Aroclors only, a sulfuric acid cleanup is used. **Do not do a sulfuric acid cleanup on pesticide samples, as target analytes will degrade.** Prior to storing sample extract vials in the refrigerator, add 100  $\mu$ L concentrated sulfuric acid to each blank, spike and sample. Shake vial on the vortex mixer for 5-10 seconds. Centrifuge samples for 3-5 minutes. Store samples with sulfuric acid. When aliquots are analyzed on the GC/ECD, be

careful to analyze hexane fraction only leaving the sulfuric acid layer (bottom layer) in the vial.

### 8.3 Sample Analysis

Analysis is carried out on a HP 5890 GC/ dual ECD system. Second column confirmation is used to positively identify pesticide results. Results are reported from the "Primary Column" analysis. However, if the results are >40% difference between the two columns, the higher result will be reported as specified in Method 8081A. Any results reported from the "Confirmation Column" analysis will be noted on the sample report.

### 8.4 Calibration

Calibration is accomplished through an external standard technique. Once the data file is collected and stored it is automatically processed and reported using the Target Thruput Software. The working range of the analytical system is defined by the range of concentrations bracketed by the five-point calibration. The lowest level of the curve represents the detection limit and the highest level represents maximum concentration possible without dilution. The concentration of the field samples must be bracketed by this range. Any sample that exceeds the calibration range of the curve will be diluted with hexane until its concentration approximates the mid-point of the calibration range.

#### 8.4.1 *Initial Calibration*

Standards are blended from commercially blended and certified stock solution to cover a range from 0.10 µg/mL up to 2.0 µg/mL and stored in the standards freezer. One µL of each standard is analyzed. Tabulation of the peak area vs. concentration (µg/mL) is performed to generate a response factor (RF) for each level using the equation:

$$RF = AX/CX$$

Where: AX = Area of the peak for the compound measured  
CX = Concentration of the compound measured (µg/mL)

Aroclor, technical Chlordane, and Toxaphene calibrations are bracket for the linear range from 1.0 µg/mL to approximately 10 µg/mL. A change in detector sensitivity may result in a change in the linear range. Standards are stored in the standards freezer. Non-overlapping Aroclors can be calibrated concurrently, (e.g., Aroclor 1016 and Aroclor 1260). One µL of each standard is analyzed. A minimum of four peaks are used to characterize technical Chlordane, Toxaphene and each Aroclor. In general, only Arochlor 1016/1260 mix is used to determine instrument linearity.

A single mid-level standard for each remaining Aroclor is analyzed unless the project specifically requests calibration on an Aroclor by Aroclor basis. Tabulation of the peak area sum vs. concentration ( $\mu\text{g/mL}$ ) is performed to generate a RF for each level using the equation:

$$\text{RF} = A_X/C_X$$

Where:  $A_X$  = Sum of peak Areas of the 4 characteristic peaks for the compound measured

$C_X$  = Concentration of the compound measured ( $\mu\text{g/mL}$ )

The average RF for each compound is measured and tabulated. The percent relative standard deviation (% RSD) is also measured for the five point. Linearity through the origin is assumed if the variability of the RF is within  $\pm 20\%$  RSD for the target compounds. The average RFs from the calibration curve are used to calculate results. If the average RF is greater than 20% then linear regression is used.

#### 8.5 Independent Source Check Standard

After analysis of the initial curve, an independent source check is analyzed at the mid-level of the calibration curve. See standard prep logbook for preparation details. The independent source is prepared from stock from either a different vendor or a different lot of the same vendor. Recovery must be within 80 to 120% for all compounds.

### 9.0 ***CALCULATIONS***

The initial calibration response factor (RF) for each compound is used to calculate sample results using the area counts ( $A_X$ ) of each target species:

$$A_X/\text{RF} = \mu\text{g/mL}$$

$$(\mu\text{g/mL}) \times (1.0 \text{ mL final volume/sample}) = \mu\text{g/sample}$$

### 10.0 ***QUALITY CONTROL***

#### 10.1 Initial Calibration Curve

The percent relative standard deviation (%RSD) for the initial curve must be less than or equal to 20%. If the %RSD is greater than 20% then linearity through the origin is not assumed and the analyte uses an optional

linear regression calibration. A second alternative is to use the mean %RSD criterion as outlined in SW846 Method 8000B Section 7.5. If the mean %RSD criterion is used, the compound(s) failing the 20% criterion requirement are noted.

#### 10.2 Independent Source Check Standard

The independent source standard prepared at the mid-point of the multilevel calibration is analyzed immediately after the initial calibration and each time the instrument is calibrated. The independent source should agree with the primary standard within 80% to 120% recovery.

#### 10.3 Continuing Calibration Verification

The continuing calibration verification (CCV) is analyzed daily prior to sample analysis. The response for each analyte should be within 15% of the initial calibration curve. If greater variability is observed, the average of all of the target analytes in the calibration table (not the average of those required for any particular project) is calculated. If the average meets the 15% criteria then the standard verification has been met. If the average deviation does not meet the 15% criteria then a new initial calibration curve may need to be analyzed and/or fresh standards may need to be prepared. If secondary confirmation is required, the CCV criteria must be met for both columns.

The CCV is analyzed every 10 samples and at the end of the analysis sequence (i.e., end of run drift check). All high, low, and multi-component standards are alternated. However, the analyst must verify that a pesticide or PCB standard is analyzed at the minimum of every 12 hours. The recovery of an individual component or the average of all the target components must be within 15% for these checks. Should the recovery fail to meet these limits on both the primary and confirmation column, all samples analyzed since the last valid check will be reanalyzed. However, if the standard analyzed is recovering high then samples do not need to be reanalyzed. The CCV is reported with "Q" flags to note high recoveries. If the standard analyzed is recovering low then a subset of samples is reanalyzed to determine when the system became out of control. All affected samples are reanalyzed.

#### 10.4 Laboratory Control Spike (LCS)

Either a 1.0 µg pesticide LCS or a 10 µg PCB LCS is spiked depending on the requested target list and extracted with each set of 20 samples. All target compounds are spiked prior to extraction from a source other than the calibration standard. LCS recovery limits are generated using in-house

control charts and are generated quarterly. Recovery limits for PCB 1016/1242 are 57 to 116% and PCB 1260 are 60 to 123% recovery. Since it is not possible to re-extract PUF samples, compounds outside the method limits will be qualified with a "Q" flag.

#### 10.5 Surrogates

Surrogates are spiked into all samples and blanks prior to extraction. Surrogate limits are given below. Surrogate limits are generated using in-house control charts and are evaluated quarterly.

<u>Surrogate</u>	<u>% Recovery</u>
Tetrachloro-m-xylene	47 - 134
Decachlorobiphenyl	47 - 159

#### 10.6 Method Blanks

A blank PUF cartridge will be extracted with each set of 20 samples extracted. Since it is not possible to re-extract PUF samples, compounds detected in the method blank will be qualified with a "B" flag. The "B" flag indicates that the compound was detected in the method blank and blank subtraction was not performed.

#### 10.7 Pesticide Breakdown Evaluation Standard

Each day prior to Pesticide analysis, a standard containing Endrin and p,p'-DDT is analyzed to determine breakdown. The standard is prepared as indicated standard prep logbook at 0.8 ug/mL. Breakdown on either column must not exceed 15% for each compound and is determined as follows:

$$\text{Endrin Brkdwn} = \frac{\text{Area Endrin Aldehyde} + \text{Area Endrin Ketone}}{\text{Area Endrin} + \text{Area Endrin Aldehyde} + \text{Area Endrin Ketone}} \times 100$$

$$\text{DDT Brkdwn} = \frac{\text{Area p,p'-DDE} + \text{Area p,p'-DDD}}{\text{Area p,p'-DDT} + \text{Area p,p'-DDE} + \text{Area p,p'-DDD}} \times 100$$

#### 10.8 Analytical Sequence

The analytical sequence begins with the Pesticide Breakdown Evaluation Standard followed immediately by the CCV. Following standard analysis, a hexane blank and/or a method blank is run followed by the analysis of field samples and one LCS per extraction batch. The CCV is analyzed every 10 samples and as an end check following the last sample of the day.

CCV recoveries should be  $\pm 15\%$  of the expected value. Sample analysis cannot proceed if either the breakdown check standard, CCV or hexane blank analysis fails to meet acceptance criteria. If the end check standard fails re-analysis of the standard is performed. If the failure is confirmed then maintenance and recalibration is required. The analytical sequence is repeated for each 12-hour clock.

#### 10.9 Method Detection Limit (MDL) Studies

The limit of detection is determined in accordance with the guidelines of Appendix B, Part 136 of the Federal Register 40 CFR. The 99% confidence level is used to determine MDLs. An MDL study is performed annually. Refer to SOP #39 for details.

#### 10.10 Method Precision

Laboratory sample duplicates are analyzed at a frequency of 10%. The relative percent difference (RPD) between duplicates must be  $\pm 20\%$  or reanalysis is performed.

#### 10.11 Retention Time (RT) Window Studies

A 72 hour retention time window must be established for every set of analytical columns and instruments. The lab will calculate new 72 hour windows whenever a new column is installed. Windows are established by making three injections of a mid-level calibration standard over a 72 hour period. The standard deviation of these retention times is calculated. This standard deviation is multiplied by 3 to calculate the window. The window is applied to the daily check's RT. The windows are used for advisory purposes and the discretion of the analyst must weight heavily on all compound identifications.

The RT window is defined as following:

- a. for a compound eluting  $< 20$  min, the larger of 0.05 min and the RT study value is used,
- b. for a compound eluting  $> 20$  min, the larger of 0.07 min and the RT study value is used.

### **11.0 WASTE DISPOSAL**

All samples, standards, and blank solutions are disposed of in the liquid organic waste drum located in the Hazardous Storage Area.

## **12.0 CORRECTIVE ACTION PROCEDURES**

A request for corrective action (CAR) is initiated any time there are deviations from either the ATL SOPs or client-prescribed QC protocols, or in any instance where sample results may be adversely affected. Corrective action procedures are documented in ATL's Corrective Actions Procedure SOP#61.

## **13.0 DATA REVIEW**

### **13.1 Initial Data Review**

As the analytical sequence is run throughout the day, the data is reviewed by the bench chemist using the following eight steps:

- 13.1.1 Check for any project-specific requirements.
- 13.1.2 Verify holding time.
- 13.1.3 Verify the CCV, LCS, and end check.
- 13.1.4 Verify method blank has no hits above detection limit.
- 13.1.5 Verify sample results.
  - a) Verify that RT and surrogates recoveries are acceptable.
  - b) Verify that the correct amount of sample was analyzed.
  - c) Verify the reasonableness of the automated peak integration.
  - d) Verify that the result concentrations are within linear range of calibration curve (upper 50% for dilutions.)
- 13.1.6 Initial and date raw data and/or logbook entry to indicate that the data is acceptable.
- 13.1.7 Apply the appropriate data flags.
- 13.1.8 Describe unusual events on data review sheet.

#### *Notes:*

- *Preparation and review of laboratory narrative are carried out as explained in the SOP # 45.*
- *Compilation of data package by the analyst/scientist is carried out as described in the data review SOP #22.*

### **13.2 Secondary Data Review**

The team leader or a QA-approved peer performs a secondary technical data review on 100% reports. This review follows all the steps mentioned in the initial data review (Section 13.1). When the chemist is signed off as fully trained, this step is not necessary unless required by a specific project.

### 13.3 Tertiary (QA) Data Review

A thorough tertiary data review is performed by the QA department on a randomly chosen 10% of the final data packages. The QA review entails verification that project and QC requirements are met. Failure to meet QC and/or project requirements results in a corrective action request and documentation. Dilution factors, analyte retention times, peak integration areas, concentration calculations, unit conversions, and detection limits are also checked. Field and trip blanks are checked and trends are observed.

### 13.4 Quaternary Data Review

Some clients requests that 100% of their final data packages undergo a fourth technical review. The quaternary reviews in this case are performed by the team leader, QA-approved peer, or QA personnel.

## 14.0 **INSTRUMENT MAINTENANCE**

Instruments are monitored on a daily basis by the bench analyst for any potential failure. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real time feedback to the analyst on the condition of the instruments. Routine maintenance includes:

### 14.1 Gas Chromatograph

Basic maintenance includes the following: (*Every 6 months or more frequently if needed*)

14.1.1 Clip 3 feet off the front end of the guard column or capillary column, and if necessary, the backend as well.

14.1.2 Replace the injection port liner and uniliner inlet liner. The liner is replaced by removing the inlet cap using a wrench and releasing the liner from the inlet body using a pair of tweezers. Care should be taken not to get fingerprints on any inside surface.

14.1.3 Change septa on the GC approximately every 100 injections or whenever the front-end maintenance is performed. Always use Supelco Thermogreen septa and take care not to leave fingerprints on any inside heated surface. Wear a pair of white cotton gloves or use tweezers to handle the septa. Lower the oven temperature to 40°C. Remove the inlet cap with a wrench, remove the old septa with a pair of tweezers and insert the new septa.

14.1.4 The column is replaced when chromatography peak shape or resolution degrades. Similarly, if the column bleed profile rises with age then the column needs replacing. Use new Restek Vespel Graphite ferrules each time and clip off approximately 1" of column

after inserting it through the ferrule. This will remove any graphite particles that may have scrapped off into the column. Tighten the column nut and ferrule finger tight and one-quarter turn with a wrench. Tightening any more only crushes the ferrule and may damage the column.

14.1.5 The bench analyst will document any routine or major maintenance in the bound instrument logbook assigned to each instrument. The date of the maintenance, what work was performed and analyst initials are included.

## **15.0 DELIVERABLES**

Data reporting packages are prepared as described in SOP# 41 – Preparation of Hardcopy Analytical Reports Using ADT.

## **16.0 REFERENCES**

SW-846 Test Methods for Evaluating Solid Waste, Third Edition, Final Update III, Revision 1, December 1996.

Method TO-4A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/600/4-89/018, January 1999.

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## Attachment A

### Detection and QC Limits

**Table A-1. Methods TO-4A/TO-10A (Pesticides and PCBs)**

Analyte	DL ( $\mu\text{g}$ )	Low Point of the Curve ( $\mu\text{g}$ )	Acceptance Criteria			
			ICAL (%RSD)	ISCV (%R)	CCV (%R)	LCS** (%R)
4,4'-DDD	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
4,4'-DDE	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
4,4'-DDT	0.10	0.10	< 20	$\pm 20$	$\pm 15$	50 – 150
4,4'-Methoxychlor	1.0	1.0	< 20	$\pm 20$	$\pm 15$	
Aldrin	0.10	0.10	< 20	$\pm 20$	$\pm 15$	50 – 150
alpha-BHC	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
alpha-Chlordane	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Aroclor 1016/1242	1.0	1.0	< 20	$\pm 20$	$\pm 15$	57 – 116
Aroclor 1221*	1.0	1.0	< 20	$\pm 20$	$\pm 15$	
Aroclor 1232*	1.0	1.0	< 20	$\pm 20$	$\pm 15$	
Aroclor 1248*	1.0	1.0	< 20	$\pm 20$	$\pm 15$	
Aroclor 1254*	1.0	1.0	< 20	$\pm 20$	$\pm 15$	
Aroclor 1260	1.0	1.0	< 20	$\pm 20$	$\pm 15$	60 – 123
beta-BHC	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
delta-BHC	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Dieldrin	0.10	0.10	< 20	$\pm 20$	$\pm 15$	50 – 150
Endosulfan I	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Endosulfan II	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Endosulfan Sulfate	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Endrin	0.10	0.10	< 20	$\pm 20$	$\pm 15$	50 – 150
Endrin Aldehyde	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Endrin Ketone	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
gamma-BHC (Lindane)	0.10	0.10	< 20	$\pm 20$	$\pm 15$	50 – 150
gamma-Chlordane	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Heptachlor	0.10	0.10	< 20	$\pm 20$	$\pm 15$	50 – 150
Heptachlor Epoxide	0.10	0.10	< 20	$\pm 20$	$\pm 15$	
Technical Chlordane*	1.0	1.0	< 20	$\pm 20$	$\pm 15$	
Toxaphene*	1.0	1.0	< 20	$\pm 20$	$\pm 15$	

#### Surrogates

TCX	NA	NA	NA	NA	< 15	47 – 134**
DCB	NA	NA	NA	NA	< 15	47 – 159**

\* The noted multi-component compounds use a one-point calibration.

\*\* Recovery limits are subject to change based on internally developed control charts

**Table A-2. Summary of Calibration and QC Procedures for Methods TO-4A/TO-10A**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five Point Initial Calibration Curve (ICAL)*	Prior to sample analysis	%RSD $\leq$ 20	Use linear regression per SW-846 or recalibrate
Independent Source Calibration Verification (ISCV)	After each initial calibration	80 to 120% recovery	Investigate the source of discrepancy, including reparation and reanalysis of standard. Recalibrate if needed
Detection Limit Verification (DLV)	With each initial calibration	$\pm$ 50% of the expected value	Check the system and reanalyze the standard. Notify the QA department via a CAR form if the criteria cannot be met.
Breakdown Check (Endrin and p,p'-DDT)	Daily, prior to CCV for pesticide analysis	Degradation $\leq$ 15%	Perform maintenance. Repeat breakdown check.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, every 10 samples, and at the end of the analysis sequence. At the minimum of every 12 hours	Recovery of an individual component or the average of all the target components within 15% of the expected values	Analyze new ICAL and/or prepare fresh standards. If the standard analyzed is recovering high, "Q" flag the high recoveries. If the standard analyzed is recovering low, reanalyze all samples
Laboratory Control Spike (LCS)** for compounds noted in Table A-1.	With each set of 20 samples	As mentioned in Table A-1	"Q" flag the compounds outside the method limits
Surrogates**	With all samples and blanks prior to extraction	As mentioned in Table A-1	"Q" flag the compounds outside the method limits
Laboratory blanks	With each set of 20 samples extracted	Results less than the laboratory detection limit	"B" flag the compounds that do not meet the acceptance criteria
Laboratory Duplicates	5% of the samples	RPD $\leq$ 20% for detections $>$ 5 X's DL.	Analyze sample a 3 <sup>rd</sup> time. If criteria are still not met, report closest matching pair and "Q" flag data.
Second-column Confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis

\* Single point is performed for technical chlordane and toxaphene.

\*\* Recovery limits are subject to change based on internally developed control charts.

**Appendix B**  
**Standard Reporting Format**

@ Air Toxics Limited  
STANDARD OPERATING PROCEDURE

**RECEIVING, LOGIN, AND TRACKING OF SAMPLES**

**SOP #50**

Revision Date: 03/09/01  
Revision #: 3  
Reason for Revision: To meet NELAP requirements  
and add login section.

Updated By: \_\_\_\_\_

Signature	Print	Title	Date
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Technical Review: \_\_\_\_\_

Signature	Print	Title	Date
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QA Review: \_\_\_\_\_

Signature	Print	Title	Date
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Laboratory Director: \_\_\_\_\_

Linda L. Freeman	Date
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## **1.0 SCOPE AND APPLICATION**

The purpose of this Standard Operating Procedure is to outline the procedures for receiving air samples, login of samples in the database and in the proper storage area, and tracking these samples through the laboratory.

## **2.0 RECEIPT OF SAMPLES**

2.1 The sample receiving team assumes the custodial responsibilities associated with receiving, inspecting, and logging samples into ATL's ATLAS Sample Tracking, an internal sample tracking software program.

2.2 The policy establishes specific guidelines for sample acceptance, which are generally accepted practices under EPA, AFCEE, Army Corps of Engineers, Navy, and NELAC protocols.

2.3 When samples do not meet the established guidelines, discrepancies are documented and the client is notified. Discrepancies are also noted in the Narrative portion of the sample report.

2.4 The Sample Acceptance Policy is posted in the sample receiving area and copy is provided to field staff with every shipment of containers or media.

2.4.1 The following terms are used in defining the receipt condition of the samples:

- Good: No obvious damage, proper temperature/pressure
- Questionable: Evidence of possible shipping damage, custody seals not intact, received with improper temperature/pressure, COC form not received or incomplete
- Poor: Definite shipping damage, integrity of samples obviously compromised, broken sample media.

## **2.5 Sample Acceptance Policy**

2.5.1 Samples received by Air Toxics Ltd. are relinquished following standard EPA approved guidelines. These include full and complete Chain-of-Custody documentation indicating: unique sample name, location, date and time of collection, collector's name, preservation type (if applicable), matrix, and any special remarks.

- 2.5.2 The chain-of-custody form is filled out in ink and indicates proper preservation and use of sample container specified by the method.
- 2.5.3 Each sample is labeled with unique, durable, and indelible identification and must be of adequate volume for the tests requested.
- 2.5.4 Never affix a label directly on a Summa™ canister. A tag is attached to each canister for this purpose.
- 2.5.5 Proper, full, and complete inspection and documentation is performed upon laboratory receipt in the following areas:
- evidence of container's physical damage
  - status of the container's custody seal
  - presence or absence of a chain-of-custody form
  - incomplete or incorrect chain-of-custody form
  - number of samples
  - name of each sample
  - sample collection date/time
  - sample location
  - name of the collector
  - preservation type (if applicable)
  - sample type (canister, XAD, DNPH etc.)
  - sample tag information complete
  - temperature (when applicable)
  - pressure (canisters)
  - presence of unlabelled samples
  - presence of mislabeled samples
  - presence of unused media
  - method required trip blanks, field blanks, equipment blanks, field duplicates, or field spikes
- 2.5.6 Any sample discrepancies against the above criteria are documented on the Sample Discrepancy Form (Appendix A) and communicated to the client via Login Fax within 1 day of sample receipt.
- 2.5.7 The client is contacted by the project manager for discrepancies of a more serious nature, e.g.
- Custody seal on the outside of the container was broken.
  - Chain-of-Custody Record was not received with sample(s).
  - Analysis method(s) is(are) not specified.
  - Sample(s) received out of holding time.
  - Flow controller used - canister samples received at <2.5" Hg.
  - Sample container (Tube/VOA vial) was received broken.
  - Container for VOA analysis received with headspace.

- Tedlar Bag received leaking.
  - Tedlar Bag received flat.
  - Canister received with a leaky valve.
  - Tedlar bag/canister received emitting a strong odor (sample cannot be analyzed).
  - Initial laboratory Vacuum/Pressure does not match final field pressure
- 2.5.8 Documentation of client notification is included on the sample discrepancy form along with any instructions from the client on how to proceed.
- 2.5.9 Project managers complete this section and return the form to the receiving group to complete the login process. The form is archived in the Work Order (WO) folder.
- 2.5.10 Whenever there is any uncertainty of how the laboratory is to proceed or when the desired method is unclear, the receiving staff places the Login process ON HOLD and delivers the WO file to a Project Manager for follow-up.
- 2.5.11 The project manager contacts the client to clarify the situation. Phone calls between the project manager and the client are documented in the Client Services database.
- 2.5.12 The phone contact and client instructions to resolve the issue are logged into the database and a hardcopy report is placed in the WO folder.
- 2.5.13 The project profile is checked for client's financial status. If the financial status is "blacklisted" the project manager will be contacted and the samples placed on hold. A status of "Credit App missing" the client will be notified about the missing credit application on the login fax/e-mail cover page and the workorder folder is stamped "financial hold". The samples will proceed normally into the laboratory.
- 2.5.14 The folder is then returned to the Receiving team to complete the Login process. Air bills, packing lists, chain-of-custody records, and any other documentation that may accompany the samples are placed in the WO folder.
- 2.5.15 Laboratory malfunctions that may occur during or after sample receipt are documented via the laboratory Corrective Action system.

2.5.16 Examples of receiving problems which would necessitate a Corrective Action Request include:

- Sample was broken during handling.
- Hold time expired due to laboratory error.
- Canister sample pressurized with wrong type of gas.
- Sample placed "on hold" was released in error.
- Sample logged in for incorrect analysis method.
- DANGER tag was not affixed to an odiferous canister sample before sending to the lab.
- Canister was released and cleaned before second analysis method was run. Receiving did not affix the multiple analysis tag.
- Canister valve was left open following pressurization. Sample vented to ambient.

2.6 If a sample is received that may be hazardous to handle, the following procedures are used:

2.6.1 SUMMA™ Canisters and Tedlar Bags:

- Canisters or Tedlar bags which emit a strong odor, are immediately placed in a fume hood by the sample custodian.
- The Hazardous Materials Coordinator or member of the Safety Committee is contacted to assess the level of hazard associated with the sample(s) and to ensure proper handling.
- Any leaking Tedlar bag sample, which contains high levels of Hydrogen Sulfide, will not be analyzed. The client is notified immediately so that re-sampling may be scheduled if necessary.

2.6.2 Liquid and Sorbent Media Samples:

- The sample recipient always wears protective gloves while unpacking these types of samples.
- If a liquid sample is received in a leaking or broken container the Hazardous Materials Coordinator is notified for proper cleanup and handling of the sample.
- The Hazardous Materials Coordinator is also notified if a sample on sorbent media is received broken, to ensure proper handling.

2.6.3 After above inspections, receiving personnel fill out the COC form(s) accurately. After signing the COC in the "Received for lab" location (or any "Received by" location if it is not indicated) the Sample Recipient must follow the signature with "ATL". The correct time and date as well as the following information, given the circumstance, is filled in:

- If it is ATL's COC, the bottom portion of the COC, designated for the person responsible for opening the sample package (includes shipping and custody seal information), is filled in.
- If it is not ATL's COC, the "Custody Seals intact" and the "temperature" is stamped in ink on the bottom portion of the COC is used and the appropriate information is filled out.

*Any discrepancies found by the sample receiving team member must be documented on a Sample Receipt Discrepancy Form (see Appendix A).*

2.7 After the samples have been properly logged into ATLAS (the samples noted on the COC), the number of samples, media and analysis type, and significant dates (promised and expiration) are listed in the ATLAS database.

2.7.1 The following information describes both the sample holding times and how to set up the promised due date in the ATLAS/Sample receiving database: *(It is important to note that both due dates are referenced to working days unless otherwise specified.)*

#### HOLDING TIMES

Sulfur --> 1 day (Tedlar bag) & 3 days (Silco can) from date of collection.

Tedlar Bags --> 3 days from date of collection.

Formaldehyde extraction --> 7 calendar days from the date of collection.

TO-8 extraction --> 48 hours from time of collection

TO-13 extraction, liquids, PUF's --> 7 days from the date of collection.

Canisters, Tubes, and Natural Gas bombs --> 14 days from date of collection

Charcoal Tubes → 14 days from date of collection

Siloxane → 14 days from date of collection.

- If the analysis is to be performed with a quick turn around time, the sample receiving personnel put all documents (COC, air-bill, bid/ship, etc.) into a red folder.
- If the analysis is to be performed on a standard turn around time, the documents are placed in a yellow folder.
- The date and turnaround time is written on the outside of the red folders so that one is able to distinguish rush work orders quickly.
- The receiving duty is prioritized based on the following questions when considering which samples to give a work order number first (in order of highest to lowest priority):

1. Are the samples previously expired or expiring the same day of receipt?
2. Do the samples have a short hold time?
3. Do the samples need to stay cold?
4. Is the analysis requested a rush turn?
5. If the samples can be run the day of receipt, does the lab need them quickly?
6. Is the analysis a standard turnaround? Do the samples have a long hold time?

### ***3.0 THE LOGIN/FAX EMAIL***

- 3.1 The login fax is sent only if the folder and information entered into ATLAS has been checked by a second receiving personnel and found to be correct. This is documented on the receiving/login checklist. (See Appendix B)
  - 3.1.1 When Login is completed, a fax or e-mail is sent to the client to confirm receipt of samples. The Login fax has four parts:
    - Page 1: Cover page with discrepancies noted
    - Page 2: Log-in summary (sample names etc.)
    - Page 3: Reporting template showing referenced method, target compound list, and reporting limits
    - Page 4: Client's COC
  - 3.1.2 A copy of either the login fax or e-mail is placed into the appropriate workorder folder.
  - 3.1.3 Discrepancies are noted on the cover page using a template of pre-approved statements. QA is responsible for maintaining the approved template. Receiving staff electronically copy relevant statements from the template and onto the FAX cover page.
  - 3.1.4 Document in ATLAS the date the login e-mail was sent.

### ***4.0 CREATING THE WORK ORDER***

- 4.1 When sample receiving receives the media, the project profile and shipping form is matched by scanning the canister bar codes or manually by matching company and project information.
- 4.2 The samples, in which the media did not originate at ATL are matched with a project profile by the client service's representative who has been in contact with the client.

- 4.3 The Sample Receiver must include the following items in each WO when it is created:
- Chain-of-Custody (COC)
  - Air-bill
  - Shipping Form\*, Contacts\*, Quotes\*
  - Project Profile
  - Sample Receipt Summary
  - Discrepancy Report (Pink Sheet)
  - Receiving Report (ATL media only)
  - Other documents (i.e., special receiving form)\*
  - \*if applicable
- 4.4 It is critical for the sample receiver to search out information on any unknown situation when receiving samples using ATLAS (i.e. Contacts, Shipping, Sample tracking, Quotes) Faxes, E-mail, or any other significant source.
- 4.5 The sample receiver attaches a print out of the Project Profile to the inside front cover of the WO folder. The Project Profiles contains the specific information laboratory personnel will need in order to process the samples. This information includes general client information as well as any specific project requirements that differ from ATL's SOPs.
- 4.6 A unique number is assigned to a WO using the Sample Tracking log. For example, the first batch of samples received in January 2001 would be identified as **0101001**. The first **01** refers to the year, the second **01** designates the month, and **001** determines the batch number. The Sample Receiver then enters the WO number and other pertinent information when creating the workorder in ATLAS, making sure that all information is correct regarding the samples just received.
- 4.6.1 The following are guidelines to assigning WO numbers when multiple analyses are requested and/or multiple COCs are received for samples:
- a. When multiple analyses are requested on given samples on the same COC(s), the work order is divided by adding a letter to the end of the WO number. For example, if three analyses are requested from samples to be assigned work order **9901001**, each given WO would be defined as **9901001A**, **9901001B**, and **9901001C**.
  - b. If multiple COCs are received for samples analyzed for the same analysis, client, and project, all COCs may be placed in one WO.

- c. When multiple COCs are received and the samples on each COC are from different projects, there must be as many WOs assigned as the number of COCs.

4.7 Fraction numbers and specific QA/QC samples (laboratory duplicates, etc.) are assigned to the samples in a WO based on the information in the Project Profiles, and the following instructions:

- When samples of different media type are received on the same COC and each media has a different analysis – the work order is split and fraction numbers are assigned by sequentially numbering the samples in order starting from fraction (01A) for that specific media type.
- If samples are received of the same media type, on the same COC, and for multiple analysis only on some of the samples - fractions are assigned by numbering all the samples sequentially, and writing only the fractions that apply to each WO on the COC.
- VOST tubes are given the fraction “A” for the Tenax (front) tube and “B” for the Tenax/charcoal (back) tube. If there is an Anasorb tube present, the Anasorb is always combined with a Tenax tube to be analyzed properly. Sorbent tubes and charcoal tubes are labeled “A” as front tube and “B” as a back tube as well.
- For work orders involving an extraction, only TO-4 and TO-13 samples are assigned fractions. For other cases, samples are labeled the work order number only.
- Duplicate analysis is not possible for samples in VOST tubes and Carbotraps.
- Duplicate analysis is possible for VOST Condensates, but only if it is specified in the Project Profile.
- Composite samples or samples that are to be combined into one are given fractions starting with the first sample labeled fraction “A”, and subsequent samples labeled the next letter. For example, if three Tedlar bags were composited, the fractions would be 01A, 01B, and 01C.

4.7 Any equipment returned on a Bid/ship must be checked in using the Canister Asset Tracking System (CATS). Any special circumstance regarding labeling and analysis of samples is always documented in the project profile.

## **5.0 SAMPLE STORAGE, TRACKING AND DISPOSAL**

- 5.1 Samples that do not require extraction must be stored in the sample cage in the main laboratory. The Sample Receiver must assure that all samples are placed in the sample cage by the end of the day. Samples that do not require extraction but must remain chilled are placed in the appropriate refrigerator located at the back of the sample cage.
- 5.2 Those samples, which require an extraction, are placed in the formaldehyde refrigerator, extractions refrigerator, or VOST refrigerator (depending on analysis), see Table 1.

Table 1

Location in Sample Cage	Contents
Section A1	Tedlar Bags
Sections A2-A3	1L summa canisters
Sections B1-D4	6L Summa canisters, tanks, and vacutainers

Location	Freezers	Contents
Cage	4	Charcoal tubes for NIOSH

Location	Refrigerators	Contents
Extractions Lab	1	Semivolatile samples and preparation media
Cage	4	VOST samples only
	5	Water for headspace analysis
Extractions Lab	6	Semi-volatile extracts, PCB and PEST extracts, and any other samples which do not belong in the other refrigerators
	7	Aldehyde and Ketone samples and preparation media
	8	Extracted samples for BIF0011
Team Leader Office	DZ	TSP Filters/PM10 Filters

- 5.3 The Sample Receiver records samples into either the Internal Sample Tracking logbook or the extraction's lab logbook titled "Extractable Sample Tracking". Samples that have already been extracted when they are received are entered into the "Extract Tracking" logbook. When entering the samples, the Sample Receiver has to include the WO number and specific fractions, location of samples, name and date.
- 5.4 As the samples are removed for analysis, the responsible analyst/scientist documents each step in the Internal Sample Tracking logbook. The following procedures are used:
- When removing a set of samples the analyst records his/her initials and the time and date of removal from the secure area.
  - As each sample is analyzed, it is logged into the instrument logbook indicating date of analysis and unique computer file number.

- Samples removed from the secure area but not currently being analyzed are still considered to be in the possession of the analyst/scientist.
- Following analysis, the analyst/scientist returns the samples to the secure area and documents this action in the appropriate logbook.
- Samples remain in the secure area until time of disposal. Disposal is indicated in the Internal Sample Tracking logbook.

*List of Appendices*

- Appendix A Sample Receipt Discrepancy Report*  
*Appendix B Receiving Login Checklist*