EPA Contract No. 68-W9-0036
EPA Work Assignment No. 51-1P19

EPA Project Officer: Diana King
EPA Work Assignment Manager: Sheila Eckman

FINAL SAMPLING AND ANALYSIS PLAN

Pine Street Canal Site
Burlington, Vermont

September 1995

Prepared by:

Metcalf & Eddy
An Air & Water Technologies Company

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FINAL
SAMPLING AND ANALYSIS PLAN
FOR
SPLIT-SAMPLING ACTIVITIES
as part of the
ARI OVERSIGHT

PINE STREET CANAL SITE
BURLINGTON, VERMONT

September 1995

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SAMPLING AND ANALYSIS PLAN

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SAMPLING AND ANALYSIS PLAN
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PREFACE

The objective of Metcalf & Eddy's (M&E's) quality assurance program is to ensure that all measurement, data gathering, and data generation activities yield data that are of adequate quality for the intended use. The key to achieving this objective is the successful implementation of a Sampling and Analysis Plan (SAP). The SAP consists of two parts: (1) the Quality Assurance Project Plan (QAPP), which describes the policy, organization, functional activities, and quality assurance and quality control protocols necessary to achieve data quality objectives dictated by the intended use of the data; and (2) the Field Sampling Plan (FSP), which provides guidance for all field work by defining in detail the sampling and data gathering methods to be used on a project.

This document constitutes a site- and project-specific SAP for the split-sampling activities for the RI/FS oversight assignment at the Pine Street Canal Site in Burlington, Vermont. The SAP for this work assignment has been written specifically for the field oversight and split-sampling activities that are associated with the Potentially Responsible Parties' (PRP) Phase IIB work of the Additional Remedial Investigation and Additional Feasibility Study (ARI/AFS), which is being conducted in accordance with the Administrative Order by Consent (USEPA Docket No. I-95-1048. This SAP was developed in conjunction with the United States Environmental Protection Agency's (EPA) Scope of Work and M&E's Final Work Plan for this assignment. The intent of this SAP is to technically oversee the field and sampling activities associated with the RI/FS Work Plan and the PRP's EPA-approved Phase IIB Work Plan.

Due to the nature of the work assignment, the limited size of the field effort in the collection and analysis of split samples, and the need to maximize cost effectiveness and thus minimize efforts in the production of materials which may be repetitive, the QAPP and FSP have been consolidated into one document. All applicable elements of the FSP have been incorporated into the QAPP format to produce this SAP.

1.0 INTRODUCTION

This SAP presents the data quality objectives (DQOs) and the corresponding quality assurance (QA) standards for M&E's field and split-sampling program that will be conducted during the Remedial Investigation/Feasibility Study (RI/FS) Phase at the Pine Street Canal Site, in Burlington, Vermont. The split-sampling program consists of the technical oversight of sample collection through the analysis and validation of M&E's split samples and the comparison of M&E split-sample data with the PRP's sample data. This SAP is designed to assure that the acquisition and analysis of oversight split samples is performed in the highest quality manner, that the results are obtained in a comparable manner to the results produced by the PRPs, and that the results will be defensible in a court of law.

Included in this SAP are the detailed field and laboratory protocols to be followed by M&E field personnel to assure the quality and integrity of the data, accuracy and precision of the analyses, representativeness of the results, and completeness of the information obtained by M&E from the analysis of split samples. Consequently, sediment samples collected for semivolatile organic and metals split-sample analyses will be analyzed through the EPA-Contract Laboratory Program (CLP) system for Routine Analytical Services (RAS) and sediment samples for grain size through the M&E Delivery of Analytical Services (DAS) program using the highest level of quality control (QC).

Split sample collection shall be performed according to protocols defined in Section 5.0 of this document. Data generated from split samples shall be compared with data submitted by the PRPs and their consultant The Johnson Company (JCO).

2.0 PROJECT DESCRIPTION

This section, which is summarized from the Final Work Plan (M&E, 1995), provides a description of the Pine Street Canal Site. The overall project objectives, as well as the objectives of the split sampling, are also discussed.

2.1 SITE LOCATION AND DESCRIPTION

The Pine Street Canal Site is located on the eastern shore of Lake Champlain in Burlington, Vermont (Figure 2-1). The site is situated in a highly industrial area approximately one-half mile south of the center of Burlington. It encompasses approximately 70 acres, most of which are vacant. The site is bounded by Pine Street to the east, Lakeside Avenue to the south, the railroad tracks to the west, and the Ultramar Petroleum property to the north (Figure 2-2). It lies in a topographically low area and includes an abandoned barge canal with a turning basin and filled-in boat slips, and vegetated wetlands south, east, and west of the canal.

2.2 SITE BACKGROUND

The primary environmental concern at the site is the past operation of the coal gasification plant near the southern end of the canal, from which coal tar residues were allegedly disposed. These residues have been detected in groundwater, canal sediments, and soils throughout the site. Previous investigations conducted on the site have identified subsurface contamination consisting primarily of polynuclear aromatic hydrocarbons (PAHs), volatile organic compounds, and metals. Detailed descriptions of the site and its location, a site chronology, and state and EPA regulatory activities are provided in several reports which outline the remedial investigation (RI) (PEER, 1990), the supplemental RI (M&E, 1992a), the treatability study (M&E, 1992b), the risk assessment (M&E, 1992c), and the feasibility study (M&E, 1992d).

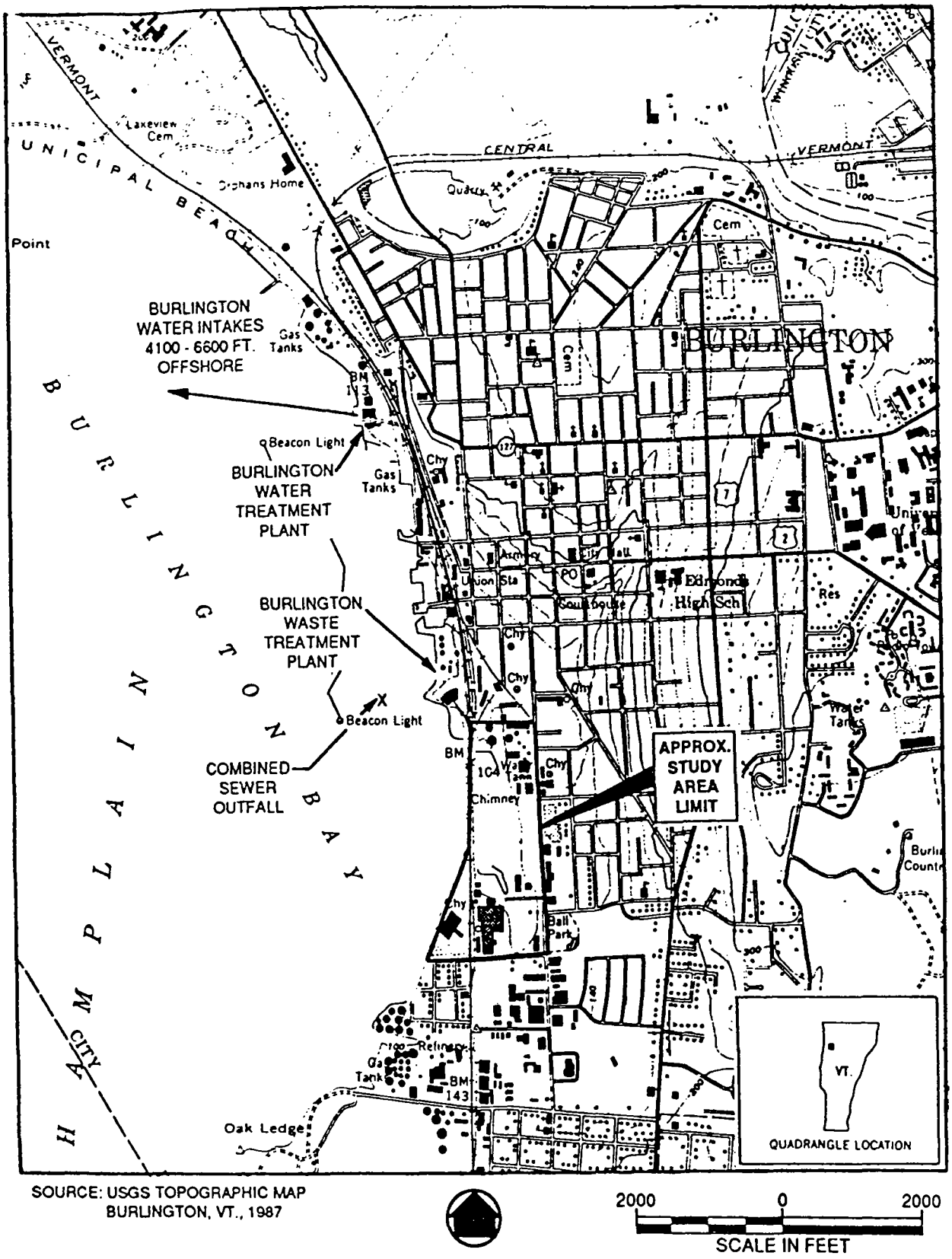
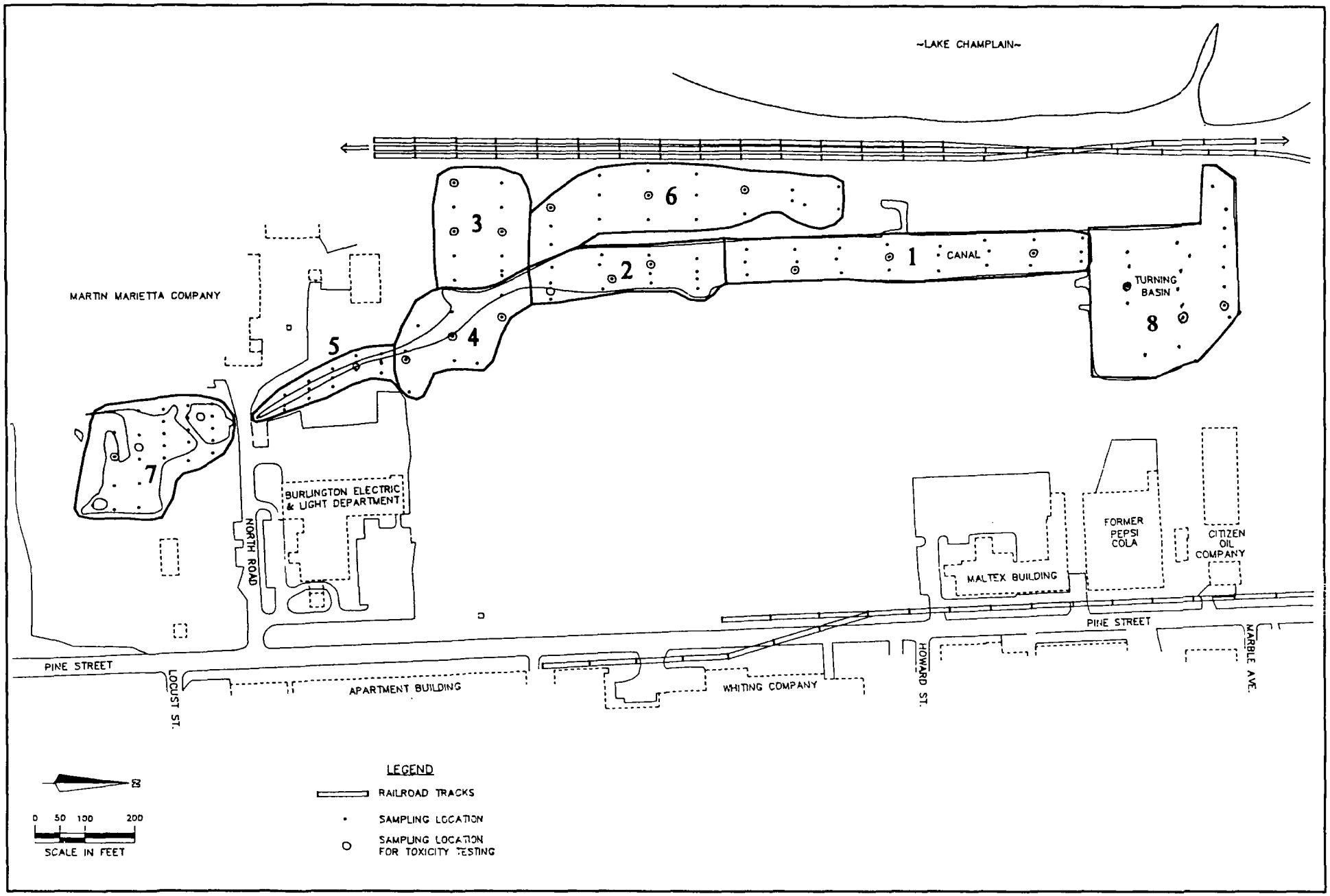


FIGURE 2-1. REFERENCE AREA



SOURCE: Johnson Co., 1995

FIGURE 2-2. PHASE 1A SOIL/SEDIMENT SAMPLES IN AREA OF FOCUS FOR PHASE 11B PINE STREET CANAL SITE, BURLINGTON, VT

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2.3 PROJECT OBJECTIVES

The PRP's Phase IIB Work Plan states that the "primary objective of the ARI/AFS is to provide a focused assessment of Site conditions and evaluation of alternatives to the extent necessary to select a remedy for the Site, as defined in the Administrative Order by Consent (AOC), that shall be consistent with the NCP and relevant guidance". In addition, the PRP's goal of the Phase II ARI is to complete the studies outlined in Phase I that were not completed and to collect supplemental field data necessary for the AFS and a revised ecological risk assessment.

M&E's work objective for this assignment is to provide EPA with technical oversight support for evaluation of the PRP's ARI activities and/or submittals for Pine Street Canal. The specific objectives for this assignment are:

1. To oversee the PRP's ARI Phase IIB, Part 2, activities and review submittals.
2. To identify and describe significant deviations in the PRP's ARI activities and/or submittals with respect to the Administrative Order by Consent, Statement of Work, and EPA-approved Work Plan.
3. To evaluate the PRP's performance using physical observations and confirm the usability of the generated data.

2.4 SCOPE OF FIELD ACTIVITIES

The scope of the PRP's field and laboratory activities for Phase IIB, Part 2, will include:

- Sediment Sampling and Toxicity Tests,
- Habitat Evaluations, and
- Literature Survey

M&E will provide oversight and collect split samples during sediment sampling and only provide oversight during the collection of samples for toxicity testing and during field screening. The sediment samples collected for whole sediment toxicity testing will be analyzed by the PRP's laboratory, with no split samples collected by M&E. However, M&E will perform an audit of

the PRP's toxicity testing laboratory. At the request of EPA, no oversight will be provided by M&E during the Habitat Evaluation or Literature Survey.

Sediment sampling will be performed by the PRPs at twenty-three different locations within eight areas (Figure 2-2) which showed high concentrations of PAH and metals contamination during Phase I sampling. M&E will collect split samples for semivolatile, metals, and grain size analyses.

The PRPs will be using toxicity test methods compatible with EPA guidance contained in *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates,* EPA 600/R-94/024 and as approved by the Ecological Work Group and Coordinating Council. All analyses will be performed within the two week holding times. All sediment samples collected by the PRPs for toxicity testing will undergo *Chironomus tentans* and Micotox analysis, sediment samples from areas 2, 3, 6, and 7 will receive FETAX toxicity tests, and the *Hyalella azteca* test will be used for sediments from areas 1, 2, 4, 5, and 8. A detailed description of the sampling program, analyses, and data evaluation is found in the PRP's ARI Phase IIB Work Plan (Johnson, 1995)

2.4.1 Collection of Split Samples

During oversight of the field activities, M&E will obtain split samples from the field samples collected by the PRPs. The PRPs will be sampling 23 locations and collecting 29 samples including quality assurance/quality control (QA/QC) samples, from which approximately 10 sediment samples will be split by M&E for laboratory analysis of semivolatile organics and metals through the CLP RAS program and approximately 5 sediment samples will be split for grain size analysis using an EPA-approved M&E DAS method. Based on a review of the analytical methods presented in the PRP's Work Plan (Johnson Co., 1995) and the M&E split-sampling procedures outlined in this SAP, all efforts have been made to ensure that the split samples will be collected and analayzed in a manner similar to the field samples collected by the PRPs. The split-sampling program and the PRP's sampling procedure is more fully described

throughout this SAP. A summary of the expected number of samples that will be split is presented in Section 5.2.

2.4.2 Schedule of Field Activities

Field activities are expected to take place the second and third week in October 1995.

2.5 DATA QUALITY OBJECTIVES

The objective of the M&E split-sampling program is to produce data that are comparable and confirm the PRP's sample data. To achieve this objective, all split data must be comparable according to DQO Level IV criteria using current EPA-SOWs for RAS methods: organic analyses (USEPA, 1993b) and inorganic analyses (USEPA, 1993c). All RAS analytical data (Level IV) and DAS analytical data (Level III) will be validated to Tier III as specified in the *EPA Region I Memorandum* (USEPA, 1993a) in accordance with *EPA Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses* (USEPA, 1988) and *EPA Region I Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses* (USEPA, 1989); modified to meet criteria in the current EPA SOWs for RAS organics and inorganics analyses (USEPA, 1993b and 1993c).

Analyses that are not part of the RAS program will be performed using the DAS program, where the highest level of data quality control will be stipulated and the deliverables will resemble RAS data as much as possible. All project-related DAS analytical specifications (M&E, 1995a and 1995b) have been approved by EPA.

Field oversight and review of the PRP's work plans will ensure that sample collection and field data collection activities are adequate for the intended uses. However, ultimately, the PRP's contractors are responsible for the PRP's field and analytical data.

Based on a review of these plans, the PRP's DQOs are as follows: "all screening samples will be performed to at least DQO Level I specifications" and "all confirmatory samples will be analyzed using DQO Level IV or V, depending on the analyte list."

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The Project Organization Chart (Figure 3-1) identifies the project staff responsible for each element of the overall program. The key individual responsible for QA is the ARCS QA Manager (QAM), who reports directly to the Corporate QA Manager. The QAM communicates with the Project Manager and the M&E QA Officer relative to the overall quality of project deliverables. The M&E Project Manager reports to the EPA Remedial Project Manager (RPM) and is responsible for overall project objectives. The Project Engineer reports directly to the Project Manager and is responsible for overseeing the implementation of the project objectives.

Analytical results generated by the CLP or DAS laboratories will be reported directly to the Project Chemist who is in contact with the Project Engineer and Lead Chemist. The Project Chemist coordinates oversight of sampling activities and is responsible for data validation. The Lead Chemist is the main contact with the Sample Management Office (SMO), the CLP laboratories, and the DAS laboratories.

All environmental samples will be submitted to EPA-approved CLP laboratories for RAS analyses and to M&E-procured (and EPA-approved) laboratories for DAS analyses. The RAS laboratories will be assigned by EPA's SMO prior to any field work involving sample collection. M&E-procured laboratories that have been approved by EPA will be assigned for DAS analyses and handled as part of the analytical services work assignment (WA#46-1HZZ).

The key individuals responsible for implementation of QA procedures and their specific QA responsibilities are as follows:

M&E ARCS Program QA Manager - Robert Reimold, Ph.D.

- Meets with the ARCS Program Manager monthly and prepares the monthly progress report on program activities and related QA activities
- Conducts periodic performance audits of environmental data collection activities (EDCAs) using non-project related staff

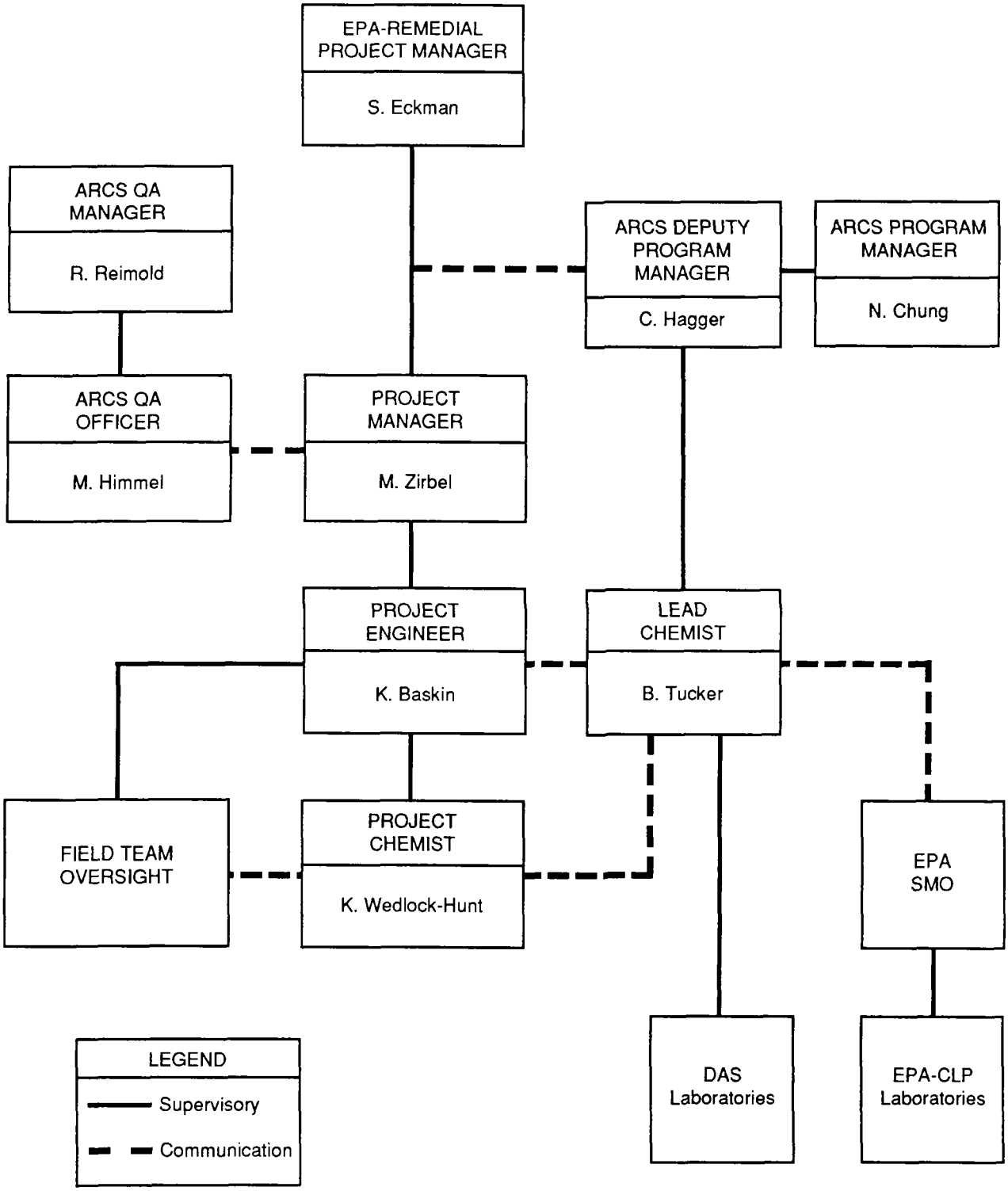

FIGURE 3-1. PROJECT ORGANIZATION CHART FOR PINE STREET CANAL SITE IN BURLINGTON, VERMONT

- Conducts semi-annual program QA audits and reports to EPA
- Oversees Document Review Team reviews and responses
- Facilitates resolution of any Technical Advisory Team (TAT) concerns
- Assures compliance to applicable ARCS QA standard operating procedures

M&E ARCS Project QA Officer - Meg Himmel

- Reports to the M&E ARCS QA Manager
- Assists in surveillance and oversight audits of field activities
- Coordinates performance and systems audits of field sampling activities and document reviews
- Drafts monthly QA reports

M&E Project Manager - Martha Zirbel

- Reports to the ARCS Deputy Program Manager and EPA RPM
- Reviews and approves of all project plans and deliverables
- Assures that technical oversight activities meet project objectives
- Assures that sampling program meets EPA objectives
- Implements recommendations made by the ARCS QAM

M&E Project Engineer - Kathleen Baskin

- Reports to the Project Manager
- Reviews and approves all sampling activities and procedures for compliance with this SAP
- Coordinates field activities with the Project Chemist and field team

M&E Project Chemist - Karen Wedlock-Hunt

- Reports to Project Engineer and Project Manager
- Coordinates analytical and sampling activities with the Project Engineer
- Assures that samples are collected, analyzed, and validated in accordance with this SAP and approved quality assurance/quality control (QA/QC) procedures
- Coordinates analytical activities with Lead Chemist
- Interacts with SMO regarding laboratory issues as directed by the Lead Chemist
- Performs data validation of M&E's samples and compares M&E's split-sample data with ESD's data
- Reviews M&E procured-laboratory invoices for accuracy and approves invoices for payment

M&E Lead Chemist - Brian Tucker, Ph.D.

- Reports to ARCS Deputy Program Manager
- Oversees writing and submittal of DAS methods
- Coordinates sample analysis activities between M&E and SMO prior to field activities and interacts with SMO regarding laboratory issues
- Ensures that data generated is evidence audited prior to validation
- Ensures that data generated is validated according to Region I Functional Guidelines
- Ensures that the validation results are communicated with EPA
- Ensures that the data generated from the field investigation is valid prior to its use in project reports

M&E DAS Laboratory/Tracking Coordinator - John Verban

- Reports to the lead chemist
- Ensures proper sample and data tracking through initialization of sample identification, chain-of-custody, and laboratory data reports
- Coordinates DAS laboratory assignments and tracks samples from collection through data validation

M&E Field Team

- Reports to the Project Engineer
- Responsible for collection of representative environmental samples
- Assures that samples are collected in accordance with approved QA/QC procedures
- Prepares, packages, and ships samples to the laboratories

4.0 QUALITY ASSURANCE OBJECTIVES FOR THE LABORATORY

Comprehensive QA objectives provide guidelines for all field and laboratory procedures. All data generated from M&E's split samples will meet the project DQOs as described in this section. The use of accepted, published sampling and analytical methods, as well as the use of standardized units, will ensure the comparability of the data to historical results and any future results.

The EPA specifies, and this project requires, that five major characteristics of data quality be addressed to meet the objectives of the split-sampling program. The five characteristics include accuracy, precision, completeness, representativeness, and comparability. Specific QA objectives pertinent to the split-sampling program for this project are presented in Sections 10 and 13. To the maximum extent possible, split-sampling objectives will not change the laboratory QA objectives.

4.1 ACCURACY

Accuracy is defined as the degree of agreement of a measurement (or measurement average) with an accepted reference or true value. It is a measure of system bias and is usually expressed as a percentage of the true value. An evaluation of accuracy incorporates both laboratory and field sampling variables.

Accuracy will be determined in the laboratory through the use of matrix spike and matrix spike duplicate (MS/MSD) analyses. Accuracy criteria for the MS/MSD are defined in the methods that will be used during the split-sampling effort and are listed for solid matrices in Tables 4-1, for the CLP RAS analyses. Accuracy information for the DAS analysis for grain size is provided in the specification located in Appendix A.

At a minimum, the field team will select one environmental split sample in 20 for each sample matrix being submitted for MS/MSD analysis. One volume of the sample will be routinely

analyzed, while two volumes will be spiked with known quantities of particular target analytes prior to analysis. For sediment samples, additional sample volume does not need to be collected, however, sufficient sample must be supplied such that both the routine and spike analyses may be conducted.

The resulting MS/MSD data will be used to evaluate accuracy as well as precision for organic analyses (Section 4.2). Since matrix effects can affect the recoveries of the spiked compounds, the percent recoveries (%R) will be calculated and used as an indication of the accuracy of the analyses performed.

Accuracy will also be determined in the laboratory by the analysis of performance evaluation (PE) samples. The PE samples are obtained through the Region I ESD laboratory and consist of known concentrations of analytes in a solid matrix. Each PE sample is contained in a sealed amber glass ampule. The PE ampules and instructions for preparing the PE samples are delivered to the laboratory along with the environmental samples. Once the laboratory results are received by M&E they will be forwarded to ESD for scoring. The PE results will be assessed during data validation.

For inorganic analyses, accuracy is also measured by analysis of a laboratory control sample (LCS). The LCS is obtained from EPA and is analyzed with each batch of samples. The LCS results will be assessed during data validation.

Method blanks will also be used to evaluate accuracy. The method blanks will be prepared in the laboratory in a similar fashion as the associated samples of a particular matrix (i.e., soil, water, etc.) and analyzed along with these samples. The results of the analysis of the method blanks are a measure of the preparation accuracy and serve as a check on any sample contamination that may be introduced during sample preparation and analysis.

TABLE 4-1. QA OBJECTIVES FOR LABORATORY MEASUREMENTS OF SOIL/SEDIMENT ANALYSES

Parameter/Analytical Level[1]	Precision[2] (as RPD)	Accuracy[3] (Recovery)	Completeness
RAS TAL METALS ANALYSES REQUESTED:			
Metals/CLP Level IV ILMO3.0			
Aluminum	35%/50%	75-125%	90%
Antimony	35%/50%	75-125%	90%
Arsenic	35%/50%	75-125%	90%
Barium	35%/50%	75-125%	90%
Beryllium	35%/50%	75-125%	90%
Cadmium	35%/50%	75-125%	90%
Calcium	35%/50%	75-125%	90%
Chromium	35%/50%	75-125%	90%
Cobalt	35%/50%	75-125%	90%
Copper	35%/50%	75-125%	90%
Iron	35%/50%	75-125%	90%
Lead	35%/50%	75-125%	90%
Magnesium	35%/50%	75-125%	90%
Manganese	35%/50%	75-125%	90%
Mercury	35%/50%	75-125%	90%
Nickel	35%/50%	75-125%	90%
Potassium	35%/50%	75-125%	90%
Selenium	35%/50%	75-125%	90%
Silver	35%/50%	75-125%	90%
Sodium	35%/50%	75-125%	90%
Thallium	35%/50%	75-125%	90%
Vanadium	35%/50%	75-125%	90%
Zinc	35%/50%	75-125%	90%

TABLE 4-1 (Continued). QA OBJECTIVES FOR LABORATORY MEASUREMENTS OF SOIL/SEDIMENT ANALYSES

Parameter/Analytical Level[1]	Precision[2] (as RPD)	Accuracy[3] (Recovery)	Completeness
RAS TCL ORGANIC ANALYSES REQUESTED:			
Semivolatile Organics/CLP Level IV OLMO1.9			
Phenol	35%/50%	26-90%	90%
Bis(2-chloroethyl)ether	NPM/50%	NPM	90%
2-Chlorophenol	50%/50%	25-102%	90%
1,3-Dichlorobenzene	NPM/50%	NPM	90%
1,4-Dichlorobenzene	NPM/50%	28-104%	90%
1,2-Dichlorobenzene	NPM/50%	NPM	90%
2-Methylphenol	NPM/50%	NPM	90%
2,2'-oxybis(1-Chloropropane)	NPM/50%	NPM	90%
4-Methylphenol	NPM/50%	NPM	90%
N-Nitroso-di-n-propylamine	38%/50%	41-126%	90%
Hexachloroethane	NPM/50%	NPM	90%
Nitrobenzene	NPM/50%	NPM	90%
Isophorone	NPM/50%	NPM	90%
2-Nitrophenol	NPM/50%	NPM	90%
2,4-Dimethylphenol	NPM/50%	NPM	90%
Bis(2-chloroethoxy)methane	NPM/50%	NPM	90%
2,4-Dichlorophenol	NPM/50%	NPM	90%
1,2,4-Trichlorobenzene	23%/50%	38-107%	90%
Naphthalene	NPM/50%	NPM	90%
4-Chloroaniline	NPM/50%	NPM	90%
Hexachlorobutadiene	NPM/50%	NPM	90%
4-Chloro-3-methylphenol	33%/50%	26-103%	90%
2-Methylnaphthalene	NPM/50%	NPM	90%
Hexachlorocyclopentadiene	NPM/50%	NPM	90%
2,4,6-Trichlorophenol	NPM/50%	NPM	90%
2,4,5-Trichlorophenol	NPM/50%	NPM	90%
2-Chloronaphthalene	NPM/50%	NPM	90%
2-Nitroaniline	NPM/50%	NPM	90%
Dimethylphthalate	NPM/50%	NPM	90%

TABLE 4-1 (Continued). QA OBJECTIVES FOR LABORATORY MEASUREMENTS OF SOIL/SEDIMENT ANALYSES

Parameter/Analytical Level[1]		Precision[2] (as RPD)	Accuracy[3] (Recovery)	Completeness
Semivolatile Organics (Cont'd)	**0LMO1.9**			
Acenaphthylene		NPM/50%	NPM	90%
2,6-Dinitrotoluene		NPM/50%	NPM	90%
3-Nitroaniline		NPM/50%	NPM	90%
Acenaphthene		19%/50%	31-137%	90%
2,4-Dinitrophenol		NPM/50%	NPM	90%
4-Nitrophenol		50%/50%	11-114%	90%
Dibenzofuran		NPM/50%	NPM	90%
2,4-Dinitrotoluene		47%/50%	28-89%	90%
Diethylphthalate		NPM/50%	NPM	90%
4-Chlorophenyl-phenyl ether		NPM/50%	NPM	90%
Fluorene		NPM/50%	NPM	90%
4-Nitroaniline		NPM/50%	NPM	90%
4,6-Dinitro-2-methylphenol		NPM/50%	NPM	90%
N-nitrosodiphenylamine		NPM/50%	NPM	90%
4-Bromophenyl-phenylether		NPM/50%	NPM	90%
Hexachlorobenzene		NPM/50%	NPM	90%
Pentachlorophenol		47%/50%	17-109%	90%
Phenanthrene		NPM/50%	NPM	90%
Carbazole		NPM/50%	NPM	90%
Anthracene		NPM/50%	NPM	90%
Di-n-butylphthalate		NPM/50%	NPM	90%
Fluoranthene		NPM/50%	NPM	90%
Pyrene		36%/50%	31-142%	90%
Butylbenzylphthalate		NPM/50%	NPM	90%
3,3-Dichlorobenzidine		NPM/50%	NPM	90%
Benzo(a)anthracene		NPM/50%	NPM	90%
Chrysene		NPM/50%	NPM	90%
Bis(2-ethylhexyl)phthalate		NPM/50%	NPM	90%
Di-n-octylphthalate		NPM/50%	NPM	90%
Benzo(b)fluoranthene		NPM/50%	NPM	90%
Benzo(k)fluoranthene		NPM/50%	NPM	90%
Benzo(a)pyrene		NPM/50%	NPM	90%

TABLE 4-1 (Continued). QA OBJECTIVES FOR LABORATORY MEASUREMENTS OF SOIL/SEDIMENT ANALYSES

Parameter/Analytical Level[1]		Precision[2] (as RPD)	Accuracy[3] (Recovery)	Completeness
Semivolatile Organics (Cont'd)	**OLM01.9**			
Indeno(1,2,3-cd)pyrene		NPM/50%	NPM	90%
Dibenz(a,h)anthracene		NPM/50%	NPM	90%
Benzo(g,h,i)perylene		NPM/50%	NPM	90%

NOTES:
1. RAS METHODS:
   Organics: Contract Laboratory Program, Statement of Work for Organics Analysis (Multi-Media/Multi-Concentration). Document No. OLM01.0 with revisions through OLM01.9. (USEPA, 1993b).
   Inorganics: USEPA Contract Laboratory Program, Statement of Work for Inorganics Analysis (Multi-Media/Multi-Concentration). Document No. ILM01.0 with revisions through ILM03.0). (USEPA, 1993c).
2. Precision - Maximum relative percent difference (RPD) between laboratory duplicates/field duplicates for inorganic analyses and duplicate matrix spike recoveries/field duplicates for organic analyses.
3. Accuracy - Acceptable matrix recovery range as specified by the method.

NPM Not part of method

Sampling accuracy will be maintained by the implementation and adherence to strict method protocols as well as the collection and analysis of equipment blanks. Equipment blanks are collected to ensure the cleanliness of sampling equipment. An equipment blank is collected at least once for each piece of equipment that comes into contact with the sample during collection. An equipment blank is generated in the field by filling sample bottles with analyte-free water that is rinsed through decontaminated field equipment.

4.2 PRECISION

Precision is a measure of agreement among individual measurements of the same property under similar conditions. Laboratory precision will be determined through the comparison of MS/MSD recoveries (as described in Section 4.1) for the organic methods and laboratory replicates for inorganic analyses. It is expressed in terms of relative percent difference (RPD) between replicate measurements or in terms of the standard deviation when three or more replicate analyses are performed. As such, the RPD between results is a measure of analytical precision. Specific criteria for precision for the RAS methods are presented in Table 4-1.

Laboratory precision is also measured through the comparison of calibration standards and continuing calibration standards and is tracked using control charts in the EPA-CLP laboratory.

Sampling precision will be determined through the collection and analysis of field duplicates. Field duplicate analysis also provides an estimate of the sample media's actual heterogeneity. At least one field duplicate will be collected for each medium sampled.

The RPD between the PRP's data and the M&E split-sample data is another measure of precision, since the split samples are considered to be field duplicates. Although the analysis is performed by different laboratories, the data should fall within the criteria prescribed for precision. This provides a measure of the differences in laboratory preparation and analyses and sample heterogeneity. However, the comparability of data sets generated by two laboratories must be considered and taken into account, especially when different analytical methods are used to test for the same analytes. This is important since subtle differences in methods (i.e., detection limits, QC criteria, types of interferences, etc.) may affect data comparability between data sets.

4.3 COMPLETENESS

Completeness is a measure of the amount of valid data obtained compared to the amount expected to be collected. It is usually expressed as a percentage. The completeness objective for this split-sampling program is to obtain all split samples as specified in this SAP, to provide a sufficient quantity of sample for each of the required field and laboratory analyses, and to obtain QC samples representative of all possible contamination sources (i.e., sample collection, storage, transportation, etc.). Specific criteria for completeness are listed in Table 4-1 for the RAS analyses.

4.4 REPRESENTATIVENESS

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, process condition, a sampling point, or an environment. For this project, grab samples will be collected. The grab samples are by definition, representative of only the conditions at the point in time collected, within sampling and analytical error.

4.5 COMPARABILITY

Comparability expresses the confidence with which one data set can be compared to another. To achieve comparability in this project, the data will be generated using standard analytical procedures and reported using units of μg/L, μg/kg, and mg/kg. By using sampling and analysis procedures consistent with EPA protocols all data sets will be comparable for the site and between other EPA sites to ensure that a consistent data base is used from which decisions concerning remedial action may be made. To ensure data comparability, EPA standard reference materials will be analyzed to establish that analytical procedures are generating valid data.

The PRPs will be utilizing EPA Method 8270 with an alumina column clean-up for semivolatile organic analyses and EPA Method SW846 for Priority Pollutant Metals plus Vanadium for their metals analyses. The methods used by M&E and the PRPs should yield comparable data as the PRP's methods are analogous to the CLP RAS methods to be used by M&E. Based on the information provided in the PRP's QAPP (Johnson Co., 1995), the detection limits specified for the semivolatile organic compounds listed in the QAPP are the same as the CLP RAS detection limits for those same compounds. A comparison of the metals detection limits was not possible as the PRP's QAPP did not provide a detection limits table for metals, however, the PRP's QAPP does state that all method detection limits used for this project "can be found in OLMO1.9 and ILMO3.0."

M&E's split-sample data will be compared with the PRP's data. As discussed in Section 4.2, the split samples are considered to be field duplicates. Comparability will be assessed by determining the RPD between the M&E data set and the PRP's data set. Data comparison is further discussed in Section 9.

To facilitate comparability, the data sets must be generated by employing methods that use the same type of instrumentation, instrument settings, detection limits, and QC criteria (i.e., calibration %RSD, continuing calibration %D, internal standards, surrogates, and LCS criteria). Efforts will be made to ensure that comparable analytical methods are used by both the PRPs and M&E. Data generated in both sets must also be reported in the same units; solid data must also be reported on a dry weight basis. For this split-sampling program, M&E split-sample data will be reported according to the protocols specified in the RAS methods and DAS methods.

The use of consistent analysis procedures, units, and instrumentation will ensure that comparable split-sample data will be generated to support decisions concerning the remedial design and remedial action. As a starting point for comparing M&E's and the PRP's data sets, a RPD criteria of 50% for solid data will be used. However, since inherent differences may exist between the data sets, which cannot be accounted for solely in consideration of comparability of the data, professional judgement will be used to assess whether deviation from the above stated RPD criteria is warranted.

4.6 PROCEDURES FOR DATA ASSESSMENT

The laboratory QA objectives for RAS methods under the split-sampling program are presented in Table 4-1. Precision values represent variability for replicate measurements of the same parameter and are expressed in terms of the RPD for duplicate (or replicate) measurements of the same samples. Accuracy values include components of both random error (i.e., variability due to imprecision) and systematic error (i.e., bias), and thus reflect the total error for a given measurement, expressed as a percentage of the true value. The QA objectives in Table 4-1 are based primarily on performance data derived from EPA-CLP RAS method validation studies of

calibration, continuing calibration, MS/MSD analyses, surrogate spike recoveries, and other QC criteria. These objectives are not intended to represent data validation criteria, rather, they represent typical performance capability of the methods.

Procedures that will be used to assess M&E's data quality include:

- Accuracy
  - Review %R for spiked samples
  - Ensure blanks are analyte free or qualify data as necessary
  - Review recoveries of PE samples
  - Qualify* laboratory data to identify any systematic errors (bias) that are discovered
- Precision
  - Determine if initial and continuing calibration data are within acceptable criteria
  - Determine if instrument's performance is within acceptable criteria
  - Determine if RPD between MS/MSD data for organics is within acceptable criteria
  - Determine RPDs between field duplicates
  - Determine if RPD between laboratory duplicates for inorganics is within acceptable criteria.
- Completeness
  - Compute fraction of data that remains valid after discarding any non-analyzed or rejected data

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* Data qualifiers are described in *EPA Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analysis* (USEPA, 1988); modified to meet criteria in the current EPA SOWs for RAS analysis of organics (USEPA, 1993b).

- Representativeness and Comparability
  - Determine whether these terms have meaning within the project framework
  - Ensure the use of standard units for reporting results

The procedures listed above for Completeness, Representativeness, and Comparability will be applied to the field data in a similar manner as they will be applied to laboratory data.

5.0 SAMPLING

This section discusses the field procedures that M&E will be performing in support of the RI/FS oversight. The specific sample containers that will be used, sample preservation techniques, and a brief description of sample packaging and shipment procedures are also provided.

5.1 FIELD OVERSIGHT PROCEDURES

M&E will collect and submit sediment split samples for laboratory analysis. A summary of samples being collected by media are presented in this SAP.

The objectives of the field oversight are to ensure that all of the field procedures including the sample handling and preservation protocols are followed according to the PRP's EPA-approved Work Plan. This section discusses in general terms the procedures that should be followed to insure consistent oversight of the PRP's activities and the collection of split samples. The collection of split samples by M&E is discussed in detail in Section 5.2.

5.1.1 General Oversight Procedures

There are a number of preparation, observation, and documentation procedures that are general to all of the field tasks that M&E will observe at Pine Street Canal. These are intended to give general guidance for the field team.

Prior to Field Oversight. These are tasks that should be completed by the field oversight personnel prior to observing field activities. These are intended to acquaint the individual with the critical aspects of the project and task. These are listed below:

- Review the PRP's work plans, project operations plans (POPs), and standard operating procedures (SOPs) for the activities in question.
- Read M&E's health and safety plan

- Request appropriate PPE, camera and any other equipment required
- Review specific oversight activities

During Field Oversight. These are activities that are conducted by the oversight individuals in the field.

- Record the activities conducted during the oversight in the field logbook. Listed below are the general items that should be recorded for all activities and specific items that should be recorded for each activity.
- Take photographs of PRP's activities
- Some tasks will require the collection of split samples. Split sampling is further discussed in section 5.2.

Field Oversight Documentation. Documentation of oversight activities is an essential part of M&E's activities for this project. M&E must document the activities conducted by the PRPs using a field logbook, photographs and a trip memo completed subsequent to returning from the field.

Field Logbook. The following items should be indicated in the field logbook:

- Arrival and departure dates and times of M&E personnel at the site
- Brief description of weather and temperature
- Names and companies of everyone on site
- Activities conducted by PRPs
- Field measurements made by PRPs
- Calibrations of field instrumentation
- Deviations from the proposed plans and SOPs
- Corrective Actions made by the PRPs

- Difficulties encountered in the field
- Locations of all activities, measurements from fixed objects may be necessary
- Decontamination procedures
- Health and safety procedures
- Overall quality of the work
- Additional items specific to each task

Photographs. M&E will compile a photo log as part of this project. The following are general items that might be photographed as part of the field oversight:

- Locations of field activities
- Equipment used for conducting field activities
- Decontamination procedures
- Deviations from proposed plans and SOPs
- Equipment calibration set up
- Site soil, groundwater, or surface water
- Equipment failures

Note: During oversight, photographs will be listed by frame number on a separate page for each field activity.

Field Oversight Reports. Upon returning from the field the oversight team will prepare a report describing the activities conducted by the PRPs and observed by M&E. This memo will highlight any deviations from approved plans and recommendations for corrective actions. If any split samples are collected, these will be documented in the oversight report.

5.2 SPLIT SAMPLING

This section discusses the sampling objectives, procedures, sample containers to be used, and the sample preservation techniques for the collection of split samples.

5.2.1 Sampling Objectives

M&E will provide technical oversight of field split sampling activities during Phase IIB soil sampling to ensure the sampling procedures adhere to the PRP's EPA-approved Work Plan. This activity will serve to assure the integrity of the samples collected and the validity of the data generated. M&E will obtain split samples from the soil collected by the PRPs during soil sampling and toxicity testing. The split samples collected will be submitted for RAS and DAS analysis. Data generated from M&E's split samples will be used for evaluation and comparison to the data obtained by the PRPs.

The objective of split sampling is to provide samples representative of those collected by the PRP's contractors. Oversight split-sample data that confirms the PRP's results increases the confidence level of the PRP's data.

Table 5-1 presents the proposed number of split samples to be collected and the analyses to be conducted, by media. Table 5-2 summarizes the sample parameters, containers, and preservation requirements that will be followed by M&E for solid samples collected during the split-sampling program.

5.2.2 Samples to be Collected

The specific locations that will be sampled will be determined based on direction provided by the EPA RPM, the actual sampling schedule of the PRPs, the days on which M&E personnel have been directed to provide technical oversight, the site conditions, and on the technical judgement of field personnel.

TABLE 5-1. SUMMARY OF ENVIRONMENTAL SAMPLES AND ANALYSES

Parameter	Method	No. of Locations	No. of Samples	PE Samples	Field QC Samples: Trip Blanks	Field QC Samples: Equipment Blanks	Field QC Samples: Field Duplicates	Field QC Samples: MS/MSD Samples	Total Collected
SEDIMENT SAMPLING AND TOXICITY SAMPLING									
Sediment Semivolatiles	CLP-RAS	10	10	1	--	1	1	1	14
Sediment Metals and CN-	CLP-RAS	10	10	2	--	1	1	1	16
Sediment Grain Size	DAS	10	10	--	--	--	1	--	11

NOTES:
CLP - Contract Laboratory Program
DAS - Delivery of Analytical Services
RAS - Routine Analytical Services

TABLE 5-2. SAMPLING PARAMETERS, METHODS, CONTAINERS, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR SAMPLES COLLECTED AT PINE STREET CANAL SITE

PARAMETER	CONTAINER (2)	PRESERVATIVE (3)	HOLDING TIME
SEDIMENT SAMPLES			
DAS ANALYSES (1)			
Grain Size	1-250 ml glass	Cool to 4 °C	14 Days
RAS ANALYSES (4)			
Semivolatile Organics	1-250 ml glass	Cool to 4 °C	7 Days to Extraction; 40 days to Analysis
Metals and CN-	1-250 ml glass	Cool to 4 °C	6 Months, 28 days for Mercury, and 14 days for Cyanide

NOTES:
1 - Methods for DAS analyses are based on DAS Specifications.
2 - Containers cleaned to meet EPA series 300 requirements. All glass containers have Teflon-lined lids
3 - All methods are cooled to 4 °C
4 - Methods are all based on EPA RAS Methods

Sediment samples for chemical analysis will be collected by the PRPs at the same time as the sediment samples for toxicity testing are collected. M&E will be splitting samples for chemical analysis and providing oversight of the toxicity testing sample collection procedures.

The PRPs will be collecting sediment samples from undisturbed sediments collected from 0 -5 cm below the sediment surface in areas 3, 6, and 7, and from 0 - 10 cm below the sediment surface in areas 1, 2, 4, 5, and 8. Based on previous sampling experience at the site, it is expected that sediment collected at the site will be high in moisture content and non-homogenous. The samples for chemical analyses will be collected using an Ekman Grab sampler. Excess water will be decanted from the sampler and debris, plant material, and large benthic organisms will be removed. The sample material will be composited to ensure that each aliquot is equivalent and placed in sample bottles, which will then be refrigerated. In the event that the Ekman Grab sampler does not provide enough sample volume, a ballcheck corer with a 3" diameter acrylic core liner or a hand corer with a 2" or 2.5" core liner will be used. The capped cores will be stored upright in a cooler. Supernatant will be removed by decanting or using a bulb syringe. The core will be extruded, composited, and placed in sample bottles. M&E split samples will be obtained from the composited material in the same manner as the PRP's sample.

After sample collection, the samples will be screened in the field by the PRPs using the Quantix Immunoassay Labstation to determine total PAH and a Spectrace 9000 Field Portable X-Ray Fluorescence Unit to determine lead, zinc, and copper. The field screening results will then be compared to the 1994 Phase IA screening data and if the results are outside the 50% criteria, then the location will be resampled. The second sample will undergo the same procedure and if it is also outside the 50% criteria, then the sample with the result closest to the 1994 Phase IA screening data will be sent to the off-site laboratory for analysis. M&E will provide oversight of the screening analysis. Based on the results of the field screening, the M&E split samples, which were collected with the PRP's selected samples, will be shipped to the CLP RAS laboratories.

5.3 FIELD GENERATED QC SAMPLES

To meet the QA objectives of the split-sampling program, field-generated QC samples will be collected by M&E. These samples will include: equipment blanks, field duplicates, MS/MSD, and EPA performance evaluation samples (PE), when available. Equipment blanks and field duplicates will be collected by M&E field personnel at approximately 10% of the number of samples collected for each media or as required by EPA Region I. Equipment blanks will be collected as split samples with the PRP's contractor's equipment blanks. Technical judgement will be used to determine the days and samples for which equipment blanks and field duplicates will be collected. Detailed information on the collection of field QC samples may be found in Section 10.

5.4 SAMPLE CONTAINERS AND PRESERVATION

Table 5-2 summarizes the sample parameters, containers, and preservation requirements that will be followed by M&E for solid samples collected during the split-sampling program.

5.4.1 Sample Preservation

Sample preservation for solid samples consists of refrigeration.

5.5 SPLIT-SAMPLING PROCEDURES

During field activities, M&E personnel will coordinate with the PRP's contractor to obtain split samples. M&E will provide sample containers and materials for packaging and shipping of split samples. All sampling equipment will be provided by the PRP's contractor, who will also collect appropriate sample volumes needed for analysis. Upon sample receipt, M&E is responsible for: sample preservation, labeling, handling, packaging, and shipping. All shipping will be performed in accordance with Section 6 of this document.

The objective of split sampling is to provide samples representative of those collected by the PRP's contractor. Proper split-sampling techniques will ensure that the data obtained can be used with confidence to compare and confirm the analytical results obtained by the PRP's contractors. As such, the order of sample collection, sample volume, containers, and preservatives will be exactly the same as the PRP's contractor's sampling order. However, because of differences between the methods used by M&E to analyze the split samples and the methods specified in the PRP's EPA-approved Work Plan, there may be instances where M&E will have to test and preserve differently than the PRP's contractor. All differences in sampling activities will be noted in detail in the field logbook.

The sampling order for analytical parameters must be exactly the same for each sample fraction split between M&E and the PRP's contractor. Ideally, sample filling should alternate between the PRP's contractor and M&E, with containers sequentially filled by parameter (e.g., semivolatiles and inorganics). If a sample needs to be composited from more than one sampling equipment volume in order to fill the number of sample containers required for both the PRP's contractor samples and M&E split samples, then equal amounts of sample volume will be placed in a stainless steel bowl and mix thoroughly will a stainless steel spoon or trowel. Portions of the composited sample will then be placed in each container until all the sample containers are filled. This method will ensure that each container will have the same mixture of sample as all other containers.

The PRP's sampling procedures are located in Appendix A of the PRP's QAPP (Johnson Co., 1995). M&E will have a copy of this document in the field to ensure that all samples, regardless of sampling method, are collected and composited in accordance with the EPA approved methods.

6.0 SAMPLE CUSTODY AND FIELD DOCUMENTATION

An overriding consideration essential for the validation of environmental measurement data is to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of the sample traceability from collection to shipment, laboratory receipt, and laboratory custody (until proper sample disposal and the introduction of field investigation results as evidence in legal proceedings when pertinent) must be documented. A sample is considered to be in a person's custody if the sample is:

- In a person's actual possession
- In view after being in a person's possession
- Locked so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel

M&E field personnel are responsible for overseeing and supervising the implementation of proper sample custody procedures in the field. M&E field personnel are also designated as the field sample custodian and are responsible for ensuring sample custody until the samples have been transferred to a courier or directly to the laboratory. Once received by the laboratory, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

6.1 CHAIN OF CUSTODY

The chain of custody procedures are initiated in the field following sample collection. The procedures consist of: (1) preparing and attaching a unique sample label and tag to each sample collected, (2) completing the Traffic Report/Chain-of-Custody (TR/COC) form for RAS analyses and the M&E COC for DAS analyses, (3) preparing the M&E COC for subcontracted analyses, and (4) preparing and packing the samples for shipment. These procedures are further described in the following sections.

6.1.1 Sample Labels

Field personnel are responsible for uniquely identifying and labeling all samples collected during a field investigation program. All labeling must be completed in indelible/waterproof ink and securely affixed to the sample container.

For RAS analyses, all sample bottles will be labeled with both an M&E label and an EPA-CLP sample number designated for RAS analyses. Similarly, for any DAS analyses, sample bottles will be labeled with an M&E label and a DAS sample number. The M&E label typically contains the following information:

- Project number
- Unique sample identification number
- Sample location/description number
- Type of analysis to be performed
- Sample volume, container type, and the type of chemical preservation used
- Sampling date and time
- Sampler's initials

The RAS sample number is a unique number that identifies each sample analyzed through the CLP system. The DAS sample number is also a unique number; however, DAS samples are not routed through the EPA-CLP process, but are generated and tracked through the analytical services work assignment (WA #46-1HZZ). Both the RAS and DAS sample numbers are preprinted on adhesive labels. The RAS sample number labels are provided by the regional sample control center (RSCC) for EPA Region I and DAS sample number labels are provided by the M&E DAS Laboratory/Tracking Coordinator. It is the field personnel's responsibility to assign the RAS or DAS sample number correctly, transcribe it accurately on the appropriate

documentation, place the labels on the correct bottles, and transcribe it accurately to the bottle tag. The RAS sample number label contains the following information:

- An alpha-numeric sample identification number as assigned by the RSCC.
- Type of analysis to be performed

The DAS sample number label will contain only an alpha-numeric sample identification number as assigned by the M&E Laboratory/Tracking Coordinator and can be used for any type of DAS analyses. If a sample number is used but there are additional sample stickers with that number remaining, these extra labels will be destroyed. Unused numbers may be retained for future use. An attempt should be made to use the numbers consecutively to avoid confusion. An example of the RAS and DAS sample number labels are shown in Figure 6-1.

6.1.2 Sample Tags

Field personnel are responsible for tagging all samples that are collected and submitted for RAS analyses. The sample tag must be completed in indelible/waterproof ink and securely attached to the sample container. An example of a EPA-CLP sample tag is shown in Figure 6-2. Sample tags will not be used for samples submitted for DAS analyses. The sample tag will contain the following information:

- Project Code (work assignment number)
- Unique sample identification number (also called the station location)
- Sampling date and time
- Designate: composite or grab sample
- Preservative: yes or no
- Type of analysis to be performed
- Signature of the person packaging the sample

AAL97 - EXTRACTABLE

AAL97 - EXTRACTABLE

AAL97 - EXTRACTABLE

AAL97 - EXTRACTABLE

AAL97 - VOA

AAL97 - VOA

AAL97

AAL97

AAL97

AAL97

ORGANIC SAMPLE NUMBERS

MAT088 - TOTAL METALS

MAT088 - TOTAL METALS

MAT088 - CYANIDE

MAT088 - CYANIDE

MAT088

MAT088

MAT088

INORGANIC SAMPLE NUMBERS

DAS Sample No.
DAM

DAS Sample No.
DAM

DAS Sample No.
DAM

DAS Sample No.
DAM

DAS Sample No.
DAM

DAS Sample No.
DAM

DAS SAMPLE NUMBERS

FIGURE 6-1. CLP SAMPLE NUMBERS (RAS & DAS)

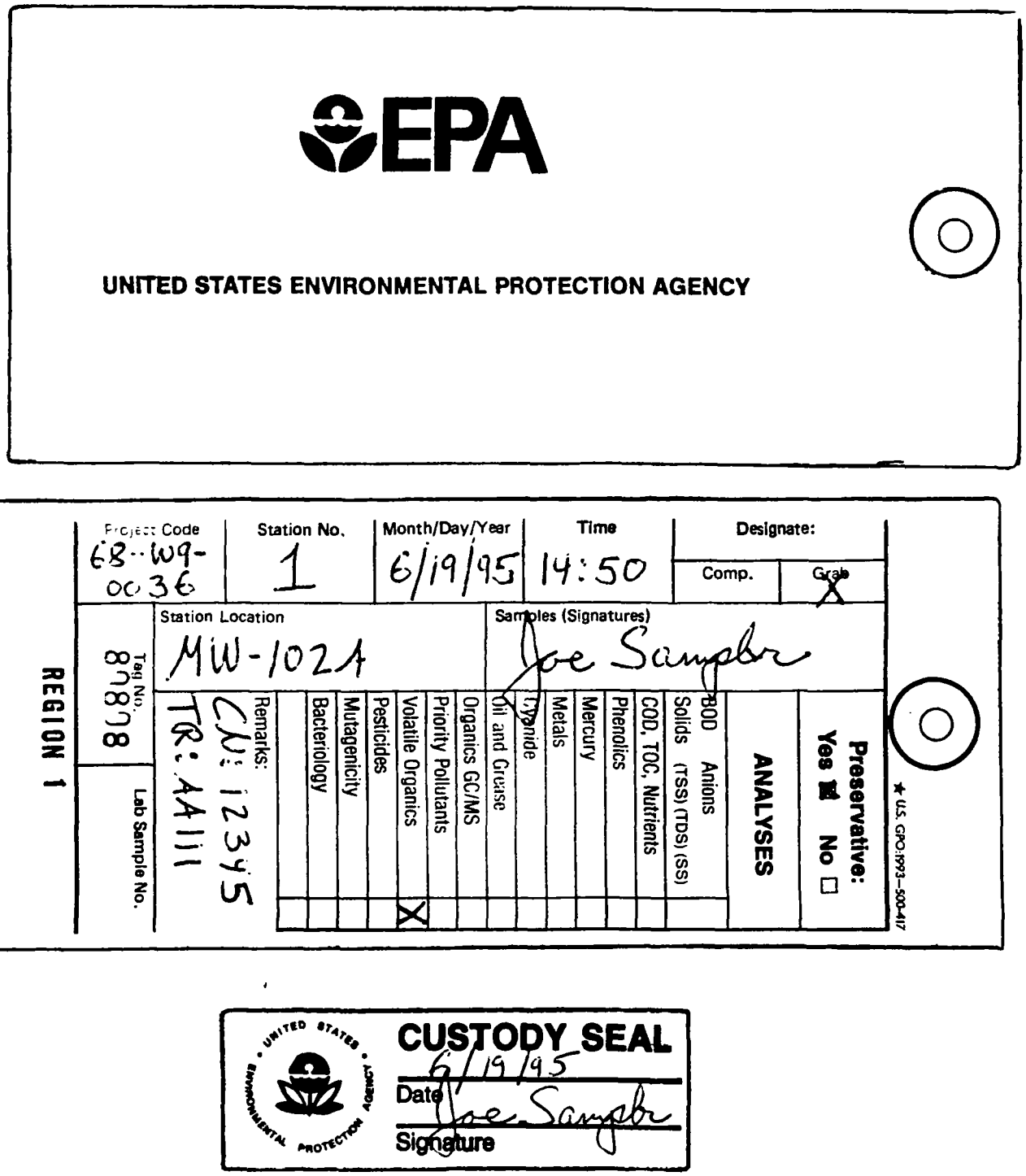


FIGURE 6-2. EPA-CLP SAMPLE TAG AND CUSTODY SEAL

- RAS sample number
- Case number

For each sampling event, a case number is assigned by SMO for RAS samples and by the M&E Laboratory/Tracking Coordinator for DAS samples. Although the DAS case number is assigned by the M&E Laboratory/Tracking Coordinator, the RSCC is notified of the assigned case number and appropriate number of samples to be collected prior to sample collection. From this point on the tracking of DAS samples is the responsibility of the M&E Laboratory/Tracking Coordinator.

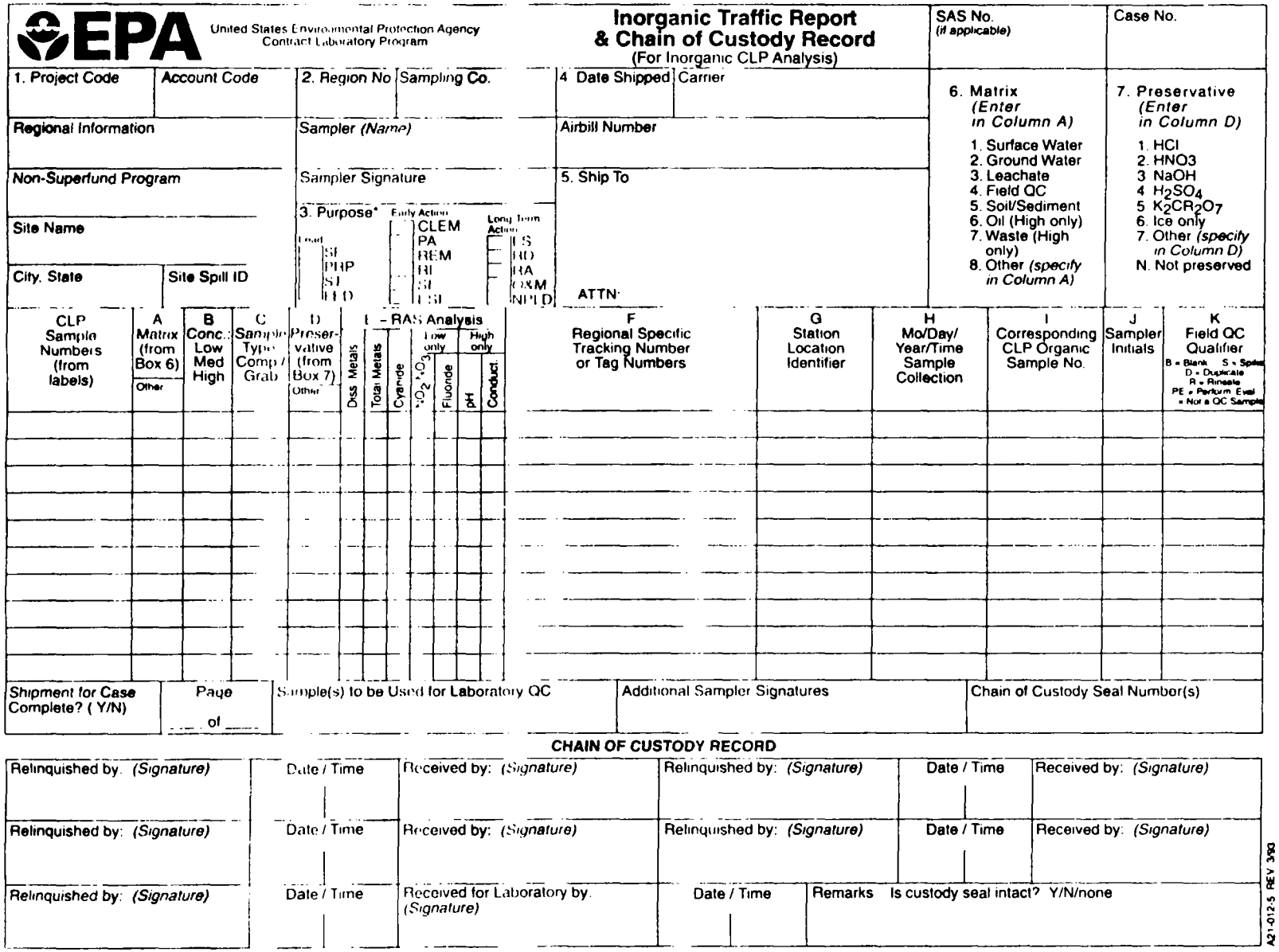
The RAS case number is five digits in length. The DAS case number is a four digit number followed by an assigned letter (M for M&E). The case number allows for tracking of samples and maintains site confidentiality. No reference to the site name will be shown on paper work.

6.1.3 Custody Seal

Custody seals will be secured across shipping container openings to ensure content integrity. The seals will contain both the date and the signature of the person affixing them and must be completed in indelible/waterproof ink. An example of a custody seal is shown in Figure 6-2.

6.1.4 EPA-Chain of Custody (COC) Forms

For inorganic and organic analyses, TR/COCs (Figures 6-3 and 6-4, respectively) must be completed for each sample set submitted for RAS analysis. An M&E COC (Figure 6-5) must be completed for each sample set submitted for DAS analyses and analyses sent to the RSCC. These forms are maintained as a record of sample collection, transfer, shipment, and receipt by the laboratory. These forms also contain pertinent information concerning sampling locations, dates, and times; signatures of at least one sampling team member; types of samples collected along with a unique sample identification number; the number of samples collected and shipped for analysis in each lot; the project name and number; and the name of the laboratory to which



EPA United States Environmental Protection Agency
Contract Laboratory Program

Inorganic Traffic Report & Chain of Custody Record
(For Inorganic CLP Analysis)

SAS No. *(if applicable)*

Case No.

1. Project Code | Account Code | 2. Region No | Sampling Co. | 4 Date Shipped | Carrier

Regional Information | Sampler *(Name)* | Airbill Number

Non-Superfund Program | Sampler Signature | 5. Ship To

Site Name | 3. Purpose* (Early Action: CLEM, PA, REM, RI, SI, FFS; Long Term Action: FS, RD, RA, O&M, NPLD; Lead: SI, PHP, SI, FFD)

City, State | Site Spill ID | ATTN:

6. Matrix *(Enter in Column A)*
1. Surface Water
2. Ground Water
3. Leachate
4. Field QC
5. Soil/Sediment
6. Oil (High only)
7. Waste (High only)
8. Other *(specify in Column A)*

7. Preservative *(Enter in Column D)*
1. HCl
2. HNO3
3 NaOH
4 H_2SO_4
5 $K_2CR_2O_7$
6. Ice only
7. Other *(specify in Column D)*
N. Not preserved

CLP Sample Numbers (from labels)	A Matrix (from Box 6) / Other	B Conc.: Low Med High	C Sample Type: Comp / Grab	D Preservative (from Box 7) / Other	E – RAS Analysis: Diss Metals	Total Metals	Cyanide	NO_2 NO_3 (Low only)	Fluoride (Low only)	pH (High only)	Conduct (High only)	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier	H Mo/Day/ Year/Time Sample Collection	I Corresponding CLP Organic Sample No.	J Sampler Initials	K Field QC Qualifier (B = Blank, S = Spike, D = Duplicate, R = Rinsate, PE = Perform Eval, = Not a QC Sample)

Shipment for Case Complete? (Y/N) | Page ___ of ___ | Sample(s) to be Used for Laboratory QC | Additional Sampler Signatures | Chain of Custody Seal Number(s)

CHAIN OF CUSTODY RECORD

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks Is custody seal intact? Y/N/none	

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***SEE REVERSE FOR PURPOSE CODE DEFINITIONS**

FIGURE 6-3. RAS INORGANIC TRAFFIC REPORT/CHAIN OF CUSTODY FORM

8/4

FIGURE 6-4. RAS ORGANIC TRAFFIC REPORT/CHAIN OF CUSTODY FORM

EPA United States Environmental Protection Agency
Contract Laboratory Program

Organic Traffic Report & Chain of Custody Record

(For Organic CLP Analysis)

SAS No (if applicable)

Case No

1. Project Code | Account Code

2. Region No. | Sampling Co.

4. Date Shipped | Carrier

Regional Information

Sampler *(Name)*

Airbill Number

Non-Superfund Program

Sampler Signature

5. Ship To

Site Name

3. Purpose*
Lead: ☐ SF ☐ PRP ☐ ST ☐ FED
Early Action: ☐ CLEM ☐ PA ☐ REM ☐ RI ☐ SI ☐ ESI
Long-Term Action: ☐ FS ☐ RD ☐ RA ☐ O&M ☐ NPLD

City, State | Site Spill ID

ATTN:

6. Matrix *(Enter in Column A)*
1 Surface Water
2 Ground Water
3 Leachate
4 Field QC
5 Soil/Sediment
6 Oil (High only)
7 Waste (High only)
8 Other *(Specify in Column A)*

7. Preservative *(Enter in Column D)*
1 HCl
2 HNO3
3 NaHSO4
4 H2SO4
5 Ice only
6 Other *(Specify in Column D)*
N Not preserved

CLP Sample Numbers (from labels)	A Matrix (from Box 6) Other	B Conc.: Low Med High	C Sample Type: Comp./ Grab	D Preser-vative (from Box 7) Other:	E RAS Analysis: VOA	BNA	Pest/ PCB	High only ARO/ TOX	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier	H Mo/Day/ Year/Time Sample Collection	I Corresponding CLP Inorganic Sample No	J Sampler Initials	K Field QC Qualifier (R - Rinsate; D - Duplicate; B - Blank; S - Spike; PE - Performance Eval; N - Matrix Spike Sample)

Shipment for Case Complete? (Y/N)

Page ____ of ____

Sample(s) to be Used for Laboratory QC

Additional Sampler Signatures

Chain of Custody Seal Number(s)

CHAIN OF CUSTODY RECORD

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by *(Signature)*	Date / Time	Received by *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by *(Signature)*	Date / Time	Received by *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by *(Signature)*	Date / Time	Remarks: Is custody seal intact? Y/N/none	

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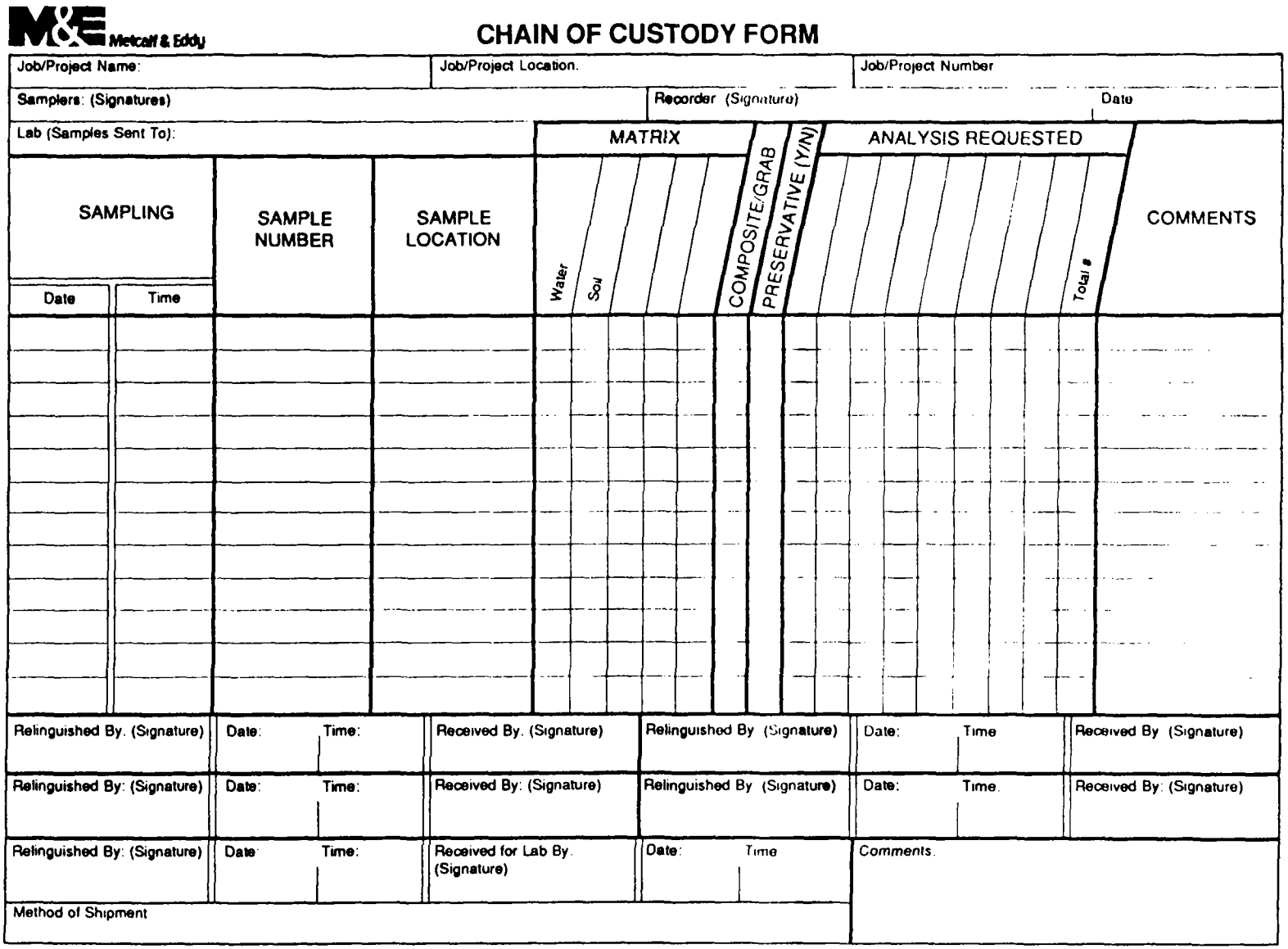
SEE REVERSE FOR ADDITIONAL STANDARD INSTRUCTIONS
***SEE REVERSE FOR PURPOSE CODE DEFINITIONS**



M&E Metcalf & Eddy

CHAIN OF CUSTODY FORM

Job/Project Name:	Job/Project Location.	Job/Project Number
Samplers: (Signatures)	Recorder (Signature)	Date
Lab (Samples Sent To):	MATRIX	ANALYSIS REQUESTED

SAMPLING		SAMPLE NUMBER	SAMPLE LOCATION	MATRIX					COMPOSITE/GRAB	PRESERVATIVE (Y/N)	ANALYSIS REQUESTED								COMMENTS
Date	Time			Water	Soil													Total #	

Relinquished By: (Signature)	Date: Time:	Received By: (Signature)	Relinquished By (Signature)	Date: Time	Received By (Signature)
Relinquished By: (Signature)	Date: Time:	Received By: (Signature)	Relinquished By (Signature)	Date: Time	Received By: (Signature)
Relinquished By: (Signature)	Date: Time:	Received for Lab By: (Signature)	Date: Time	Comments:	
Method of Shipment					

Distribution: **Original** to Lab **Copy 1** to Field Files, **Copy 2** to Project Manager

FIGURE 6-5. METCALF & EDDY CHAIN OF CUSTODY FORM

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the samples are being sent. They must be completed to ensure proper transfer of custody from the time of sample collection to analysis. The appropriate copies must be sent to the SMO, RSCC, and the laboratory.

6.1.5 Transfer of Custody

Samples will be accompanied by an approved and completed TR/COC or M&E COC form during each step of custody transfer, and shipment. When physical possession of samples is transferred, both the individual relinquishing the samples and the individual receiving them will sign, date, and record the time on the COC form. In the case of sample shipment by an overnight courier, a properly prepared airbill (see section 6.2) will serve as an extension of the TR/COC or M&E COC form while the samples are in transit.

At the start of business the day following shipment, field personnel will either notify the SMO or RSCC of RAS sample shipments, by telephone. This notification enables SMO to track the shipment of samples from the field to the laboratory and ensure timely receipt of the samples at the laboratory. The following information should be reported to the SMO and documented:

- Field team person's name, phone number, and EPA region
- Case number of the project
- Batch numbers (PCDD/PCDFs only)
- Exact number(s), matrix(ces), and concentration(s) of samples shipped
- Laboratories to which samples were shipped
- Analyses required
- Carrier name and airbill number(s) for the shipment
- Method of shipment (e.g., overnight)
- Date of shipment

- Information on completions, changes, delays, continuations, etc., pertinent to the Case and sampling project

If the RAS sample shipment is made after 5:00 p.m. eastern standard time (EST), the SMO will be notified at the start of business the next day (8:00 a.m. EST). The SMO will also be notified by 12:00 p.m. EST Friday for RAS sample shipments that will be received at the laboratory on Saturday.

Appropriate copies of the TR/COC must be sent to the SMO to document collection of RAS samples to be analyzed through the EPA-CLP system. Upon completion of each sampling round, copies of all TR/COC and a copy of the appropriate DQO summary form (Figure 6-6) will be sent to the following addresses:

Attn: Christine Clark
Regional Sample Control Coordinator
U.S. Environmental Protection Agency
Environmental Services Division
60 Westview Street
Lexington, MA 02173-3185
(617) 860-4615

U.S. EPA Contract Laboratory Program
Sample Management Office
P.O. Box 818
Alexandria, VA 22313
(703) 519-1360

For DAS analyses, it is not necessary to contact SMO, but rather the M&E Laboratory/Tracking Coordinator is informed of the sample shipment information by the Field Team Leader the day following shipment; copies of the M&E COC forms are then provided within a short period of time. Notification of sample shipment enable the M&E Laboratory/Tracking Coordinator to track the shipment of DAS samples from the field to the laboratory and ensures timely receipt of the samples at the laboratory. Within one day of sample receipt, the DAS laboratory is required to transmit (via facsimile) a confirmation of sample receipt to the M&E Laboratory/Tracking Coordinator. Upon receipt of data packages from the laboratory, a Data Receipt Notification Form (Figure 6-7) is filled out and submitted to the RSCC by facsimile.

5-2

DQO SUMMARY FORM

1. SITE

NAME _____
LOCATION _____
NUMBER _____

EPA REGION _____

PHASE _____
RI1 RI2 RI3 ERA FS RD RA
(Circle One)

2. MEDIA *(Circle one)*	SOIL	GW		SW/SED	AIR	BIO	OTHER
3. USE *(Circle All That Apply)*	SITE CHARAC. (H&S)	RISK ASSESS.	EVAL ALTS.	ENG'S DESIGN	PRP DETER	MONITORING REMEDIAL ACTION	OTHER

4. OBJECTIVE _____

5. SITE INFORMATION
AREA _____ DEPTH TO GROUNDWATER _____
GROUNDWATER USE _____
SOIL TYPES _____
SENSITIVE RECEPTORS _____

6. DATA TYPES *(Circle Appropriate Data Types)*

A. ANALYTICAL DATA

pH	PESTICIDES	TOX
CONDUCTIVITY	PCB	TOC
VOA	METALS	BTX
ABN	CYANIDE	COD
TCLP	_____	_____

B. PHYSICAL DATA

PERMEABILITY	HYDRAULIC HEAD
POROSITY	PENETRATION TEST
GRAIN SIZE	HARDNESS
BULK DENSITY	_____
_____	_____

7. SAMPLING METHOD *(Circle Method(s) To Be Used)*

ENVIRONMENTAL	BASED	GRAB	NON INTRUSIVE	PHASED
SOURCE	GRID	COMPOSITE	INTRUSIVE	_____

8. ANALYTICAL LEVELS *(Indicate Level(s) And Equipment & Methods)*
LEVEL 1 FIELD SCREENING — EQUIPMENT _____
LEVEL 2 FIELD ANALYSIS — EQUIPMENT _____
LEVEL 3 NON-CLP LABORATORY — METHODS _____
LEVEL 4 CLP/RAS — METHODS _____
LEVEL 5 NON STANDARD _____

9. SAMPLING PROCEDURES
BACKGROUND — 2 PER EVENT OF _____
CRITICAL (LIST) _____
PROCEDURES _____

10. QUALITY CONTROL SAMPLES *(Confirm Or Set Standard)*

A. FIELD
COLLOCATED — 5% OR _____
REPLICATE — 5% OR _____
FIELD BLANK — 5% OR _____
TRIP BLANK — 1 PER DAY OR _____

B. LABORATORY
REAGENT BLANK — 1 PER ANALYSIS BATCH OR _____
REPLICATE — 1 PER ANALYSIS BATCH OF __ _____
MATRIX SPIKE — 1 PER ANALYSIS BATCH OR _____
OTHER _____

11. BUDGET REQUIREMENTS
BUDGET _____ SCHEDULE _____
STAFF _____

CONTRACTOR _____ PRIME CONTRACTOR _____
SITE MANAGER _____ DATE _____

FIGURE 6-6. DQO SUMMARY FORM

DAS DATA RECEIPT NOTIFICATION

Site Name: _____

Contractor: _____

DAS#: _____

Lab Code: _____

SDG No.: _____

Number of Samples: _____

DAS Sample Numbers: _____

_____

_____

_____

Blanks: _____

Duplicates: _____

PE Samples: _____

Ship to Lab Date: _____

Data Package Receipt Date: _____

CSF Receipt Date: _____

Matrix	Parameter	Sample Count
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

FIGURE 6-7. DATA RECEIPT NOTIFICATION FORM

6.1.6 Laboratory Sample Custody Procedures

Upon sample receipt, the laboratory representative responsible for accepting incoming sample shipments must compare the samples received against the list on the TR/COC or M&E COC forms and test the temperature of the samples to determine if they are at a temperature of 4°C when received. If the samples were damaged during transfer or were not at 4°C±2°C, the remaining samples must be carefully examined to determine whether they were affected. Any samples so affected or not maintained at 4°C must also be considered damaged and the pertinent information noted on the TR/COC or M&E COC forms (specifying which samples were damaged and that the samples were removed from the sampling program). Field personnel are notified through the SMO and the RSCC of any sample damage as soon as possible so that resampling can take place or the testing program can be modified.

The laboratory representative must also: (1) verify that sample holding times have not been exceeded, (2) sign and date the TR/COC or M&E forms, (3) list the received samples in the laboratory sample master log-in book that will, at a minimum, contain the following information:

- Project identification number
- Sample numbers
- Tag numbers
- Type of samples
- Date received by transfer personnel (i.e. by the overnight carrier)
- Date received by the laboratory

The laboratory representative must also: notify the Laboratory Manager of sample arrival; alert the Laboratory Manager of any analyses requiring immediate attention due to short holding times; and store the samples according to the requirements of the analytical protocols.

6.1.7 Laboratory Sample Tracking Procedures

Following documentation of the receipt of samples in the laboratory, the samples are tracked from storage through the laboratory analytical system until analysis is complete and the samples are sent for disposal.

6.2 SAMPLE PACKAGING AND SHIPPING

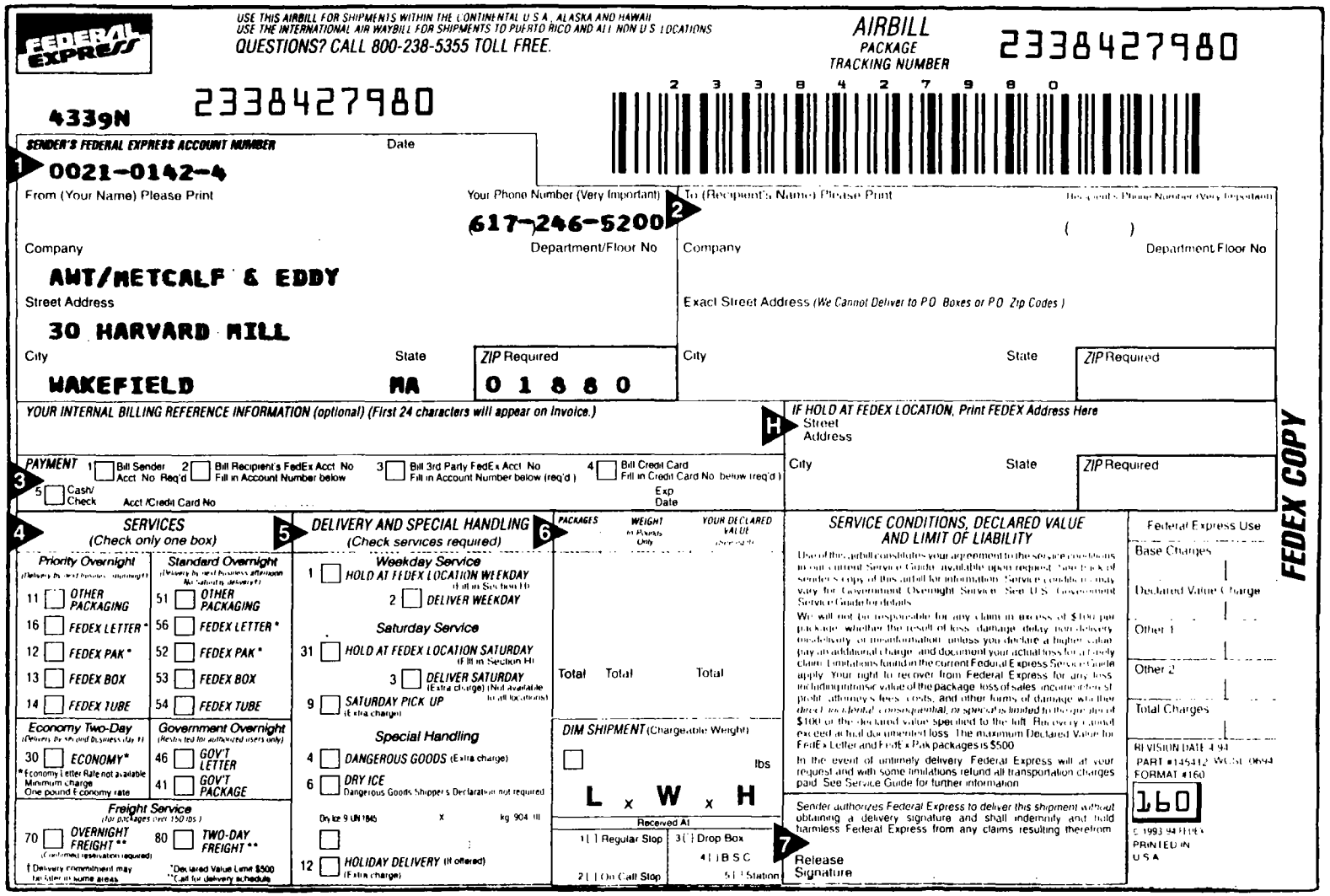
Following sample collection, all samples will be assembled at an on-site location for batching and paperwork checks. At this central location, like sample types are matched (i.e., solids, liquids, etc.) with similar sample types from all sample locations. Labels, tags, and logbook information are checked to be sure there is no error in sample identification. The samples are packaged to prevent breakage and/or leakage and the shipping containers are labeled in accordance with the Department of Transportation (DOT) regulations for transport.

All samples will be shipped directly to the laboratory via overnight carrier. M&E field personnel will determine whether it is best to transport the packages directly to the shipping office or to arrange for on-site pick-up. For each sample shipment, an overnight airbill must be properly completed. An example of an overnight carrier (i.e., Federal Express) airbill used when shipping non-hazardous and hazardous samples is shown in Figures 6-8 and 6-9, respectively.

In order to ensure safe, secure delivery of all collected samples to the EPA-CLP or DAS laboratories, packaging and shipping procedures have been prepared. These procedures are developed so that resulting shipments will comply with applicable DOT regulations for air or surface transportation.

Prior to shipping samples, M&E field personnel will evaluate whether the samples are considered to be non-hazardous or hazardous based on M&E's standard operating procedure for sample shipment (Appendix B). Note that the definitions of hazardous and non-hazardous, as

FIGURE 6-8. FEDERAL EXPRESS SHIPPING BILL FOR NON-HAZARDOUS SAMPLES



FEDERAL EXPRESS

USE THIS AIRBILL FOR SHIPMENTS WITHIN THE CONTINENTAL U.S.A., ALASKA AND HAWAII
USE THE INTERNATIONAL AIR WAYBILL FOR SHIPMENTS TO PUERTO RICO AND ALL NON U.S. LOCATIONS
QUESTIONS? CALL 800-238-5355 TOLL FREE.

AIRBILL
PACKAGE
TRACKING NUMBER
2338427980

4339N 2338427980

SENDER'S FEDERAL EXPRESS ACCOUNT NUMBER
1 0021-0142-4

Date

From (Your Name) Please Print

Your Phone Number (Very Important)
617-246-5200

Company
AWT/METCALF & EDDY

Department/Floor No

Street Address
30 HARVARD MILL

City
WAKEFIELD

State
MA

ZIP Required
0 1 8 8 0

2 To (Recipient's Name) Please Print

Recipient's Phone Number (Very Important)
( )

Company

Department/Floor No

Exact Street Address (We Cannot Deliver to P.O. Boxes or P.O. Zip Codes.)

City

State

ZIP Required

YOUR INTERNAL BILLING REFERENCE INFORMATION (optional) (First 24 characters will appear on invoice.)

H IF HOLD AT FEDEX LOCATION, Print FEDEX Address Here
Street Address

City

State

ZIP Required

3 PAYMENT 1 ☐ Bill Sender Acct No Req'd 2 ☐ Bill Recipient's FedEx Acct No Fill in Account Number below 3 ☐ Bill 3rd Party FedEx Acct No Fill in Account Number below (req'd) 4 ☐ Bill Credit Card Fill in Credit Card No below (req'd)
5 ☐ Cash/Check Acct/Credit Card No Exp Date

4 SERVICES (Check only one box)

Priority Overnight (Delivery by next business morning)
11 ☐ OTHER PACKAGING
16 ☐ FEDEX LETTER*
12 ☐ FEDEX PAK*
13 ☐ FEDEX BOX
14 ☐ FEDEX TUBE

Standard Overnight (Delivery by next business afternoon)
51 ☐ OTHER PACKAGING
56 ☐ FEDEX LETTER*
52 ☐ FEDEX PAK*
53 ☐ FEDEX BOX
54 ☐ FEDEX TUBE

Economy Two-Day (Delivery by 5th day)
30 ☐ ECONOMY*
*Economy Letter Rate not available. Minimum charge: One pound Economy rate

Government Overnight (Restricted to authorized users only)
46 ☐ GOV'T LETTER
41 ☐ GOV'T PACKAGE

Freight Service (for packages over 150 lbs.)
70 ☐ OVERNIGHT FREIGHT** (Confirmed reservation required)
80 ☐ TWO-DAY FREIGHT**
†Delivery commitment may be later in some areas
*Declared Value Limit $500
**Call for delivery schedule

5 DELIVERY AND SPECIAL HANDLING (Check services required)

Weekday Service
1 ☐ HOLD AT FEDEX LOCATION WEEKDAY (Fill in Section H)
2 ☐ DELIVER WEEKDAY

Saturday Service
31 ☐ HOLD AT FEDEX LOCATION SATURDAY (Fill in Section H)
3 ☐ DELIVER SATURDAY (Extra charge) (Not available to all locations)
9 ☐ SATURDAY PICK UP (Extra charge)

Special Handling
4 ☐ DANGEROUS GOODS (Extra charge)
6 ☐ DRY ICE Dangerous Goods Shipper's Declaration not required
Dry Ice 9, UN 1845 x kg 904 III
12 ☐ HOLIDAY DELIVERY (If offered) (Extra charge)

6 PACKAGES | WEIGHT in Pounds Only | YOUR DECLARED VALUE (Required)
Total | Total | Total

DIM SHIPMENT (Chargeable Weight)
☐ lbs
L x W x H

Received At
1 ☐ Regular Stop 3 ☐ Drop Box
2 ☐ On Call Stop 4 ☐ B S C
5 ☐ Station

SERVICE CONDITIONS, DECLARED VALUE AND LIMIT OF LIABILITY

Use of this airbill constitutes your agreement to the service conditions in our current Service Guide, available upon request. Ask for a copy of sender's copy of this airbill for information. Service conditions may vary for Government Overnight Service. See U.S. Government Service Guide for details.

We will not be responsible for any claim in excess of $100 per package, whether the result of loss, damage, delay, non-delivery, misdelivery, or misinformation, unless you declare a higher value, pay an additional charge, and document your actual loss in a timely claim. Limitations found in the current Federal Express Service Guide apply. Your right to recover from Federal Express for any loss, including intrinsic value of the package, loss of sales, income, interest, profit, attorney's fees, costs, and other forms of damage, whether direct, incidental, consequential, or special, is limited to the greater of $100 or the declared value specified to the left. Recovery cannot exceed actual documented loss. The maximum Declared Value for FedEx Letter and FedEx Pak packages is $500.

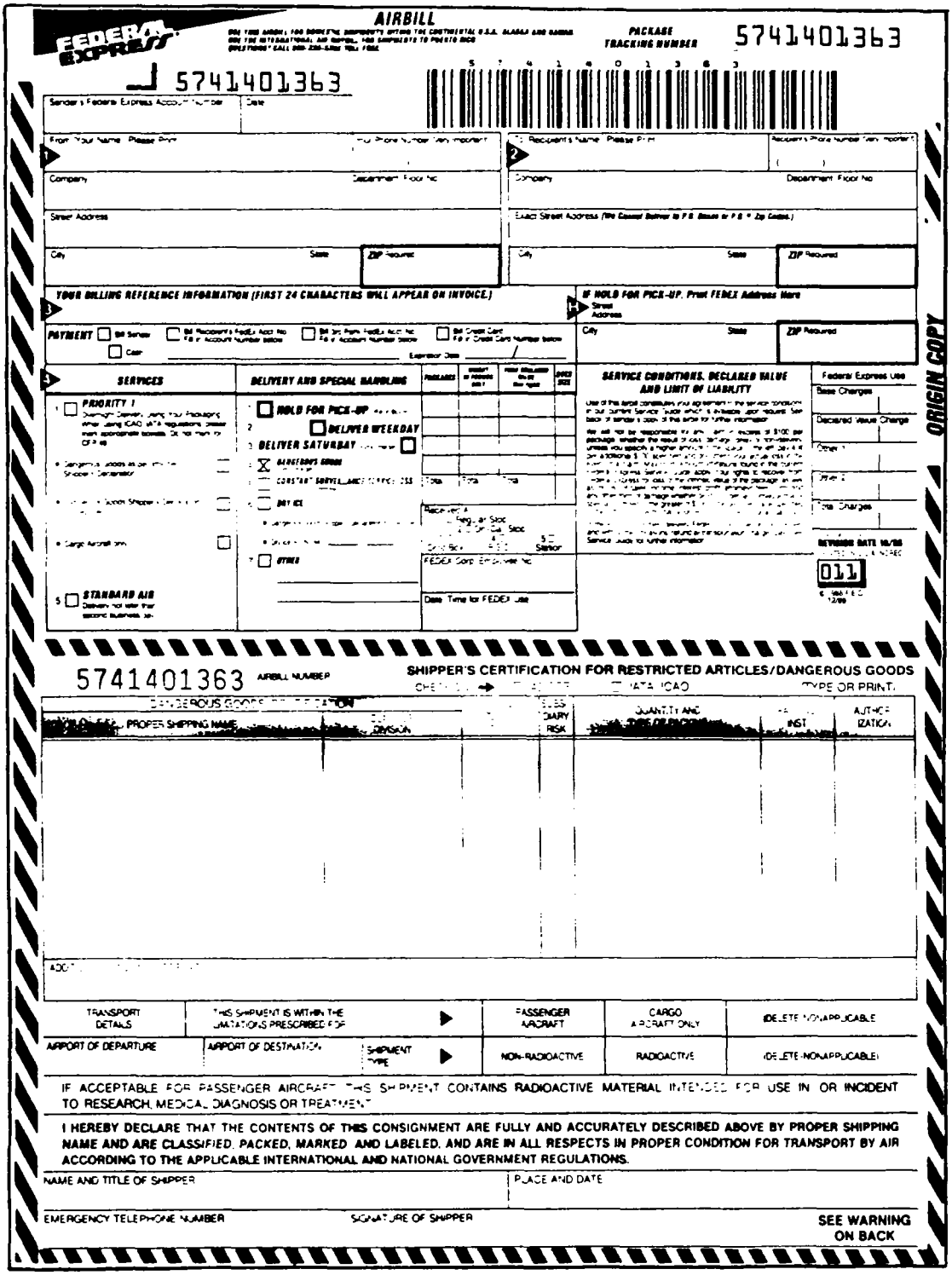
In the event of untimely delivery, Federal Express will, at your request and with some limitations, refund all transportation charges paid. See Service Guide for further information.

7 Sender authorizes Federal Express to deliver this shipment without obtaining a delivery signature and shall indemnify and hold harmless Federal Express from any claims resulting therefrom.
Release Signature

Federal Express Use
Base Charges
Declared Value Charge
Other 1
Other 2
Total Charges

REVISION DATE 4.94
PART #145412 WCSI 0694
FORMAT #160
160
© 1993 94 FEDEX
PRINTED IN U.S.A.

FEDEX COPY



AIRBILL

PACKAGE TRACKING NUMBER 5741401363

5741401363

ORIGIN COPY

SERVICES

DELIVERY AND SPECIAL HANDLING

SERVICE CONDITIONS, DECLARED VALUE AND LIMIT OF LIABILITY

Federal Express Use

5741401363 AIRBILL NUMBER

SHIPPER'S CERTIFICATION FOR RESTRICTED ARTICLES/DANGEROUS GOODS

TYPE OR PRINT

PROPER SHIPPING NAME

CLASS DIVISION

QUANTITY AND TYPE OF PACKING

AUTHORIZATION

TRANSPORT DETAILS

THIS SHIPMENT IS WITHIN THE LIMITATIONS PRESCRIBED FOR

PASSENGER AIRCRAFT

CARGO AIRCRAFT ONLY

AIRPORT OF DEPARTURE

AIRPORT OF DESTINATION

SHIPMENT TYPE

NON-RADIOACTIVE

RADIOACTIVE

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

I HEREBY DECLARE THAT THE CONTENTS OF THIS CONSIGNMENT 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 ON BACK

FIGURE 6-9. FEDERAL EXPRESS SHIPPING BILL FOR HAZARDOUS SAMPLES

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used in regards to sample shipment are based on DOT regulations described in 49 CFR parts 172 and 173. Based on M&E's SOP, all environmental samples being shipped are considered to be hazardous unless pre-existing data (i.e., previous investigations) or field information can be used to evaluate whether the samples are non-hazardous. Unless field collected information indicates otherwise, all environmental samples collected will be treated as non-hazardous aqueous or solid samples for packaging and shipping purposes. Other guidelines to consider when evaluating whether a sample should be considered hazardous includes: 1) is the sample a hazardous material, 2) does the sample possess hazardous characteristics, or 3) is the sample a hazardous substance with a reportable quantity. However, the final determination of how to ship the sample must be made using the M&E SOP for sample shipment (Appendix B).

6.2.1 Hazardous Packaging and Shipping

Due to the nature of the site, and the possibility that environmental samples collected could exhibit hazardous characteristics (ie. high concentrations of coal tar), it may be necessary to ship some samples as hazardous in accordance with the DOT hazardous shipment requirements. The packaging and shipping of these environmental samples and other chemicals must comply with DOT regulations described in 49 CFR Parts 172 and 173 and M&E's SOP in Appendix B for the shipment of environmental samples. Packaging procedures must also include:

- Proper identification and classification of the hazardous materials
- Proper description and shipping name of the materials
- Use of DOT approved shipping containers
- Proper marking of the shipping container to include:
    - Commodity description and DOT labeling
    - "This end up" labels for shipping containers of liquid samples
    - Name and address of shipper
    - Characteristics of hazardous materials such as corrosivity, ignitability, or flammability
- Sample container labeling consistent with shipping papers

- Inclusion of proper shipping papers listing the name and classification of the hazardous material and assurance that the labeling is consistent with the shipping papers

- Samples of highly toxic hazardous materials or pure-product material (i.e., gas, fuel oil, PCB-oil) must be sealed in a paint can prior to shipment to the laboratory; The paint can must be labeled with the sample identification number.

The packaging and shipping criteria have been designed to maintain chain of custody protocol as well as to prevent breakage of the sample containers.

6.2.2 Non-Hazardous Packaging Procedures

General packaging procedures are as follows:

- Place a layer of cushioning material (e.g., vermiculite) in the bottom of the watertight insulated metal or equivalent strength plastic shipping containers.

- Wrap the properly labeled and secured glass sample bottles and 40 ml vials with plastic bubble wrap. Place the wrapped containers into watertight zip-lock bags and seal the bags closed. Plastic sample containers are placed in zip-lock bags without bubble wrap.

- Place sample bottles (top side up) into the shipping container arranging the bottles so that the glass bottles are surrounded by plastic bottles.

- Using the necessary packing material, pack the sample bottles to ensure that they do not shift during transport.

- Place a sufficient amount of ice (either ice cubes packed in sealed zip-lock bags or frozen gel packs) on top of and around the sample bottles. Fill any void spaces in the shipping container with packing/cushioning material.

- Seal the appropriate TR/COC or M&E COC forms in a zip-lock bag and tape it securely to the inside of the shipping container lid.

- Close and lock/latch the shipping container. If the shipping container used is a camping type cooler, tape the drain plug closed to prevent any leakage of water as the ice packs melt during transport.

- Apply several wraps of COC tape around the shipping containers perpendicular to the seal to ensure that the lid remains closed if the latch is accidentally released or damaged during shipment. Add dated and signed COC seals and wrap with clear tape over the COC seals and at least once around the shipping container. Do not obscure any stickers or labels on the shipping container with the COC tape.

- Place a completed overnight carrier airbill on the lid of the shipping container. Include the name, address, and telephone number of the receiving laboratory and the return address and telephone number of the shipper on the airbill.

- Place a "This End Up" label on all four sides of the shipping container.

- Each shipping container must not weigh more than 150 pounds.

- Prior to shipment, the carrier should be notified adequately ahead of time to schedule the hazardous material shipment.

6.3 DOCUMENTATION

A simple program has been designed to ensure that field analyses yield valid, useful data. Field personnel must maintain a concise, detailed field logbook containing accounts of all field activities and actions taken as well as written documentation of observations made. All sampling procedures, instrument calibration, and information pertinent to sampling conditions, progress, and field data collection must be documented following a prescribed set of guidelines. The documentation serves as a permanent and traceable record of all activities related to the field oversight activities and the split-sampling program. The record must be legible and accessible to allow ease in verifying sampling activities and addressing future questions which may arise concerning such issues as sample integrity, sample traceability, etc.

6.3.1 Sample Designation/Identification

The establishment of a standard sample designation/labeling protocol is essential to ensure adequate QA/QC in regards to the traceability of samples and their associated analytical data. Proper labeling allows for the tracking of samples beginning from the time of sample collection

through analysis, and following project completion should future data correlation be deemed necessary. The proper labeling of samples is also critical in ensuring that samples are analyzed within the required holding times.

All samples will be identified using a unique sample identification scheme suitable to the project and sampling protocol. The numbering scheme for the split-sampling program will be devised by the Project Engineer and Project Chemist. The sample location number and the EPA sample number will be recorded on the TR/COC or M&E COC forms accompanying each sample shipment submitted to the EPA-CLP or DAS laboratories.

6.3.2 Corrections to Documentation

All documentation must be recorded in permanent ink. Corrections to errors in documentation or recorded calculations will be made by first striking out the error with a single line so as not to obliterate the original entry. Then the replacement entry or value will be inserted where appropriate. The person originating the change will initial each separate change and date it. All revisions, deletions, and changes must be made in indelible ink.

6.3.3 Photographs

M&E field personnel will document, through the use of color photography, various field and sampling procedures as deemed necessary. Examples of items that may require such photographic documentation include:

- Boring procedures
- Sample locations and surrounding environments
- Physical appearance of environmental samples

6.3.4 Records

M&E field personnel have the responsibility to maintain the daily documents pertaining to sample identification and control. Special emphasis is placed on the logbooks and completeness and accuracy of the logbooks. Project logbooks, field logbooks, field data forms, and EPA-CLP COC forms must contain entries made with indelible ink that are dated, signed, and contain statements that are legible, accurate, and descriptive of project activities. Because the logbooks, data forms, and EPA-CLP COC forms provide the basis for future reports, the records must contain accurate facts and observations. Language must be objective, factual, and free of personal interpretations or other terminology that may prove inappropriate.

6.3.4.1 Field Logbooks. Field logbooks will be maintained by M&E field personnel. The field logbook is a bound book with consecutively numbered pages; no pages may be skipped when filling in the logbooks. The integrity of field documentation is further ensured by the use of field logbooks containing paper treated to repel the rain or any other aqueous splashings experienced during field documentation. Should more than one field logbook be required, they will be numbered sequentially. All logbooks will be placed in the ARCS' files for this project upon completion of field activities. The front of each field logbook will contain the following information:

- Project name and number
- Name of the contract under which the project is being conducted
- Date(s) of use

The field logbook will contain a diary of all pertinent field activities performed by M&E. Standard information recorded in the field logbook will include: general observations made in the field, identification and calibration of instruments used, and field data.

An example of typical field information so recorded may include:

- Date and time of personnel entries onsite, weather conditions, air temperature
- List of the personnel present onsite during each sampling day, including all M&E personnel, PRP's personnel, subcontractors, and visitors
- Tasks accomplished by day and personnel performing tasks
- List of equipment decontaminated by M&E and reference to the procedures used
- List of start/stop times of all activities, such as boring, decontamination of equipment, well sampling, etc...
- QC samples associated with the environmental samples collected
- Phone calls made: subject, person spoken with, time, phone number, etc...
- Sample preservation techniques and tests checking for oxidizing agents, sulfide, or carbonates
- Air monitoring information gathered by M&E
- Level of personnel protection mandated (e.g., Level B, C, D)
- List of PRP's samples and M&E's split samples collected by media, parameter, sample location, and sample depths
- Specific split sample description (matrix, sample volume color turbidity, consistency, odor, evidence of contamination)
- Descriptions and corresponding numbers of any photographs taken
- Documentation of all packaging, shipping sample custody, and related form numbers
- Comments relative to any problem areas that occurred during the day's activities, their final resolution, and any anticipated impact on the outcome of the field investigation. Notes of conversations with all coordinating officials

6.3.4.2 Project Logbook. A unique project-specific logbook will be maintained during the technical oversight of field and split-sampling activities. The logbook will be comprised of a

bound book with consecutively numbered pages. Should more than one project logbook be required, they will be numbered sequentially.

In general, the project logbook will contain information summarized from the field logbooks. The project logbook need not list all the actual field data and instrument calibration information; references to other documents that contain specific field activity descriptions, outlines of any administrative occurrences that have affected the field work for any given sampling activity as well as a summary of the field activities, must be documented in the project logbook and kept up-to-date on a daily basis.

7.0 CALIBRATION PROCEDURES AND FREQUENCIES FOR ANALYTICAL INSTRUMENTS

Calibration of the laboratory and field instrumentation is essential to ensure the highest quality data possible. The following sections briefly describe the calibration procedures, however, detailed calibration procedures are provided in the analytical procedures and individual instrument and field monitoring device manuals.

7.1 LABORATORY INSTRUMENTATION

Instruments and equipment used in the EPA-CLP laboratories are controlled by a formal calibration program. The M&E DAS program also specifies calibration procedures for each analytical instrument. These programs verify that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment which measure a quantity, or whose performance is expected at a stated level, are subject to calibration. The detailed calibration protocols for the DAS analyses are presented in the DAS specifications found in Appendix A. The CLP calibration information is presented in the EPA RAS statements of work.

Before any instrument is used as a measurement device, the instrumental response to known reference materials must be determined. The manner in which various instruments are calibrated is dependent on the particular type of instrument and its intended use. All final sample measurements are made within the calibrated range of the instrument. Preparation of all reference materials used for calibration will be documented in a standards preparation notebook.

Instrument calibration typically consists of initial calibration and continuing calibration. Initial calibration establishes the calibration range of the instrument and determines instrument response over that range. Typically, three to five analyte concentrations are used to establish instrument response over a concentration range. The instrument response over the range is generally absorbance, peak height, etc., which can be expressed as a linear model with a correlation

coefficient (i.e. as a response factor), or as an amount versus response plot. Continuing calibration may be used within an analytical sequence to verify stable calibration throughout the sequence and/or to demonstrate that instrument response did not drift during a period of non-use of the instrument. If drift is detected, the corrective actions that are taken are defined in the appropriate EPA-CLP or DAS method being performed.

Calibration is further ensured by the periodic analysis of calibration verification standards (QC samples) during the course of analysis of field samples. The types of QC samples and the frequency at which they are analyzed to monitor an instrument response are specific to the EPA-CLP and DAS method protocols.

Method detection limits (MDLs) achievable by the EPA-CLP and M&E DAS laboratories are based on program requirements, sample matrix, and in-house instrument capabilities. For the CLP program, specific contract required detection limits (CRDLs) and contract required quantitation limits (CRQLs) are set by the EPA. In the M&E DAS program, the DAS quantitation limits (DAS QLs) are set on a project specific basis to ensure the project DQOs can be achieved. The CRDLs and CRQLs in the CLP program and DAS QLs may be higher than published MDLs, which are generally determined using clean matrices (e.g., deionized water) free of interferences and which are analyzed under optimal laboratory conditions. For actual sample analysis, the MDLs may not be routinely achievable and the CRDLs/CRQLs and DAS QLs are employed.

Individual sample detection limits may vary from the CRDLs/CRQLs or DAS QLs reported by the laboratory. These variances may be due to sample dilution requirements, variability in the sample weight or volume used relative to that specified in the analytical procedure, dry weight adjustments for solid samples, the presence of background contaminants, or other conditions related to the sample matrix or instrumental analysis.

7.2 FIELD TEST EQUIPMENT

The following calibration procedures are meant to summarize the detailed information found in specific instrument manuals. Field personnel should be familiar with the calibration procedures for each field instrument. Instrument manuals will accompany each field device and they should be consulted as necessary. All calibrations of field instruments will be recorded in either instrument logs or a field logbook.

7.2.1 Photoionization Detector (PID)

The photoionization detector (PID) will be used in the field to monitor volatile organic vapors, as required in the M&E Health and Safety Plan, and to screen for the magnitude of volatile organic contamination in soils. The PID will also be used to detect the vertical extent of contamination cuttings from soil borings and/or test pits. The PID expected to be used in this investigation is Photovac Microtip Analyzer, equipped with a 10.6-eV detector lamp. This choice provides adequate sensitivity for compounds of interest while maintaining field durability.

When calibrating the Microtip, the gas standards used for calibration should be at ambient temperature and pressure, and at the proper flow rate. Calibrations using toxic or hazardous gases must be done in a hood or an open area. The frequency of calibration is dictated by the frequency of use and the toxicity of the species measured. After the analyzer has been serviced or repaired, it should be calibrated to verify proper operation and performance. It is recommended that calibration be checked frequently at first (daily) and then regularly as confidence in the instrument increases.

An accurate and reliable method of calibration uses an analyzed gas cylinder which is discharged into a Tedlar gas bag. The Microtip sample intake is connected to the Tedlar bag during instrument calibration. Additional material on calibration is given in the instrument operations manual.

Calibration of the PID by the Analyzed Gas Cylinder Method. The analyzed gas cylinder is a compressed gas cylinder containing the species of interest at a known concentrations in an air matrix at or near the concentrations expected in the field. If the species of interest are unstable in air, another gaseous matrix will be used.

The calibration gas must be stable within the cylinder during the period of use. If calibration is required in the field, a small cylinder is recommended. The cylinder material will be compatible with the calibration gas. The cylinder will be equipped with a regulator which fits properly and is also compatible with the calibration gas. The operator will contact the supplier in the event of uncertainties. Extreme care will be taken in the handling of gas cylinders: the contents are under high pressure and, in some cases, the contents may be hazardous.

A gas calibration cylinder will not be used below a pressure of 200 to 300 psi, because the loss of pressure could cause the concentration and/or flow rate to vary. The cylinder will not be used past the recommended shelf-life of its contents.

The Microtip will be set-up properly prior to the overall instrument calibration. The instrument is set up by removing the instrument from the carrying case and turning it on. The LED display will show:

"Warming up, please wait"

When the instrument is finished warming up the display will read:

"Ready"

The concentration presently detected, "event #", time, and date will also be displayed. Before continuing the battery should be checked. Press BATT and the battery voltage will be displayed. Normal operating voltage is 9 to 14 volts. Press BATT a second time to return to ready mode. The instrument is now ready for calibration.

Once the instrument has warmed up, proceed with directions as follows to calibrate. The zero gas that is used is ambient outdoor air. If the background level is greater than zero ppm or if the air is of otherwise questionable quality then a supply of zero gas must be used.

1. Fill Tedlar Bag: A Tedlar bag will be used to supply the calibration gas to the instrument. If a supplied zero gas is used then separate bags should be used for the zero gas and the calibration gas. The following steps describe how to fill the bag.

   - Connect the regulator to the gas cylinder and hand tighten the fittings.
   - Open the valve on the gas bag by turning the valve stem fully counterclockwise.
   - Attach the gas bag adapter unit to the regulator and hand tighten the fittings.
   - Turn the regulator knob counterclockwise about half a turn to start the flow of gas.
   - Fill the gas bag about half full then close the regulator fully clockwise to turn off the flow of gas.
   - Disconnect the bag from the adapter and empty it. The bag should be flushed with the calibration gas at least one time. Fill the gas bag after flushing.
   - Close the bag by rotating the valve clockwise.

2. Select the Desired Calibration Memory: the Microtip can be calibrated with up to 5 different gases and/or bulb selections. Press SET UP and select the desired Calibration Memory with the arrow keys. Press ENTER when your selection is made. Press EXIT to return to the "ready" mode.

3. The actual calibration is performed as follows:

   - Press CAL and expose the Microtip to the zero gas. If a supplied gas is used then attach the gas bag containing the zero gas. (Make sure the bag stem valve is open by turning it fully counterclockwise).
   - Press ENTER and Microtip sets its zero point.

- Microtip will then ask for the span gas concentration. Enter the span gas concentration and press ENTER.
- Connect the gas bag to the inlet and press ENTER.
- When the Microtip returns to the "Ready" mode connect the sampling probe to the gas inlet. The instrument is now ready for use.

Recharging the Battery. On the bottom of this instrument is a button. To remove the battery pack, push in the button and slide the battery back away from the instrument (it may be helpful to use a pen to push in this button to avoid getting a finger caught as the battery pack is removed). To recharge the battery pack, plug the charger into the battery pack and then plug the charger into an AC outlet. Allow the battery to recharge for at least 8 hours to attain a full charge.

Calibration Check. A short-cut method may be used to quickly check the calibration of the PID in the field. Immediately after a calibration has been completed, a reading is taken using an isobutylene standard. The reading obtained is used as a benchmark throughout the day. Readings of the isobutylene standard may be taken and adjustments to the PID readings may be made as required. This is an indirect method of calibration, one that maintains the calibration on the isobutylene standard to indicate the fidelity of the calibration using the original gas mixture.

7.3 PREVENTIVE MAINTENANCE

Preventive maintenance of field equipment is required to ensure the collection of valid field measurements. All necessary maintenance procedures conducted by M&E are fully documented in the instrument logbooks and are further discussed in Section 12.0.

8.0 ANALYTICAL PROCEDURES

The analytical procedures to be used for samples collected during this field effort will consist of EPA-approved methods for RAS parameters and other EPA approved methodologies for DAS parameters. These procedures provide project-specific detection limits as well as QC requirements.

The PRPs will be utilizing EPA Method 8270 with an alumina column clean-up for semivolatile organic analyses and EPA Method SW846 for Priority Pollutant Metals plus Vanadium for their metals analyses. The methods used by M&E and the PRPs should yield comparable data as the PRP's methods are analogous to the CLP RAS methods to be used by M&E. Based on the information provided in the PRP's QAPP (Johnson Co., 1995), the detection limits specified for the semivolatile organic compounds listed in the QAPP are the same as the CLP RAS detection limits for those same compounds. A comparison of the metals detection limits was not possible as the PRP's QAPP did not provide a detection limits table for metals, however, the PRP's QAPP does state that all method detection limits used for this project "can be found in OLMO1.9 and ILMO3.0."

8.1 STANDARD ANALYTICAL METHODS

Samples collected during this field effort will be analyzed by RAS methods through the EPA-CLP system. DAS methods will be performed by M&E-procured laboratories, most of which have CLP contracts, and all of which have been prequalified by M&E. The analytical methods that will be performed specifically for this work are summarized in Table 8-1.

8.2 PROJECT-SPECIFIC DETECTION LIMITS

In order to attain the DQOs specified in section 4.0 of this SAP, project-specific detection limits for individual analytical methods are required. For RAS methods, CRDLs for inorganic analyses and CRQLs for organic analyses will be used. The project specific detection limits for

the RAS methods are presented in Table 8-2. Detection limits for DAS methods are presented in the DAS specifications provided in Appendix A. It should be noted that the detection limits specified by any method may not be attained for all samples because of such factors as matrix effects, dilutions, or sample weight variation/percent moisture adjustments (in the case of solids).

Project specific detection limits are not presented for the analytical level II field measurements because method detection limit studies are not routinely conducted for these parameters.

TABLE 8-1. ANALYTICAL METHODS FOR SEDIMENT SAMPLES

Parameter	Method	Analytical Level	Reference
Laboratory Analyses:			
TCL Semivolatile Organics	CLP-RAS	IV	Contract Laboratory Program, Statement of Work for Organics Analysis, (Multi-Media/Multi-Concentration). Document No. OLM01.0 including revisions OLM01.1 through OLM01.9. (USEPA, 1993b)
TAL Metals	CLP-RAS	IV	Contract Laboratory Program, Statement of Work for Inorganics Analysis, (Multi-Media/Multi-Concentration). Document No. ILM01.0 with revisions through ILM03.0. (US EPA, 1993c)
Grain size[1]	DAS	III	DAS Specification

NOTES:

1. All DAS Specifications are provided in Appendix A

TCL - Target Compound List
TAL - Target Analyte List

TABLE 8-2. PROJECT-SPECIFIC DETECTION LIMITS FOR RAS ANALYSES

Parameter/Analytes	Aqueous CRDLs and CRQLs	Soil/Sediment Detection Limit[1][2]
TAL Metals	**(µg/L)**	**(mg/kg)**
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5,000	1,000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6
Magnesium	5,000	1,000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5,000	1,000
Selenium	5	1
Silver	10	2
Sodium	5,000	1,000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
TCL Semivolatile Organics	**(µg/L)**	**(µg/kg)**
Phenol	10	330
Bis(2-chloroethyl) ether	10	330
2-Chlorophenol	10	300
Bis(2-chloroethyl) ether	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
2,2'-oxybis (1-Chloropropane)	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-dipropylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Bis(2-choloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330

TABLE 8-2 (Continued). PROJECT-SPECIFIC DETECTION LIMITS FOR RAS ANALYSES

Parameter/Analytes	Aqueous CRDLs and CRQLs	Soil/Sediment Detection Limit[1][2]
TCL Semivolatile Organics (Cont'd)	**(μg/L)**	**(μg/kg)**
1,2,4-Trichlorobenzene	10	330
Napthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methlyphenol	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	25	800
2-Chloronaphthalene	10	330
2-Nitroaniline	25	800
Dimethylphthalate	10	330
Acenaphthylene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	25	800
Acenaphthene	10	330
2,4-Dinitrophenol	25	800
4-Nitrophenol	25	800
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	25	800
4,6-Dinitro-2-methylphenol	25	800
N-nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	25	800
Phenanthrene	10	330
Carbazole	10	330
Anthracene	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	10	330
Benzo(a)anthracene	10	330
Chrysene	10	330
Bis(2-ethylhexyl)phthalate	10	330
Di-n-octylphthalate	10	330

TABLE 8-2 (Continued). PROJECT-SPECIFIC DETECTION LIMITS FOR RAS ANALYSES

Parameter/Analytes	Aqueous CRDLs and CRQLs	Soil/Sediment Detection Limit[1][2]
TCL Semivolatile Organics (Cont'd)	**(µg/L)**	**(µg/kg)**
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

(1) The CLP statement of work for inorganics analysis does not provide CRDLs for soils. The soil detection limits presented are the aqueous CRDLs adjusted to equivalent soil concentrations based on a 1.0 gram sample and a final digested volume of 200 ml.

(2) The quantitation limits for soil/sediments are highly matrix dependent. The quantitation limits presented are for guidance only. Sample specific quantitation limits for soil/sediment organic analytes will be higher than those presented due to the percent solids adjustment required for these matrices.

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9.0 DATA REDUCTION, VALIDATION, AND REPORTING

This section describes data reduction, validation, and reporting procedures which will be used by the EPA-CLP laboratories and the M&E DAS subcontract laboratories participating in this project and by M&E personnel as a final review. A flowchart outlining the steps which typically occur during the data review process is shown in Figure 9-1. Primary responsibility for implementation of these procedures within the laboratory resides with the laboratory manager. The laboratory manager conducts the final review and approves of all data reports before transferring the information to the point of contact (POC) officer within the EPA-CLP and M&E DAS laboratories. The POC officer, in turn, communicates with the SMO. Final responsibility for data validation resides with the M&E lead chemist.

9.1 LABORATORY DATA REDUCTION, VALIDATION, AND REPORTING PROCEDURES

The EPA-CLP and M&E DAS laboratories will adhere to the protocols described in the DAS specifications presented in Appendix A and the RAS protocols described in:

- *Contract Laboratory Program, Statement of Work for Organics Analysis (Multi-Media/Multi-Concentration). Document No. OLM01.0 including revisions OLM01.1 through OLM01.8.* (USEPA, 1993b)

- *Contract Laboratory Program, Statement of Work for Inorganics Analysis (Multi-Media/Multi-Concentration). Document No. ILM01.0 including revisions through ILM03.0.* (USEPA, 1993c)

The EPA-CLP laboratories analyzing the samples will be responsible for preparing the analytical data packages and providing the information concurrently to SMO and RSCC. A copy of the analytical data package will be obtained by M&E from the RSCC for data validation. For RAS analyses, the EPA-CLP laboratories must provide the analytical raw data and field QC sample summary forms as required by the SOWs for organic analysis (OLM01.0 with revisions through OLM01.9) and for inorganic analysis (ILM01.0 with revisions through ILM03.0). For DAS analyses, the M&E subcontracted laboratories must provide the analytical raw data and field and QC summary forms required by the DAS specifications. In general, the DAS specifications require that the laboratory submit the following deliverables:

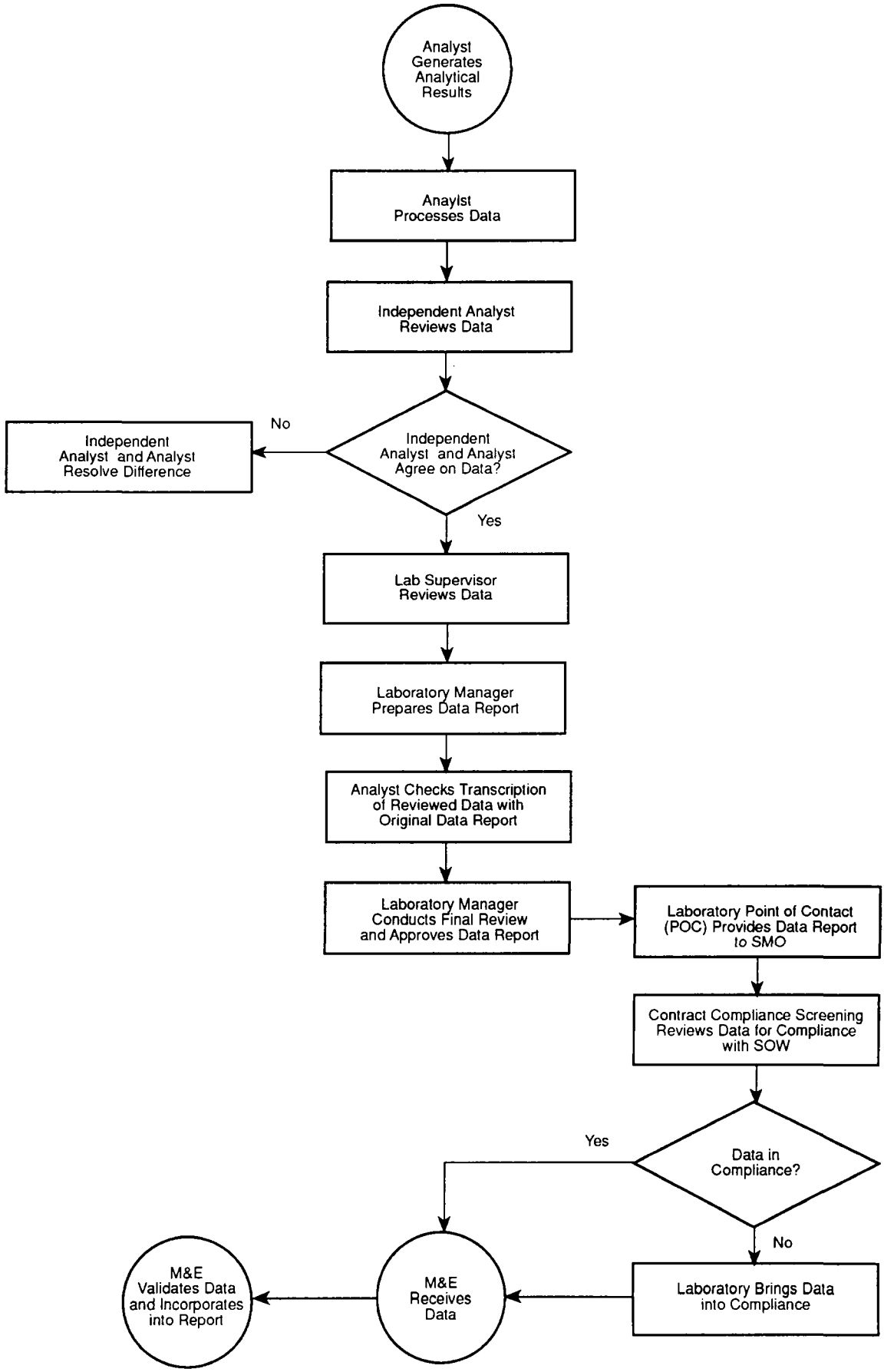


FIGURE 9-1. DATA REVIEW FLOWCHART

4535/jp

- Tabulated sample results; positive results and detection limits for nondetects. For soil samples, results reported on dry weight basis, and sample weight and percent moisture content must be reported.

- Laboratory analysis notebook pages or bench sheets; all raw sample data.

- Tabulated results of duplicate and matrix spike analyses; tabulations of blank results. Raw data for these analyses must also be provided.

- Standard curve raw data. Plotted standard curves. Linear regression equation.

- Examples of sample result calculations for each analysis. All equations, dilution factors, and information required to reproduced the laboratory results *must* be provided.

- Sample preparation logs.

- Copies of each DAS specification, chain-of-custody forms, telephone logs, sample tags, and shipping airbills.

- Narrative explaining all anomalies and corrective action(s) taken. Included in the narrative should be a tabulation of the DAS sample numbers with the corresponding laboratory numbers.

9.2 M&E INTERNAL DATA REDUCTION, VALIDATION AND REPORTING PROCEDURES

Described below are the types of procedures that are followed by M&E during the reduction, validation, and reporting of field and analytical data. Field and analytical data collected during this field investigation program will be used to evaluate the nature and extent of contamination and to conduct an engineering evaluation/cost analysis.

9.2.1 Data Reduction

Data reduction consists of compiling and summarizing data collected during field activities. Field and analytical data will typically be summarized in a tabular or other appropriate format. All information and data will be reported and verified for accuracy with the original sources of data. For analytical data, units designated by the analytical method will be reported.

Data produced for internal records and not reported as part of the analytical data include laboratory worksheets and notebooks, sample tracking system forms, instrument logs, standards

records, maintenance records, calibration records, and associated quality control. From non-laboratory sources these data typically include field logbooks, sample and QC sample tracking sheets, well development logs, instrumentation and calibration logs, and geologic logs. These data are generated during the field activities, and where relevant, are summarized for interpretation or use throughout the data evaluation process.

9.2.2 Data Validation

Quality control data provided by the laboratory will enable M&E to evaluate the validity of the analytical data in terms of accuracy, precision, and environmental significance. M&E will conduct data validation to meet Tier III criteria for the RAS and DAS data. The analytical level II and I field measurements will be reviewed but will not be formally validated.

All level IV data will be validated following the techniques specified in:

- *Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analysis*. February 1, 1988, Modified November 1, 1988. (USEPA, 1988)
- *Region I Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analysis*. June 13, 1988, Modified February 1989. (USEPA, 1989)

The functional guidelines will be modified to meet criteria in the current SOWs for RAS organics and inorganics analyses (USEPA, 1993b; 1993c).

All DAS data will be reviewed and validated to ensure that it meets the data requirements specified in the DAS specifications that are included in Appendix A. The following are typical actions implemented for analytical data produced with DAS specifications:

Holding Times: If the holding time is exceeded, all positive results will be flagged as estimated (J) and all non-detects will be flagged as estimated (UJ). If holding times are grossly exceeded, the data may be rejected (R).

Calibration: If the calibration criteria are exceeded, all positive results will be flagged as estimated (J) and all non-detects will be flagged as estimated (UJ). If the calibration criteria are grossly exceeded, all non-detects may be flagged as unusable or rejected (R).

Blanks: If blank contamination is present, an action level of 5 times the blank contaminant concentration will be set. If the sample analyte concentration is greater than the action level, the concentration will be reported unqualified. If the sample analyte concentration is less than the action level, the concentration will be reported and flagged to be the qualified detection limit (U).

EPA PE Sample: If the results of the EPA performance evaluation (PE) samples are outside the EPA acceptance windows, all positive results will be flagged as estimated (J). If the results of the EPA-PE sample are reported warning low, all non-detects will be flagged as estimated (UJ). If the results of the EPA-PE sample are reported action low, all positive results will be flagged as estimated (J) and all non-detects will be flagged as unusable or rejected (R).

Sample Duplicate: If laboratory or field duplicate analyses result in a RPD greater than 30% for aqueous samples or 50% for soil/sediments, all positive results will be flagged as estimated (J) and all non-detects will be reported unqualified. If one value is non-detected and the other is above the detection limit, all positive results will be flagged as estimated (J) and all non-detects will be flagged as estimated (UJ).

Matrix Spike: If the results of the matrix spike are greater than 25% above the true concentration, all positive results will be flagged as estimated (J) and all non-detects will be reported unqualified. If the results of the matrix spike are greater than 25% below the true concentration, all positive results will be flagged as estimated (J) and all non-detects will be flagged as estimated (UJ). If the results of the matrix spike are less than 10% of the true concentration, all positive results will be flagged as estimated (J) and all non-detects will be flagged as unusable or rejected (R).

9.3 DATA COMPARISON

M&E will compare the split-sample data with the PRP's sample data. The comparison will be used to assess the quality of the PRP's data. Comparability and confirmation between the data sets will enhance the confidence in the PRP's data. M&E will conduct a comparison of the validated split-sample data set with the corresponding PRP's data set. Individual data points will be compared using the guidelines provided in Section 9.3.1.

9.3.1 Comparison of Data

EPA's current field duplicate precision acceptance criteria are used as comparability criteria for this project. As stated in the functional guidelines used for data validation (EPA, 1988 and 1989) precision acceptance criteria is a maximum of 30%D for aqueous samples or 50%D for solid samples, with additional provisions for data near the reporting limit (RL). The RL is the

sample-specific detection limit (SSDL or sample quantitation limit [SQL]) reported by each laboratory for each individual sample. As described below, the comparison criteria are further expanded to include project-specific acceptance criteria. Figure 9-2 presents a flow diagram showing the comparison process that is used to evaluate split-sample data. The criteria discussed below are defined on Figure 9-2 by the number shown in parentheses for each criterion.

General Comparison Criterion

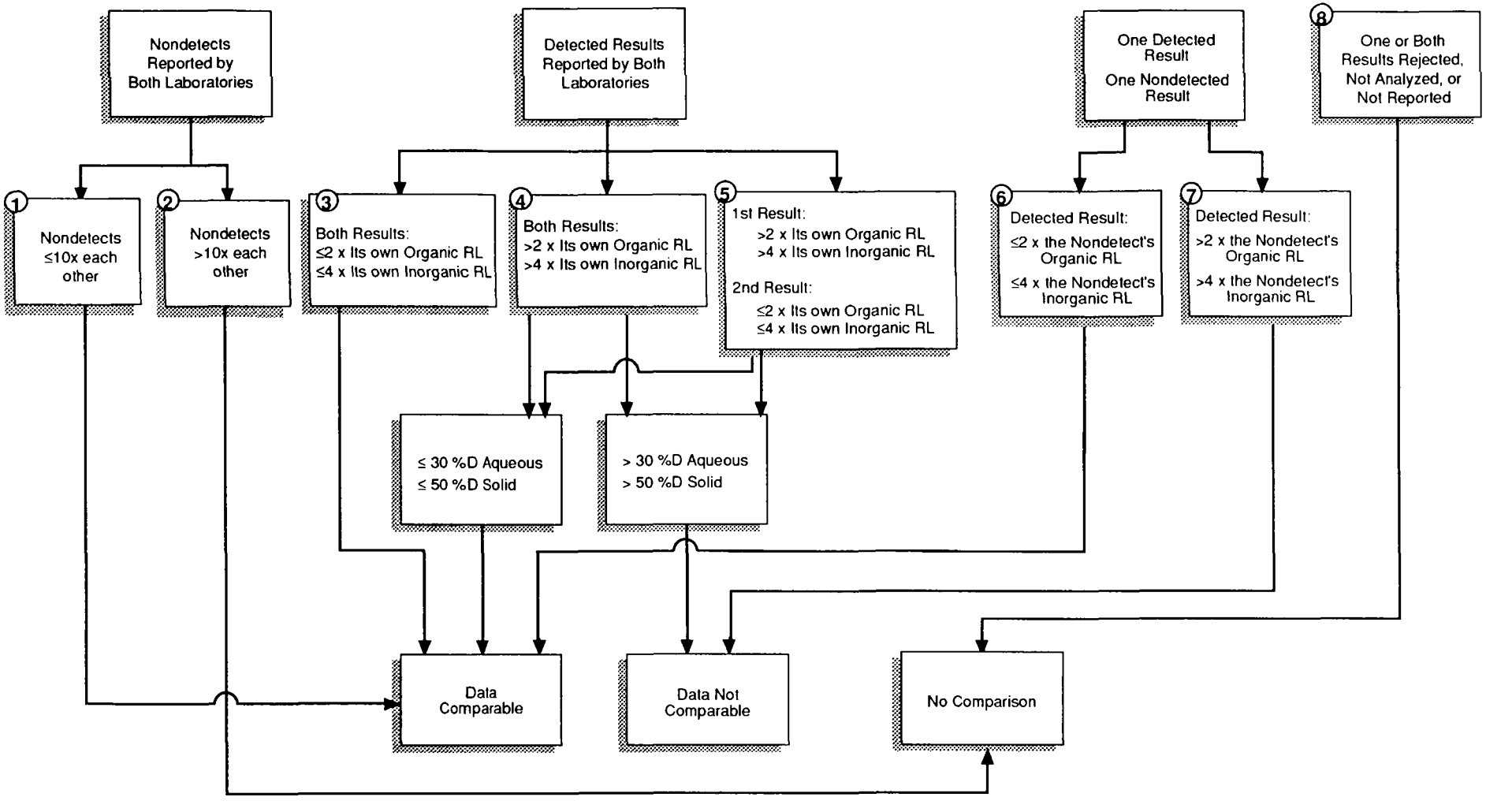
Data for each analyte are considered comparable if:

- Both results are reported nondetected by each laboratory and the RLs are less than or equal to 10 times (10x) each other (Box 1).
- Both split-sample results are less than or equal to 2 times (2x) their own RL for organic or 4 times (4x) their own RL for inorganic analytes (Box 3).
- Both detected split-sample results are greater than 2x their own RL for organic analytes, or greater than 4x their own RL for inorganic analytes, and the calculated %D is less than or equal to 30 for aqueous samples and 50 for solid samples (Box 4).
- One detected split-sample result is less than or equal to 2x its RL for organic analytes, or less than 4x its RL for inorganic analytes; and the other detected result is greater than 2x its RL for organic analytes, or greater than 4x its RL for inorganic analytes; and the calculated %D is less than or equal to 30 for aqueous samples and 50 for solid samples (Box 5).
- One split-sample result is nondetected and the detected split-sample result is less than or equal to 2x the nondetected results' RL for organic analytes, or 4x the nondetected results' RL for inorganic analytes (Box 6).

No Comparison Criterion

For each analyte, there is no basis for comparison if:

- If both split-sample results are nondetected and the difference between their RLs is greater than a factor of 10; this also requires discussion of data useability based on technical judgement (Box 2).
- One or both split-sample results are rejected, not analyzed, or not reported (Box 8).



NOTES: %D = Percent Difference
RL = Reporting Limit (SQLs or Sample Quantitation Limits were Used for M&E Data and for the PRP'S Data, if Available)

FIGURE 9-2. DATA COMPARISON FLOW DIAGRAM FOR INDIVIDUAL ANALYTES*

Field Duplicate Comparison Criterion

When M&E and the PRPs each have a field duplicate for the same split-sample pair, the percent relative standard deviation (%RSD) is calculated for each analyte. The calculation for %RSD is:

$$\%RSD = [[\{\Sigma(v\text{-}avg)^2\}/(n\text{-}1)]^{1/2}/avg] \times 100$$

where, avg = average of the detected values
n= number of detects
v = i^{th} detected value

The general and no comparison criteria stated above are then applied. The only exception being that the acceptance criteria for %RSD is a maximum of 20 for aqueous samples and 40 for solid samples.

When only one field duplicate pair from either M&E or the PRPs exists as part of the split-sample pair (i.e., one data set has a field duplicate pair and the other data set does not), the %D is first calculated between the field duplicate pair, and the field duplicate pair's comparability assessed according to the general and no comparison criteria described above. To assess comparability between the field duplicate pair and its associated split sample, the general and no comparison criteria are applied as described above, with the following exceptions:

- If the comparison criteria for the field duplicate pair is not met when both results are detected for an individual analyte, then there is no basis for comparison with the corresponding split sample.
- If the comparison criteria for the field duplicate pair is not met when one result is detected and the other result is nondetected for an individual analyte, then there is no basis for comparison with the corresponding split-sample data.
- If the comparison criteria for the field duplicate pair is met for an analyte, then:
  - If both field duplicate results are nondetected and the RLs are not the same, the lowest RL is compared with the associated split-sample result and the general and no comparison criteria are applied.

- If both results are detected, the %D is calculated using the field duplicate result closest to the associated split-sample result and the general and no comparison criteria are applied.

- If one field duplicate result is detected and the other is nondetected, the detected result is compared with the associated split-sample result and the general and no comparison criteria are applied.

Case-by-Case Comparison Criteria

For each analyte, comparability may be assessed using technical judgement on a case-by-case basis if:

- One split-sample result is nondetected because of validation qualification for laboratory or field blank contamination or potential laboratory contamination is noted for unvalidated data (e.g., B qualifier).

- One of the general comparison criteria described above are not met.

- Other situations exist that are not addressed in the general comparison criteria or site-specific events impact the comparison results.

NOTE: When technical judgement is used to consider data comparable, (e.g., other site-specific conditions or objectives, etc...) supporting information is presented in the text.

Overall Analytical Fraction Comparison Criteria

For each split-sample and field duplicate pair, the above criteria are applied on an analyte-by-analyte basis. To evaluate whether split-sample and field duplicate data compares for each analytical fraction analyzed (i.e., volatile organics, metals), the percentage of detected analytes that are comparable by fraction for each sample is assessed. If 75% of the detected data (where an analyte is detected in either one or both split/field duplicate samples) within an analytical fraction meet the comparison criteria, the fraction is comparable. If 75% of the detected data do not meet the comparison criteria, the nondetected results (where an analyte is not detected in both split or field duplicate samples) are then considered in the same manner. If 75% of all the data (both detected and nondetected) meet the comparison criteria, then the fraction is comparable. Non-comparable data (where one or both split-sample or field duplicate

results are rejected, not analyzed, or not reported) are not included in the assessment of the analytical fraction. The above criteria serves as guidelines for overall comparability of the fraction, however, in some cases technical judgement is used to supplement the guidelines.

Overall Split Sample Comparison Criteria

Each split-sample and field duplicate pair is considered comparable overall if comparability is achieved for 75% of the analytical fractions. This criteria serves as a guideline for overall comparability of the split-sample and field duplicate pairs, however, in some cases technical judgement is used to supplement the guideline.

A report will be prepared to discuss the comparability of the data sets, including discrepancies between the data sets, and an overall evaluation of the data comparison.

10.0 INTERNAL QUALITY CONTROL CHECKS

The QC program developed by M&E will use both known and unknown (or "blind") QC samples, and will follow all EPA-CLP QC requirements. The various types of field and laboratory QC samples are described below. The type and frequency of analyses for each QC sample is shown in Table 10-1. The QC requirements for DAS analyses are given in the DAS Specifications (Appendix A).

10.1 FIELD GENERATED QC SAMPLES

Quality control samples that will be collected in the field and submitted to the laboratories along with the environmental samples are discussed in this section. Locations where QC samples will be collected will be determined in the field based on technical judgement and sample volume. The types of QC samples that will be collected during the field investigation program include: equipment blanks, and field duplicates.

10.1.1 Equipment Blank

One equipment blank will be collected per sampling episode per sampling medium or at a frequency equal to approximately 10% of the samples collected for each media, whichever is more frequent. The analysis of these blanks serves to verify the cleanliness of the sampling equipment. An equipment blank is collected by rinsing decontaminated field equipment with water, transferring the water to a sample bottle, and sending the sample for analysis. HPLC-grade water should be used for QC samples submitted for organic analyses. For other QC samples, DIUF water should be used. The equipment blank is analyzed for the same parameters as the samples associated with that equipment.

10.1.2 Field Duplicates

Approximately 10% of all the samples will be collected in duplicate and submitted for laboratory analysis. Duplicates are two samples collected independently from one sampling location during a single episode of sampling. Duplicates provide information about sample variability.

TABLE 10-1. QC SAMPLE TYPES, CRITERIA, AND CORRECTIVE ACTION

Type	Purpose	Frequency	Criteria	Corrective Action
Field Generated QC Samples:				
Equipment Blank	Verifies effective decontamination procedures used in field	10% of samples collected per media	No compound of interest >5 times CRQL/CRDL	Qualify data or resample
Field Duplicate	Measure samples variability (submit "blind" to lab)	10% of samples	±30% RPD[2] (aqueous) ±50% RPD[2] (soil)	Compare to lab replicates; check systems for possible matrix interferences or improper sample collection procedure
Matrix Spikes and Duplicates (MS/MSD)	Checks recovery from real matrix	1 per 20 samples or 1 per batch as supplied from the field	Recoveries as specified in Tables 4-1 and 4-2	Qualify data or recalibrate, reanalyze, and document corrective action
Laboratory Generated QC Samples:				
Laboratory Control Samples (for AA,ICP)	Verifies analyst proficiency with method and instrumentation	1 per 20 samples or 1 per batch as supplied from the field	<20% RSD[1] ±15% recovery	Check system; require additional operator training
Calibration Check Sample	Verifies calibration curve	≥ 1 per day of analysis	±20% initial calibration	Recalibrate; check system
Method Blank	Verifies clean reagents, instrument systems, and lab environment	1 per 12-hour analysis day per 20 samples or 1 per batch[3] as supplied from the field	No compound of interest >5 times CRQL/CDRL	Reanalyze; if second blank exceeds criteria, clean and recalibrate system; document corrective action

TABLE 10-1 (Continued). QC SAMPLE TYPES, CRITERIA, AND CORRECTIVE ACTION

Type	Purpose	Frequency	Criteria	Corrective Action
Laboratory Replicates (including matrix spike duplicates)	Checks precision of analytical method	1 per 20 samples or 1 per batch as supplied from the field	RPD[2] as specified in Tables 4-1 and 4-2	Compare with field duplicates; check matrix interferences
Surrogate Standards	Measures recoveries in actual sample matrices	All GC/MS and all GC samples	Recoveries as specified in Tables 4-1 and 4-2	Reanalyze samples; qualify or reject data

NOTES:
1. At least 5 replicates are needed.
2. Between duplicate measurements.
3. The term "batch" refers to samples analyzed together in a specified group using the same methods, the same types and lots of reagents, and the same timeframe for analysis.

RPD - Relative Percent Difference
RSD - Relative Standard Deviation
VOC - Volatile Organic Compounds
CRQL - Contract Required Quantitation Limit
CRDL - Contract Required Detection Limit

Duplicate samples will be collected based on technical judgement and adequate volume being present at a sampling location.

10.1.3 Matrix Spike/Matrix Spike Duplicates

Matrix spike and matrix spike duplicates (MS/MSDs) are a quality control requirement performed by the laboratory as discussed in 10.2.5. It is necessary to collect three times the usual required volume for aqueous samples to be analyzed as MS/MSDs. No additional sample volume is necessary for solid samples. At a minimum, additional volume for one sample will be provided to the laboratory for every group of 20 samples collected per media and matrix. Matrix Spike/Matrix Spike Duplicate samples will be collected based on technical judgement and adequate volume being present at a sampling location.

10.1.4 Documentation and Review of Quality Control Activities

Custody of field quality control samples will be documented from the time of QC sample collection throughout transfer of the sample to the laboratory. Documentation of sample collection, shipment, laboratory receipt, and laboratory custody must be maintained in order to accomplish this. Field quality control samples will be packed and delivered along with their corresponding environmental samples. A detailed description of sample custody and field documentation can be found in section 6.0.

10.2 LABORATORY GENERATED QC SAMPLES

The EPA-CLP laboratories will comply with the QC sample requirements for the RAS methods used during this field investigation program. The QC sample types generally required by the analytical methods are described below and are referenced in the SOWs for organic analysis (OLM01.0 with revisions through OLM01.9) and for inorganic analysis (ILM01.0 with revisions through ILM03.0). The type and frequency of laboratory QC samples is shown in Table 10-1.

The QC requirements of each DAS method are specified in the individual DAS specifications included in the Appendix A.

10.2.1 Laboratory Control Standard

One laboratory control standard (LCS) will be analyzed for every 20 samples or every batch of samples supplied from the field. A LCS is analyzed for each batch of samples tested for metals and inorganics. The LCS is routinely used to establish the precision and accuracy of an instrument or procedure. The analytical results of the LCS are recorded in the instrument logbook and on the control chart; results must be within the acceptable control limits. A LCS solution is prepared by adding known quantities of an EMSL-Cincinnati Standard, a NIST Standard Reference Material, or a reference-traceable stock material to deionized water or the solvent of interest. A LCS solution is typically carried through the entire sample preparation and analysis procedure.

10.2.2 Calibration Check Sample

One calibration check sample (CCS) will be analyzed for all project-specific parameters per day of analysis. A CCS is chosen as one of the mid-range working calibration standards that is reanalyzed periodically throughout the sample analysis to verify that the original calibration is still valid.

10.2.3 Method Blank

One method blank will be analyzed with every 20 samples or every batch of samples supplied from the field. A method blank is comprised of laboratory-pure, analyte-free water carried through the entire sample preparation and analysis procedure. Analysis of the method blank provides a check of the background contamination due to sample preparation procedures.

10.2.4 Laboratory Replicates

One sample for every 20 samples or every batch of samples supplied from the field will be analyzed in replicate for project-specific parameters. A replicate sample is produced by dividing a single collected sample into two equal parts for the purpose of determining analytical precision.

10.2.5 Matrix Spike/Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates will be performed for every 20 samples or every batch of samples supplied from the field. The analyte spike will be added prior to digestion/distillation of the sample. If the spike recovery is not within the acceptable criteria limits specific to this project, the data of those samples associated with that spiked sample must be handled appropriately.

10.2.6 Surrogate Spikes

All collected samples requiring organic analysis by GC/MS or GC will be spiked with an appropriate set of surrogate standards prior to sample preparation. The surrogate standards will encompass the full range of types of organics to be analyzed in the sample and will also serve as checks on any matrix interference exhibited by the samples. If the percent recoveries of the surrogates are outside the acceptable project-specific criteria limits, the associated samples may be reanalyzed if the problem appears to be due to lab error. An independent analyst or laboratory unit leader will determine whether to reanalyze the sample or qualify the data.

10.2.7 Documentation and Review of Quality Control Activities

Laboratory quality control samples will be documented as specified by RAS methods. A list of the required deliverables for DAS analyses are described in the individual DAS Specifications included in Appendix A. The QC activities pertinent to the analysis of each shipment of samples from the field will be documented in discrete sections of the RAS and DAS data report and include:

- A case narrative describing any problems encountered with method blanks, matrix spike, and matrix spike duplicates, surrogate recoveries, initial calibration, and continuing calibration
- Compilation of method blanks data
- Compilation of matrix spike and matrix spike duplicates
- Surrogate recoveries data
- Initial calibration and continuing calibration information

All QA activities are documented in the laboratory, but are only delivered upon request of the contracting agency. Delivery of the entire QA/QC package must be contracted prior to sample analysis.

11.0 PERFORMANCE AND SYSTEM AUDITS

Quality assurance audits play an important role in the EPA ARCS QA/QC program. An audit is a systematic check to determine the quality of operation of some function or activity. This section describes the role of the QA auditor and the nature of QA audits.

11.1 AUDITOR RESPONSIBILITIES

The QA auditor is a non-project related staff member selected by the ARCS QA Manager (QAM), who performs the QA performance and systems audits. Since QA audits represent, by definition, independent assessments of a measurement system and associated data quality, the auditor must be functionally independent of the measurement effort to ensure objectivity. However, the auditor must be sufficiently familiar with the objectives, principles, and procedures of the measurement efforts to be able to perform a thorough and effective evaluation of the measurement system. Especially important is the ability of the auditor to identify components of the system that are critical to overall data quality. For this reason, the QA audit focuses heavily upon those elements. The auditor's technical background and experience should also provide a basis for appropriate audit standard selection, audit design, and data interpretation.

The M&E QA organizational structure (see Section 3.0) has been set up to ensure the independence of the QA function. The Corporate QA Officer sets the corporate QA policies and oversees implementation of these policies. The ARCS QAM reports to the Corporate QA Officer and is given the responsibility for management, scheduling, and overseeing audit activities. The ARCS QAM is also responsible for selecting personnel to perform and design both performance and system audits. The QA Officer, who reports directly to the ARCS QAM oversees the QA process for individual projects and notifies the ARCS Project Manager of discrepancies and potential problems discovered during audits. The Project Manager initiates corrective action, documents action taken, and reports accomplishment of corrective remedies to the ARCS QAM.

11.2 PERFORMANCE AUDITS

The performance audit is a small-scope audit on specific Environmental Data Collection Activities (EDCAs) performed by the QA auditor at the direction of the QAM. Performance audits may be performed on an ongoing basis during the oversight project as field data are generated, reduced, and analyzed. All numerical manipulations, including manual calculations, will be documented. All records of numerical analyses must be legible, of reproduction quality, and sufficiently complete to permit logical reconstruction by a qualified individual other than the originator.

Other indicators of the level of field performance are the analytical results of the field QC samples (i.e., equipment blanks, field duplicates, and PE samples). Each blank analysis is an indirect audit of the effectiveness of measures taken in the field to ensure sample integrity (i.e., field decontamination procedures). The results of the field duplicate analysis are an indirect audit of the ability of the field personnel to collect representative sample portions of each matrix type. A PE sample tests the laboratories ability to correctly identify and quantitate analytes in the sample.

M&E has chosen the following EDCAs for performance auditing:

- Document control activities
- Planning activities (design and design oversight)
- Laboratory activities (including QA/QC procedures)

The types and frequencies of the audits pertinent to sampling activities at a given site are described in the following sections.

11.2.1 Laboratory Activities

All analyses on M&E split samples will be performed by EPA-CLP laboratories previously approved by the EPA to perform such analyses for ARCS projects. The EPA-CLP laboratories are periodically audited by the EPA Superfund branch and are only awarded samples when the audit is approved. Similarly, DAS laboratories have been audited by M&E and are only awarded a contract upon the successful conclusion of an audit.

11.3 SYSTEM AUDITS

The system audit is a review of the entire project or program using performance audits as key information.

11.4 POST-AUDIT COMMUNICATIONS

Following each audit, the auditor is responsible for drafting an audit report to be sent to the Project Manager with copies to the ARCS QAM and audit participants. The report will include findings and recommended actions as appropriate. A post-audit communications session is then scheduled and conducted to discuss the preliminary audit results. If the audit reveals recommended action(s) the project manager will issue a Corrective Action Report (Figure 11-1) to the auditor to document that the prescribed corrective action has been implemented.

11.4.1 Corrective Action Responsibility

The ARCS QAM will review the audit and corrective action reports to assure effective resolution is implemented.

CORRECTIVE ACTION REPORT

Audit No.:

Program:

Project:

Audit Date:

Report Date:

Prepared by:_____ Date:_____

Distribution:

Program Manager:_____

Quality Assurance Manager:_____

FIGURE 11-1. CORRECTIVE ACTION REPORT

12.0 PREVENTIVE MAINTENANCE

This section briefly describes the general preventative maintenance requirements that are followed for laboratory and field equipment.

12.1 LABORATORY EQUIPMENT

The ability to generate valid analytical data requires that all analytical instrumentation be properly calibrated and regularly maintained. The EPA-CLP laboratories maintain full service contracts on all major instruments. These service contracts not only provide routine preventive maintenance but also emergency repair service. The EPA-CLP system requires the EPA-CLP laboratories to document instrument maintenance as part of the laboratory contract. Similarly, DAS laboratories are required to maintain laboratory equipment and provide contingencies for equipment failures.

12.2 FIELD EQUIPMENT

Preventive maintenance of field equipment is provided on a routine basis and is required in order to ensure the collection of valid field measurements. The type and frequency of such preventive maintenance (Table 12-1) varies according to model and type of field equipment.

12.2.1 Instrument Calibration and Maintenance

The routine calibration procedures, maintenance procedures, and frequencies used for field instrumentation are summarized in Table 12-1. Preventative maintenance and calibration by manufacturer service representatives are provided on a routine basis.

TABLE 12-1. PREVENTIVE MAINTENANCE REQUIREMENTS FOR FIELD EQUIPMENT *(1)*

Instrument	Services	Frequency *(2)*	Reference
Photoionization Detector (PID)	Check calibration daily with each use	As required	Manufacturer's Manual
	Check response Check electrolyte	As required	
	Clean UV lamp and ion chamber Replace lamp	Prior to field work and upon return	

Notes:

1. The battery should be checked for each instrument prior to use; the instrument should be recharged according to Section 7.2. This Table includes only those instruments that will be operated by M&E.
2. Services will also be used if instrument malfunction is suspected.

12.2.2 Instrument Maintenance Logbooks

Each field instrument is assigned an instrument logbook. All maintenance activities are recorded in the instrument log. The information entered in the instrument log includes:

- Date of service,
- Person performing service,
- Type of service performed and reason for service,
- Replacement parts installed (if appropriate), and
- Miscellaneous information.

If service is performed by the manufacturer, a copy of the service record is taped into the page facing the notebook page where the above information is entered. This information is kept by the M&E ARCS equipment manager in the Wakefield office.

13.0 PROCEDURES FOR ASSESSING PRECISION, ACCURACY, AND COMPLETENESS

The following are procedures for evaluating the precision, accuracy, and completeness of analytical data generated as part of the split-sampling program. Data quality and QA objectives were further discussed in Section 4.0 and data validation requirements are described in greater detail in Section 9.0.

13.1 EVALUATION OF ANALYTICAL PRECISION AND ACCURACY

Precision and accuracy for the RAS analyses will be ensured by adherence to protocols specified in the current statements of work (SOWs):

- *Contract Laboratory Program, Statement of Work for Organics Analysis (Multi-Media/Multi-Concentration). Document No. OLM01.9 including all applicable revisions.* (USEPA, 1993b)
- *Contract Laboratory Program, Statement of Work for Inorganics Analysis (Multi-Media/Multi-Concentration). Document No. ILM03.0 including all applicable revisions.* (USEPA, 1993c)

Precision and accuracy for grain size will be ensured by adherence to the DAS method presented in Appendix A. This DAS method has been approved by the EPA ARCS Contracting Officer.

13.2 EVALUATION OF COMPLETENESS

Completeness is measured as the percentage of valid data points obtained compared to the amount expected to be collected. Data may not be considered valid if sample analyses exceeded the holding time criteria, if the QC sample criteria were not met and sample re-analysis was not performed, or if sample containers were broken or otherwise destroyed prior to sample analysis.

14.0 CORRECTIVE ACTION PROCEDURE

Corrective action procedures will be initiated when failure to properly follow M&E's project plans or SOPs in the field is recognized. Errors in following sampling protocols or improperly or inadequately decontaminating sampling equipment may ultimately make it impossible to meet the DQOs. Therefore, the deficiencies noted in the following standard protocol will be addressed immediately upon recognition.

Corrective action procedures for this project may be the result of a field oversight surveillance activity, a direct result of performance and/or system audits as described in Section 11.0, or an observation made by field personnel or other trained personnel. The person recognizing the failure is responsible for bringing the error to the attention of the responsible party, making note of the problem in the field notebook, and, if appropriate, orally notifying the M&E Project Manager and the EPA RPM of the error.

In the case of problems that are the result of M&E employees not adhering to M&E plans and SOPs, if the problem recurs, the person recognizing the deficiency will address the error through submittal of a Recommendation for Corrective Action (RCA) form (Figure 14-1) to the ARCS QAM and the M&E Project Manager, and the M&E Program Manager. The QAM, in turn, will file the original RCA, send a memo along with a copy of the RCA to the person in a position to effect the corrective action, and request a written response to the memo within a specified period of time. The issue addressed in the RCA is subject to follow-up action by the ARCS QAM, the M&E Project Manager, and the M&E Program Manager. The M&E Project Manager and the M&E Program Manager will assess the need to inform the RPM. Any deficiency that results in a reduced level of data quality or impacts the project budget or schedule will be brought to the immediate attention of the EPA RPM.

Corrective action procedures to be implemented within the EPA-CLP laboratories will be addressed from within the EPA-CLP system. Quality control records addressing daily instrumental calibration, instrumental control limits, method detection limits, and analyses of

RECOMMENDATION FOR CORRECTIVE ACTION

A. Initial Information

Job #:	Date:	URGENCY LEVEL ☐
Originator:		1. Potential safety or property hazard 2. Potential for failure to achieve quality objectives 3. Suggested improvement
Organization or Individual Responsible for Action:		

B. Problem Identification

Site or Lab:	Date Identified:
System:	Issue Raised First Through Normal Channels: ☐
Description of Problem:	

C. Recommended Corrective Action

Description:	Implement by:

D. Distribution

Date to QA Manager:	Date to Responsible Management:

E. Problem Resolution

Proposed by:	Date Proposed:	Scheduled Implementation:
Planned Corrective Action:		
Implemented Corrective Action:	Date Implemented:	
	Manager Signature:	
	Date Filed by QA Manager:	

FIGURE 14-1. RECOMMENDATION FOR CORRECTIVE ACTION (RCA) FORM

quality control samples will be maintained as required by the EPA-CLP protocol. Any problems encountered and their corresponding corrective action will be documented by the laboratory and are made available to project personnel, through the EPA SMO or the EPA TPO for Region I, in the form of case narratives and contract compliance screening reports.

15.0 QUALITY ASSURANCE REPORTS

When the PRP's data for split-sampling activities are available, the M&E Project Engineer will prepare a summary report on the performance of the measurement systems and the corresponding data quality and the Project Chemist will prepare a Field Oversight Report as described in Section 5.1.1 and a Split-Sampling Report as described in Section 9.3. These QA reports will address, at a minimum, the following:

- Results of performance audits of all oversight field sampling and laboratory analysis activities performed during the subject reporting period
- Results of system audits
- Assessment of measurement data accuracy, precision, completeness, and comparability including review of all EPA-CLP laboratory measurement data
- Any situations found during the M&E's field and sampling activities which require corrective actions as described in Section 14.0

The issues of accuracy, precision, completeness, representativeness, and comparability will be addressed according to the guidelines and objectives outlined in Sections 4.0 and 13.0 to ensure proper data reduction, validation, and reporting.

16.0 REFERENCES

Johnson Company (JCO). 1995. *Work Plan for ARI Phase IIB.* September 8, 1995.

Metcalf & Eddy (M&E). 1992a. *Supplemental Remedial Investigation Final Report, Pine Street Canal Superfund Site, Burlington, Vermont.* March 1992.

Metcalf & Eddy (M&E). 1992b. *Treatability Study Final Report, Pine Street Canal Superfund Site, Burlington, Vermont.* April 1992.

Metcalf & Eddy (M&E). 1992c. *Baseline Risk Assessment Final Report, Pine Street Canal Superfund Site, Burlington, Vermont.* May 1992.

Metcalf & Eddy (M&E). 1992d. *Feasibility Study, Pine Street Canal Superfund Site, Burlington, Vermont.* November 1992.

Metcalf & Eddy (M&E). 1995a. *Memo from Metcalf & Eddy to H. Horahan.* March 24, 1995.

Metcalf & Eddy (M&E). 1995b. *Memo from Metcalf & Eddy to H. Horahan.* March 31, 1995.

PEER Consultants, (PEER) 1990. *Draft Remedial Investigation Report for Pine Street Canal Site, Burlington, Vermont.* May 1990.

U.S. Environmental Protection Agency (U.S. EPA). 1983. *Methods for Chemical Analysis of Water and Wastes.* EPA-600/4-79-020. March 1983.

U.S. Environmental Protection Agency (U.S. EPA). 1988. *Region I Laboratory Data Validation Functional Guidelines for Evaluating Organics Analysis.* February 1, 1988, Modified November 1, 1988.

U.S. Environmental Protection Agency (U.S. EPA). 1989. *Region I Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analysis.* June 13, 1988, Modified February 1989.

U.S. Environmental Protection Agency (U.S. EPA). 1990. *Region I Volatile Organic Field Screening Method for Aqueous Samples.* September 1990.

U.S. Environmental Protection Agency (U.S. EPA). 1992. *Guide to Management of Investigation - Derived Wastes.* EPA Office of Solid Waste and Emergency Response. Publication 9345.3-03FS. January 1992.

U.S. Environmental Protection Agency (U.S. EPA). 1993a. *Region I Tiered Organic and Inorganic Data Validation Guidelines.* July 1993.

U.S. Environmental Protection Agency (U.S. EPA). 1993b. *Contract Laboratory Program, Statement of Work for Organics Analysis, (Multi-Media/Multi-Concentration). Document No. OLM01.0 including revision OLM01.1 through OML01.9.* July 1993.

U.S. Environmental Protection Agency (U.S. EPA). 1993c. *Contract Laboratory Program, Statement of Work for Inorganics Analysis, (Multi-Media/Multi-Concentration). Document No. ILM01.0 with revisions through ILM03.0.* July 1993.

APPENDIX A

DAS SPECIFICATIONS FOR PINE STREET CANAL

ANALYTICAL SPECIFICATION

FOR

TOTAL ORGANIC CARBON,

TOTAL COMBUSTIBLE ORGANICS,

GRAIN SIZE, MOISTURE CONTENT,

AND pH IN SOIL AND SEDIMENT

Prepared by:

METCALF & EDDY, INC.
WAKEFIELD, MASSACHUSETTS
MARCH 1995
DR. BRIAN TUCKER

1. SCOPE

Metcalf & Eddy Inc. (M&E), under Contract No. 68-W9-0036 to the U.S. Environmental Protection Agency, Region I, is conducting an investigation at a Superfund site that requires, in part, analysis of Total Organic Carbon, Total Combustible Organics, Grain Size Distribution, Moisture Content, and pH in soil, sediment, or other solid samples. The number of samples and the matrix will be identified in each work order. In the event that historical results are available, the range of past concentrations reported will be provided to the laboratory. Performance Evaluation (PE) samples may be submitted for analysis. If PE samples are submitted, instructions for preparation and analysis of the PE samples will be provided in the work order.

2. PURPOSE

Data generated from this specification will be compared to prior data generated by a Potentially Responsible Party hired consulting firm at this site. This will provide a means for determining the quality of the consulting firm's data. This specification may also be used at other Superfund sites to characterize the extent of the contamination, define excavation limits, confirm onsite laboratory or field test kit screening results, and/or determine the efficacy of remedial activities.

3. DEFINITION OF WORK

Sediment samples are to be analyzed for Total Organic Carbon (TOC) via the Lloyd Kahn method, Total Combustible Organics (TCO) via ASTM D 2974-87, Grain Size Distribution via ASTM D 422-63, Moisture Content via ASTM D 2974-87, and pH by OLM01.9, Section II, Part 1.7.1.1, page D16/SV. The chain of custody will indicate which samples are scheduled for the individual analyses.

4. SCHEDULE

Samples will be shipped at most one day after collection by an overnight delivery service. Saturday delivery may be required. Contacts for shipping and the anticipated sample collection dates and number of samples will be provided in the work order. Data delivery inquiries may be made to Dr. Brian Tucker, M&E, at (617) 224-6433 or the project chemist specified in the work order.

Holding Times:

The samples must be analyzed for all parameters except pH within fourteen (14) days of collection. pH analyses must take place within seven (7) days of sample collection.

Delivery of Data:

Sample data must be delivered to M&E within thirty-five (35) days of laboratory receipt of the last sample per sample delivery group (SDG). An SDG is defined as twenty or fewer samples submitted over a period of fourteen (14) calendar days or fewer. Results must be delivered under chain of custody to:

Dr. Brian Tucker
Metcalf & Eddy Inc.
30 Harvard Mill Square
P.O. Box 4071
Wakefield, MA 01880-5371
Phone (617) 224-6433
Fax (617) 245-6293

5. ANALYTICAL REFERENCES

Total Organic Carbon: Lloyd Kahn Method for Determination of Total Organic Carbon in Sediment, U.S. EPA, Region II, July 1986

ASTM D2974-87 July 1987
Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils

ASTM D 422-63 Nov. 1963 (Reapproved 1990)
Standard Test Method for Particle Size Analysis of Soils

EPA SOW OLM01.9 July 1993
Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration

6. SAMPLE PRESERVATION

Preservation will be performed by cooling and maintaining the samples at 4°C.

7. ANALYTICAL PROCEDURES

TOC The Lloyd Kahn Method must be followed except as indicated below:

- If the sediment is less than 30% solids, a larger sample must be analyzed in order to meet the reporting limit requirements. Determine percent solids as per Method B ASTM D2974-87.

- Each sample must be analyzed in duplicate. The limits for the Percent Difference (%D) are less than or equal to 20.%. If the criterion is not met, reanalyze both the sample and the duplicate. If the %D criterion is still not met, flag the sample and duplicate results with a "*". Report both values and the average value in mg/kg on a dry weight basis.

- The date of preparation of each solution used in the analysis must be clearly documented in the analyst's logbook and provided in the final data package.

- A five point standard calibration curve must contain a laboratory blank and span the expected sample concentration range. The lowest standard must be equal to the reporting limit. The correlation coefficient must be greater than or equal to 0.995 before the samples can be analyzed. If the criterion is not met, generate a new standard curve that meets the criterion and reanalyze the samples. If a sample falls outside of the calibration range, either use less (no less than 50.% of what the Method specifies) sample or recalibrate using standards that span the desired range. Report the source of the calibration standards.

- A Continuing Calibration Check Standard must be analyzed after every 10 samples, including QC samples, and at the end of the analytical run. The Continuing Check Standard must agree within $\pm$10.% of the initial curve. If the standard is outside of these limits, recalibrate the instrument and reanalyze all samples and blanks run since the last compliant Continuing Calibration Check Standard.

- A Calibration Check Standard (from a separate source than the calibration standards), at a concentration of 200. mg/kg, must be analyzed after the standards and prior to blank and sample analysis, and after the last sample in the analytical sequence. The recovery results must agree within $\pm$15.% of the true value. If the check standard analyzed prior to sample analysis does not meet the criterion, a new standard curve must be generated. If the check standard analyzed at the end of the analytical sequence does not meet the criterion, reanalyze all samples, blanks, and

check standards. The Calibration Check Standard must meet the criterion prior to sample analysis. Report the source of the check standard.

- A Method Blank must be analyzed prior to sample analysis and after the analysis of 20 samples (including QC samples) or at the end of the analytical sequence if there are less than 20 samples in the sequence. The blank is to be brought through the entire preparation and analytical procedure. If contamination is present at 50. mg/kg or greater, determine and document the source of contamination, and prepare and analyze a new blank until the contamination is less than 50. mg/kg. This criterion must be met prior to sample analysis.

- A Matrix Spike (MS) must be performed at a frequency of one (1) per SDG. The sample to be used as the MS will be noted on the traffic report. The concentration of the MS, at the time of analysis, is at the mid-range standard concentration or at twice the sample concentration, whichever is higher. Report the results for the spiked and unspiked sample and the Percent Recovery (%R). The %R must be between 80.% and 120.%. If this criterion is not met, prepare and analyze a new spiked sample, using the same field sample. If the %R is still outside the limits, flag the matrix spike and unspiked sample results with a "#".

- If a scrubber is incorporated in the instrumentation, the efficiency of that scrubber must be measured daily before running samples. The efficiency will be measured as per manufacturer's instructions and the result reported along with each day's run or with each sample delivery group of 20 samples or less, whichever is more frequent.

TCO Method ASTM D2974-87 must be followed except as indicated below:

- ASTM Method D2974-87 lists several methods. Method D (Section 10) will be used to measure the ash content by combustion at 750°C.

- The sample size will be no less than 20 grams of dry ground and homogenized soil.

- The Method D2974-87 does not stipulate an exact time for ashing. Air drying of the sample prior to ashing is not necessary provided an aliquot equivalent to 20 grams of dry soil is used. The minimum ashing time is 2 hours at 750°C in the muffle furnace. This 2 hours does not include the time to bring the sample to temperature. The temperature must be checked and adjusted if the temperature is $< 740°C$ or $> 760°C$ prior to sample analysis.

- The desiccation cooling time must be constant for all samples and blanks and must be recorded in the laboratory notebook. Thirty (30) minutes to a maximum of sixty (60) minutes is adequate for uniform cooling.

- The analytical balance used to determine the mass of each fraction must comply with the requirements described in Section 3.1 of the Method. The balance must be checked daily prior to weight determination with Class "S" weights and the calibration must be within the manufacturer's tolerance limits, or the balance must be recalibrated. The calibration must be recorded and reported.

- A method blank must be run on an empty drying dish. The blank results for weight loss must be accounted for in the evaluation of sample results.

- Run each sample in duplicate. The %D results must be $\pm$ 15.%. Report the mean if the criterion is met. If not, reanalyze the sample once. If the $\pm$ 15.% criterion is not met on reanalysis, report only the individual results and flag with a "*".

GRAIN SIZE Method ASTM D422-63 must be followed except as indicated below:

- Sieve analysis and hydrometer analysis are requested. Sieve requirements are specified in Section 3.6 of the Method. For quantitative determination of the distribution of particle sizes larger than 75 μm, retained on the No. 10 (2.00 mm) sieve, follow the procedure specified in Section 6 of the Method.

- The hydrometer portion of the analysis must be performed with a ASTM approved hydrometer (Section 3.3). The origin, model serial number, and manufacturer must be recorded on the data sheets.

- The analytical balance used to determine the mass of each fraction must comply with the requirements described in Section 3.1 of the Method. The balance must be checked daily prior to weight determination with Class "S" weights and the calibration must be within the manufacturer's tolerance limits, or the balance must be recalibrated. The calibration must be recorded and reported.

- Each sample must be sieve analyzed in duplicate. The results must agree within $\pm$ 20.% or another sample is analyzed. If the criterion is still not met, report all individual results and flag them with a "*". The grain size must be performed down to the No. 10 sieve.

- Each sample for hydrometer analysis may be analyzed once.

- The thermometer must be certified accurate $\pm$ 0.5°C and must be checked daily.

- The water bath must be kept at a constant temperature of 20°C and the temperature must be checked before and after the analysis of each sample and recorded.

- The composite correction for the hydrometer reading must be performed as in Section 7 of method. The hydrometer must not be in error by more than 10% or another hydrometer must be used.

- The hydroscopic moisture must be measured as in Section 8 of the method.

MOISTURE CONTENT

Method ASTM D2974-87 must be followed except as indicated below:

- ASTM Method D2974-87 lists several methods for determining Moisture Content. Method B (Section 7) will be used to determine moisture content.

- The sample size used must be sufficient to produce no less than 20 grams of dry soil when the determination is complete. Any free water must be decanted and discarded prior to analysis.

- The Method D2974-87 does not stipulate exact times for air drying or oven drying although it must be oven dried a minimum of 16 hours at 105°C. The air drying of the sample is not necessary and the sample can be weighed directly provided the 20 gram minimum is observed. The oven dried sample must be brought to constant weight. The temperature of the oven must be checked and adjusted if necessary prior to sample determination.

- The desiccation cooling time must be constant for all samples and blanks and must be recorded in the laboratory notebook. Thirty (30) minutes to a maximum of sixty (60) minutes is adequate for uniform cooling.

- The analytical balance must be checked once per 8 hour day. A method blank must be run on an empty drying dish. The blank results for weight loss must be accounted for in the evaluation of sample results.

- Run each sample in duplicate. The %D results must be ± 10.%. Report the mean if the criterion is met. If not, reanalyze the sample once. If the ± 10.% criteria is not met on reanalysis, report all individual results and flag them with a "*".

pH EPA SOW OLM01.9, Exhibit D for Semivolatiles, Section II, Part 1.7.1.1, Page D-16/SV must be followed except as indicated below:

- The sample size will be no less than 50 grams of homogenized soil. Do not decant any free water. If the moisture content of the soil is more than 50.%, then the sample aliquot must be increased to insure that at least 50 grams of soil is used to determine pH.

- Calibrate the pH meter according to the manufacturer's instructions using three pH buffer solutions of 4, 7, and 10 pH units. Record the expiration dates of the pH solutions in the laboratory logbook.

- Run each sample in duplicate. The results must agree within ± 10.% or another sample is analyzed. If the criterion is still not met, report all individual results and flag them with a "*".

ALL ANALYSES

- M&E will provide the laboratory with the airbill number and the number of samples shipped prior to the arrival of each shipment at the laboratory. If the shipment does not arrive as scheduled, the laboratory will use the airbill number to track the shipment and notify M&E (Dr. Brian Tucker at (617) 224-6433). Within two days of sample receipt, the laboratory will fax a signed copy of the sample chain-of-custody (or other form which identifies the number and condition of samples received) to M&E, ATTN: Dr. Brian Tucker, at fax number (617) 224-5927.

- The laboratory will use the M&E case number and sample number (DAM####) when reporting sample results.

8. QUALITY CONTROL REQUIREMENTS

Total Organic Carbon/Lloyd Kahn Method QC Requirements
Detection Limit: 100. mg/kg

QC Checks Required	Frequency of QC Checks	Limits	Corrective Actions
Standard Calibration Curve	Daily, prior to blank and sample analysis	Correlation Coefficient $\geq$ 0.995	Generate a new calibration curve until a correlation coefficient of $\geq$ 0.995 is achieved. Limits must be met prior to sample analysis.
Continuing Calibration Check (Mid-Range standard)	1 per 10 analytical samples and at end of analytical run	$\pm$ 10%	If outside limits, recalibrate instruments and rerun samples analyzed since last compliant continuing calibration check.
Calibration Check Standard	Daily, prior to blank and sample analysis, and at the end of the analytical sequence	$\pm$ 15% of true value	If the criterion is not met for the initial check standard, generate a new standard curve and reanalyze the check standard. The criterion must be met prior to sample analysis. If the final check standard is outside the limits, reanalyze all samples analyzed after the last acceptable check standard.
Method Blank	Daily, prior to sample analysis and 1 per 20 samples analyzed	<50 mg/kg	If the concentration is $\geq$ 50 mg/kg, determine the source of contamination, and prepare and analyze a new blank. All associated samples must be reanalyzed. Repeat until criterion is met.
Laboratory Duplicate	Perform on every sample	%D $\leq$ 20%	If the %D is outside the limits, repeat the analysis of both the sample and duplicate. If the %D is still outside the limits, flag the results with a "*".
Matrix Spike (Mid-Range concentration)	1 per SDG	80-120% R	If the %R is outside the limits, repeat the analysis. If %R is still outside the limits, flag the matrix spike and unspiked sample results with a "#".
CO_2 Scrubber Efficiency	Daily	No response	Follow manufacturer's instructions. Limits must be met prior to sample analysis.

TCO QC Requirements
Detection Limit: 100. mg/kg

QC Checks Required	Frequency of QC Checks	Limits	Corrective Actions
Oven temperature check	Once per 8 hour day, check prior to sample analysis	105°C ± 2°C	Readjust oven. Limit must be met prior to sample analysis.
Muffle furnace temperature check	Once per batch of samples analyzed at the same time	750°C ± 10°C	Readjust furnace and rerun any affected samples.
Method Blank	Once per batch of 10 analytical samples	≤ 50. mg	Clean all ceramic ware and rerun all affected samples.
Balance Check	Once per 8 hour day	± 0.2 mg	Contact service representative. Balance must be within limit prior to any weight determination.
Duplicate	All analytical samples	%D ≤ 15.%	If outside the limits, repeat analysis. If still outside limits, flag results with an asterisk ("*").

Grain Size QC Requirements

Laboratory must submit sieve certification performed within six months prior to sample analysis, complying with E11 Specification for Wire-Cloth Sieves for Testing Purposes, Annual Book of ASTM Standards, Volume 14.02.
Detection Limit: 0.1 mg (down to #200 mesh sieve), lowest discernible gradation of the hydrometer

QC Checks Required	Frequency of QC Checks	Limits	Corrective Actions
Duplicate	All samples	%D ≤ 20.%	If outside the limits, repeat analysis. If still outside limits, flag the results with an asterisk ("*").
Balance Check	Daily prior to sample analysis	± manufact. tolerance	Check the balance for malfunctioning, recalibrate. Limits must be met prior to sample analysis.
Hydrometers 151H, 152H Check	Daily prior to sample analysis	± 10.%	Check the temperature and insure 20°C and retest. If out, go to new hydrometer and test. Must meet specification prior to analysis of samples.
Thermometer Check	Daily prior to sample analysis	± 0.5°C	Replace the thermometer and retest.

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Moisture Content QC Requirements
Detection Limit: 0.01 g

QC Checks Required	Frequency of QC Checks	Limits	Corrective Actions
Duplicate	All samples	%D ≤ 10.%	If outside the limits, repeat the analysis. If still outside limits, flag the results with an asterisk ("*").
Balance Check	Once per 8 hour day	± manufact. tolerance	Check the balance for malfunctioning, recalibrate. Limits must be met prior to sample analysis.
Oven temperature check	Once per 8 hour day, check prior to sample analysis	105°C ± 2°C	Readjust oven. Limit must be met prior to sample analysis.

pH Determination QC Requirements
Detection Limit: 0.1 pH unit

QC Checks Required	Frequency of QC Checks	Limits	Corrective Actions
Duplicate	All samples	%D ≤ 10.%	If outside the limits, repeat the analysis. If still outside limits, flag the results with an asterisk ("*").

9. ANALYTICAL DELIVERABLES

The laboratory deliverables must resemble as closely as possible a Contract Laboratory Program (CLP) RAS data package as possible. The data package must be of good readable copy quality and any missing deliverable must be provided to M&E within 48 hours from the time requested at no additional charge. The following items are required as documented deliverables.

Analytical Deliverables Required for TOC Analysis:

- Narrative explaining all anomalies and corrective actions taken. Included in the narrative should be a tabulation of the M&E sample numbers with the corresponding laboratory sample numbers. The narrative must encompass all samples and field QC samples in the sample delivery group.

- Tabulated sample results which include positive results and quantitation limits for non-detected results. Report both TOC analysis results, the average of the two analyses and the %D. All soil/sediment results must be in dry weight, reported in mg/kg. Report the percent solids.

- Laboratory analysis notebook pages or bench sheets, all sample raw data such as preparation logs, analysis logbooks and all instrument strip charts and print-outs for all runs of samples and associated standards and QC samples. Include scrubber efficiency check results.

- Tabulate the Matrix Spike results (%R), the Calibration Check Standards (%D), Continuing Calibration Check (%D), and blank results. The measured value and the actual concentration of spikes must be provided with the percent recovery tabulation. The raw data for the analyses specified above must be provided.

- Standard curve raw data with instrument print-outs and concentrations. Provide plotted Standard Curves. Include the linear regression equation.

- Example of sample result calculations for each analysis. All equations, dilution factors, and information required to reproduce the laboratory results must be provided.

- Original signed chain of custody forms, sample tags, custody seals, and shipping airbills. Copies of all telephone logs and communications between M&E and the laboratory.

- Copies of the sample log in sheets containing recorded cooler temperatures and sample preservation checks, must be delivered with the package.

Analytical Deliverables Required for TCO Analysis:

- A narrative explaining all anomalies and corrective actions taken. Included in the narrative should be a tabulation of the M&E sample number with the corresponding laboratory sample number. The narrative must encompass all samples and field QC samples in the sample delivery group.

- All moisture contents must be tabulated for samples and blanks.

- All total combustible organic contents must be tabulated for samples and blanks.

- A record of balance calibration must be recorded in a notebook and copied as a deliverable per 8 hour day.

- A record of the thermometer calibration must be recorded in a notebook and copied as a deliverable per 8 hour day. The temperature of the drying oven must be recorded per 8 hour day in the notebook.

- A record of temperature calibration of the muffle furnace must be recorded in a notebook and copied as a deliverable per 8 hour day. The temperature of the muffle furnace must be recorded per 8 hour day in the notebook.

- All weights must be recorded and tabulated.

- A sample calculation must be included with each day's sample tabulation.

- Results must be recorded in mg/kg dry weight basis for total combustible organic content. Moisture content will be recorded in a percent basis.

- All copies of laboratory notebook pages with raw data must be included.

- Original signed chain of custody forms, sample tags, custody seals, and shipping airbills. Copies of all telephone logs and communications between M&E and the laboratory.

- Copies of the sample log in sheets that record cooler temperature and sample preservation checks must be included.

Analytical Deliverables Required for Grain Size Distribution:

- Narrative explaining all anomalies and corrective actions taken. Included in the narrative should be a tabulation of the M&E sample number with the corresponding laboratory sample number. The narrative must encompass all samples and field QC samples in the sample delivery group.

- The report must include Sections 18.1.1, 18.1.2, 18.1.3.1, 18.1.3.2, 18.1.4, 18.1.5, 18.1.6 from Method D 422-63 and all the hydrometer specifications from ASTM E100.

- Laboratory analysis notebook pages or bench sheets; all sample raw data.

- Tabulated results of duplicate analyses and percent moisture results. Report both analytical results, the average of the two analyses and the %D.

- Tabulated results of hydroscopic moisture of samples.

- Example of sample result calculations for each analysis. All equations and information required to reproduce the laboratory results must be provided.

- Record of the water bath temperature before and after analysis of samples.

- Original signed chain of custody forms, sample tags, custody seals, and shipping airbills. Copies of all telephone logs and communications between M&E and the laboratory.

- Copies of the sample log in sheets that record cooler temperature and sample preservation checks must be included.

Analytical Deliverables Required for Moisture Content Determination:

- Narrative explaining all anomalies and corrective actions taken. Included in the narrative should be a tabulation of the M&E sample number with the corresponding laboratory sample number. The narrative must encompass all samples and field QC samples in the sample delivery group.

- A record of balance calibration must be recorded in a notebook and copied as a deliverable per 8 hour day.

- A record of temperature calibration of the oven must be recorded in a notebook and copied as a deliverable per 8 hour day. The temperature of the oven must be recorded per 8 hour day in the notebook.

- All weights must be recorded and tabulated.

- Tabulated results of duplicate analyses and percent moisture results. Report both analytical results, the average of the two analyses and the %D.

- A sample calculation must be included with each day's sample tabulation.

- Moisture content will be recorded in a percent basis.

- All copies of laboratory notebook pages with raw data must be included.

- Original signed chain of custody forms, sample tags, custody seals, and shipping airbills. Copies of all telephone logs and communications between M&E and the laboratory.

- Copies of the sample log in sheets that record cooler temperature and sample preservation checks must be included.

Analytical Deliverables Required for pH Determination:

- Narrative explaining all anomalies and corrective actions taken. Included in the narrative should be a tabulation of the M&E sample number with the corresponding laboratory sample number. The narrative must encompass all samples and field QC samples in the sample delivery group.

- A record of the pH meter calibration must be recorded in a notebook and copied as a deliverable per 8 hour day.

- Tabulated results of duplicate analyses and percent moisture results. Report both analytical results, the average of the two analyses and the %D.

- All copies of laboratory notebook pages with raw data must be included.

- Original signed chain of custody forms, sample tags, custody seals, and shipping airbills. Copies of all telephone logs and communications between M&E and the laboratory.

- Copies of the sample log in sheets that record cooler temperature and sample preservation checks must be included.

Complete Sample Delivery Group File (CSF) Audit

Due to the litigative nature of each Superfund site, Region I EPA requires that all analytical data, quality control data, and tabulated raw or supporting data be delivered with each sample delivery group (SDG). With each sample delivery group, an EPA Region I Complete SDG File Completeness Evidence Audit must be carried out. The CSF Completeness Evidence Audit Forms are included in Attachment A and must accompany each sample delivery group (data package). The laboratory through these audit forms must demonstrate that each piece of sample data, raw data, calibration data, and any other data requirements of this specification are included by the laboratory in the data package.

The forms included in Attachment A are for all types of data packages. For this specification the laboratory will use the forms supplied to the best of their ability where deliverable items are applicable.

10. EXCEPTIONS

If QC requirements or action limits are exceeded; or if analytical samples are destroyed or lost; or if matrix interference is suspected; or there are any other problems contact:

Dr. Brian Tucker
Metcalf & Eddy Inc.
30 Harvard Mill Square
P.O. Box 4071
Wakefield, MA 01880-5371
Phone (617) 224-6433
Fax (617) 245-6293

APPENDIX B

M&E STANDARD OPERATING PROCEDURE (SOP) FOR SAMPLE SHIPPING AND PACKAGING

STANDARD OPERATING PROCEDURE
ENVIORNMENTAL SAMPLE PACKAGING AND SHIPPING

HWD-A-6b

FEBRUARY 12, 1993

REVISION NO. 1

STANDARD OPERATING PROCEDURE
ENVIRONMENTAL SAMPLE PACKAGING AND SHIPPING

1. PURPOSE

The purpose of this procedure is to establish a standard method for determining the proper sample handling, packaging, and shipping of environmental samples to ensure adequate sample integrity to meet EPA requirements and to ensure that the packing and shipping requirements of U.S. Department of Transportation (DOT) are met.

2. SCOPE

This document establishes the rationale to determine the hazardous classification, proper shipping name, proper packaging, marking and labeling and shipping of environmental samples. Environmental samples are small quantities of water, soil, sediment, sludge, air, animal tissue, plant tissue, or chemical taken expressly for the purpose of chemical analysis, physical analysis, or treatability study. This document establishes the responsibility for ensuring sample integrity as well as ensuring proper hazardous handling of environmental samples. All environmental samples must be considered hazardous and must be handled, packaged and shipped as such. the overriding consideration addressed here is the assurance that the environmental samples have been handled, packaged, and shipped within DOT regulations and with the utmost regard to safety as well as, reaching the analytical laboratory without damage. The samples must have met preservation requirements and must agree with accompanying chain-of-custody record.

3. RESPONSIBILITIES

3.1 Quality Assurance Manager

The project Quality Assurance Manager is responsible for

- Ensuring that personnel are aware of the EPA and DOT requirements for sample handling, packaging, storage and shipping of environmental samples from the field to the analytical laboratory.

- Ensuring that personnel are properly trained in packaging, marking, labeling, and producing shipping papers for environmental samples.

3.2 Field Chemist/Sampler/Engineer

The project field sampling personnel are responsible for

- Verifying and attachment of appropriate traffic report/chain-of-custody records to accompanying samples in transit and the placement of custody seals on sample containers to ensure sample integrity

- Determining the hazard classification, proper shipping name, packaging, marking, labeling and shipping papers for environmental samples to be transported from the field to the analytical laboratory.

- Relinquishing the samples to the laboratory via overnight carrier as appropriate

- Verifying visual and air monitoring survey of samples prior to shipment.

- Informing the laboratory of sample shipment and estimated time of arrival.

- Complying with the overnight carrier requirements, IATA regulations, and DOT regulations for shipping.

4.0 REFERENCES

Code of Federal Regulations

49 CFR 171.8 Hazardous Material Definition
49 CFR 172.101 and Names and "By Analogy"
49 CFR 171.101 and 171.11-12a Shipping Names
49 CFR Parts 173, 178, 179 Packaging
49 CFR 172,300 Marking
49 CFR 172,400 Labeling
49 CFR 172,200 Shipping Papers
49 CFR 172,500 Placards
49 CFR 171.15 171.16 Emergency, Incident
40 CFR 302 Hazardous Substance Release
40 CFR 355 SARA Releases
International Air Transportation Association (IATA)
Dangerous Goods Regulations

5. PROCEDURES

5.1 Introduction

This standard operating procedure will outline and example the hazard classification, packaging, shipping, marking labeling, and shipping documentation of unknown hazardous environmental samples (soil, water, etc.) to be shipped to a analytical laboratory. All samples taken at a hazardous water site or a site under investigation for hazardous substances must be shipped as hazardous and must be treated as such. Samples taken from a site where the contamination has been previously characterized must be packaged, labeled, and shipped according to the known hazard regulations. Samples containing free phase chemicals must be handled as high hazards and treated as pure chemical products.

5.2 Objectives

The objectives for establishing hazard classification naming, handling, packaging storing shipping marking labeling and proper shipping documentation include:

- Establishing a hazard classification, proper name, proper packaging, marking, labeling, and shipping documentation
- Packaging environmental samples to ensure minimal breakage during sample transport
- Preservation of samples per analytical protocol
- Proper storage of samples prior to shipment to ensure sample integrity
- Complying with DOT, IATA, and overnight carrier regulations and rules for transportation by surface and/or air
- Ensuring proper chain-of-custody protocol during sample shipment

5.3 Classification of Hazardous Materials

The proper way to establish the hazard classification for a known chemical, an unknown chemical, or a unknown environmental sample such as soil, sediment, water, sludge, or air samples is to refer to the 49 CFR 171.8 and 49 CFR 172.101. This reference defines hazardous substances, materials, and clarifies the seven methods in which materials may be classified as hazardous. Table 172.101 details the proper shipping name, hazard class, or division ID number, as well as other pertinent information.

If during the planning of a project the field sampler, chemist, or field engineer knows the exact nature of chemical contamination they will encounter at the project site then that knowledge can be used to determine if the samples are hazardous. Sample can be deemed hazardous by characteristics definition, hazardous due to being on the 49 CFR 172.101 list, hazardous due to reportable quantity spill, or by the fact that the mixture contains a component that is hazardous or that the material is by Analogy hazardous. When the material is a free product that can be identified the exact shipping name must be used. If the material is mixed with soil, water, or other environmental media and the sampler knows that some portion of that mixture is hazardous the waste could be defined as:

- Hazardous Waste liquid N.O.S. NA3082
- Hazardous Waste Solid N.O.S. NA3077

In most cases the project planners cannot predict what they will find nor will they be able to identify with any certainty the material found. When soil, sediment, sludge, water or other environmental media is sampled and the constituents of the sample is totally unknown, then the sampler will use the definitions:

Environmentally hazardous substance liquid N.O.S. UN3082
Environmentally hazardous substance solid N.O.S. UN3077

The dangerous air bill in Figure I details the proper designation.

5.4 Proper Shipping Name

Once the field personnel have classified their sample then they can move on to choosing a shipping name. Table 172.101 gives the proper shipping names or hazardous material description. This proper shipping name can also be used to determine the maximum reportable quantity if there were to be a spill of the material. The proper name can also be used to satisfy the IATA naming regulations and determine the requirement of a dangerous airbill. The airbill in Figure 1 shows the proper way to prepare the dangerous goods airbill. A proper DOT shipping name includes: the proper shipping name; the hazard class; the UN/NA identification number; and the packing group number. This information is provided in the DOT 49 CFR 172.101 table.

5.5 Packaging

After the classification has been performed and the proper shipping name has been decided then the proper packing is chosen. Table 172.101 also describes the proper packaging. That table leads the reader to 49 CFR 173, 178 and 179. Specifically 49 CFR 173.3, 173.4 packing and exemption, exemptions for small quantities are important. The area where the environmental sampler must be acutely aware is 49 CF 173.24. This section discusses the integrity of the package, compatibility of the contents, closures etc. The environmental sampler must be aware of the materials being packaged and take precautions so that if the contents of the outer package (cooler) were spilled, that spill would not be considered a release of hazardous waste and would require a major clean-up. One area the sampler must also be acutely aware of is; the rules surrounding small quantities of chemical preservatives such as nitric acid,

FEDERAL EXPRESS

AIRBILL

PACKAGE TRACKING NUMBER 2906775421

46 9198M 2906775421

Sender's Federal Express Account Number: 0021-0142-4 | Date:

From (Your Name) Please Print: STAN HATFIELD | Your Phone Number (Very Important): ( )

Company: METCALF & EDDY

Department/Floor No.:

Street Address: 10 HARVARD MILL

City: WAKEFIELD | State: MA | ZIP Required: 0 1 8 8 0

To (Recipient's Name) Please Print: | Recipient's Phone Number (Very Important): ( )

Company: | Department/Floor No.:

Exact Street Address (We Cannot Deliver to P.O. Boxes or P.O. Zips): | City: | State: | ZIP Required:

YOUR BILLING REFERENCE INFORMATION (FIRST 24 CHARACTERS WILL APPEAR ON INVOICE)

IF HOLD FOR PICK-UP, Print FEDEX Address Here: Street Address: | City: | State: | ZIP Required:

PAYMENT: ☐ Bill Sender ☐ Bill Recipient's FedEx Acct. No. or Credit Card No. in Account Number below ☐ Bill 3rd Party FedEx Acct. No. or Credit Card No. in Account Number below ☐ Bill Credit Card No. in Credit Card Number below ☐ Cash — Expiration Date: /

SERVICES	DELIVERY AND SPECIAL HANDLING	PACKAGES	WEIGHT IN POUNDS ONLY	DECLARED VALUE (See rate)	FEDEX USE ONLY
1 ☐ PRIORITY 1 Overnight Delivery Using Your Packaging. When using ICAO/IATA regulations, please mark appropriate box(es). Do not mark for CFR 49.	1 ☐ HOLD FOR PICK-UP (Fee in Box 4)				
• Dangerous Goods as per attached Shipper's Declaration ☐	2 ☐ DELIVER WEEKDAY				
• Dangerous Goods Shipper's Declaration not required ☐	3 ☐ DELIVER SATURDAY (Extra charges) ☐				
• Cargo Aircraft only ☐	4 ☒ DIRECT SIGNATURE (Extra charges)				
2 ☐ STANDARD AIR Delivery not later than second business day	5 ☐ CORPORATE SURVEILLANCE SERVICE (CSS) (Extra charges Premium Signature not required)				
	6 ☐ DRY ICE • Dangerous Goods Shipper's Declaration not required • Dry Ice, 9, UN 1845, ____ x ____ kg				
	7 ☐ OTHER	Total	Total	Total	

Received At: 1 ☐ Regular Stop 2 ☐ On-Call Stop 3 ☐ Drop Box 4 ☐ B.S.C. 5 ☐ Station

FEDEX Corp. Employee No.:

Date/Time for FEDEX Use:

SERVICE CONDITIONS, DECLARED VALUE AND LIMIT OF LIABILITY

Use of this airbill constitutes your agreement to the service conditions in our current Service Guide which is available upon request. See back of sender's copy of this airbill for further information.

We will not be responsible for any claim in excess of $100 per package, whether the result of loss, damage, delay or non-delivery, unless you specify a higher amount in the space to the left, pay 40¢ per additional $100 specified and document your actual loss. In the event of a claim, Maximum amount limitations found in the current Federal Express Service Guide apply. Your rights to recovery from Federal Express for loss of the intrinsic value of the package, as well as for loss of sales, income, interest, profit, attorneys' fees, costs and any other form of damage whether direct, incidental, consequential or special is limited to the greater of $100 or the declared value specified in the left. In no event shall your recovery exceed your actual loss.

In the event of unlawful delivery, Federal Express will at your request and with some limitations, refund all transportation charges paid. See Service Guide for further information.

Federal Express Use	
Base Charge	
Declared Value Charge	
Other 1	
Other 2	
Total Charges	

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ORIGIN COPY

2906775421 AIRBILL NUMBER

SHIPPER'S CERTIFICATION FOR RESTRICTED ARTICLES/DANGEROUS GOODS

CHECK ONE ➡ ☐ 49 CFR ☒ IATA/ICAO (TYPE OR PRINT)

DANGEROUS GOODS IDENTIFICATION: PROPER SHIPPING NAME	CLASS OR DIVISION	UN/ID NO.		PACKING GROUP	QUANTITY AND TYPE OF PACKAGING
RQ - Environmental Hazardous Substance. Liquid, NOS	9	UN-3082		9	DESCRIBE PACKAGING & QUANTITY HERE
RQ - Environmental Hazardous Substance. Solid, NOS	9	UN-3077		III	
LIST ANALYSES:					

ADDITIONAL HANDLING INFORMATION

TRANSPORT DETAILS	THIS SHIPMENT IS WITHIN THE LIMITATIONS PRESCRIBED FOR ➡	PASSENGER AIRCRAFT	~~CARGO AIRCRAFT ONLY~~	(DELETE-NON-APPLICABLE)
AIRPORT OF DEPARTURE: CITY	AIRPORT OF DESTINATION: CITY	SHIPMENT TYPE ➡ NON-RADIOACTIVE	~~RADIOACTIVE~~	(DELETE-NON-APPLICABLE)

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

I HEREBY DECLARE THAT THE CONTENTS OF THIS CONSIGNMENT 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: Susan Pogodzienski, M&E | PLACE AND DATE:

EMERGENCY TELEPHONE NUMBER: 1-800-535-5053 | SIGNATURE OF SHIPPER: Susan Pogodzienski | SEE WARNING ON BACK

Figure 1. Federal Express Airbill for Dangerous Goods

hydrochloric acid, and sodium hydroxide. Shipment of these materials in any quantity are subject to many regulations described in 49 CFR 171-178. For environmental samples the general packaging scheme for both environmentally hazardous NOS solids and liquids and hazardous wastes NOS solids and liquids is as follows.

The general packaging procedures shall be as follows:

- Place a layer of cushioning material (e.g., vermiculite) in the bottom of the watertight insulated metal or equivalent strength plastic shipping containers.
- Wrap the properly labeled and secured glass sample bottles and purgeable vials with plastic bubble wrap. Place the wrapped containers into watertight zip lock bags and seal the bags closed.
- Place sample bottles (top side up) into the shipping container arranging the bottles so that the glass bottles are surrounded by plastic bottles.
- Using the necessary absorbent packing material, pack the sample bottles to ensure that they do not shift during transport.
- Fill any void spaces of the shipping container, around and on top of the sample bottles, with ice cubes or chips sealed in plastic bags or with blue ice packages. Federal express will not allow the use of ice cubes or chips. Contact the local representative for instructions.
- Seal the appropriate chain of COC form(s) or Packing Lists in a zip-lock plastic bag, and tape it securely to the inside of the shipping container lid.
- Close and lock/latch the shipping container. Seal the space between the container body and lid with waterproof tape. (If the shipping container used is a picnic cooler, tape the drain plug closed to prevent any leakage of water as the ice packs melt during transport.)

- Apply several wraps of chain-of-custody tape around the shipping containers perpendicular to the seal to ensure that the lid remains closed if the latch is accidentally released or damaged during shipment. Do not obscure any stickers or labels on the shipping container with the chain-of-custody tape.

- Place a completed overnight carrier airbill (see Figure 1 for an example of a properly completed Federal Express Dangerous Goods Airbill) on the lid of the shipping container. Include the name, address, and telephone number of the receiving laboratory and the return address and telephone number of the shipper on the airbill.

- Place a "This End Up" label on the lid and on all four sides of the shipping container.

- Place required DOT hazard label on all four sides of the shipping container.

- Each shipping container must not weigh more than 150 pounds if it is to be shipped overnight by Federal Express. This may vary depending on geographical area.

- The shipper must be notified prior to sampling in order to schedule the hazardous material shipment. Depending on the hazard class the environmental sample may not be shipped with edible commodities.

When the environmental sampler encounters neat chemical products in soil, on top of water, or at the bottom of the water column then he/she must consult 49 CFR 173, 178 and 179 for packaging. The small known toxic, high concentration liquid or solid must be handled with much more care. The sample container or bottle must be left with a small headspace for expansion and contraction. The container must be solid and have good integrity. The sample container must fit inside a paint can. The following is the procedure to use for known toxic, or medium to high concentration samples:

1. Label the container and close the container tightly leaving a small headspace.

2. Rap the sample container in bubblewrap.

3. Place the container in a polyethelene plastic bag with either a ziplock or some type of twist secured closure.

4. Place wrapped container into a paint can containing 1/3 vermiculite. Fill the remaining void space with vermiculite.

5. Place the lid on the paint can container and firmly press the lid into the grooved closure position. If gently tapping with a rubber hammer is necessary to get full closure, then do so. Care must be taken not to dent or ruin the grooved closure.

6. Place at least 4 metal spring clips onto container lid and container and press into position to lock on the cover.

7. Label the paint can with sample designation. Mark (See later section marking and labeling) the paint can to indicate hazard.

8. Pack paint cans as if they were common samples in a cooler using the same care taken with less toxic environmental samples.

9. Vermiculite or absorbent packing material in cooler.

The packaging must meet the criteria 173.27 for overnight transportation by aircraft. The outer containment must meet the criteria in parts 178, 179. The common Coleman camping type cooler used in the past for environmental samples may not be applicable. The individual type brand must be checked to ascertain if the container meets the DOT specification.

5.6 Chain-of-Custody

An integral part of the environmental sampling episode is the documentation of the sample. The sample must be identified by affixing a label, tag, or some type container etching. The sample must be recorded in a log. The sample must be recorded on a chain-of-custody form, cross referencing the sample to a specific location and sampling time. The chain-of-custody is a record of all sample custody transfers. A sample is considered to be in a person's custody if it is:

- In a persons possession
- In view after being in physical possession
- Locked so that no one can tamper with it after having been placed in physical custody
- In a secured area, restricted to authorized personnel

The chain-of-custody forms must be completed in such a manner to ensure concise and accurate sample transfer. An example of a standard chain-of-custody form (COC) is included in Figure 2. The COC Form(s) are placed in a locking plastic bag (ziplock) and taped to the inside of the cooler lid.

In the case of sample shipment by an overnight airborne courier or land-based carrier, a properly prepared air bill, bill of lading, or shipping will serve as an extension of the chain-of-custody form while the samples are in transit.

Figure 2. Chain of Custody Form

M&E Metcalf & Eddy

CHAIN OF CUSTODY FORM

Job/Project Name	Job/Project Location	Job/Project Number
Samplers (Signatures)	Recorder (Signature)	Date
Lab (Samples Sent To)		

SAMPLING		SAMPLE NUMBER	SAMPLE LOCATION	MATRIX		COMPOSITE/GRAB	PRESERVATIVE (Y/N)	ANALYSIS REQUESTED						COMMENTS
Date	Time			Water	Soil								Total #	

Relinquished By (Signature)	Date	Time	Received By (Signature)	Relinquished By (Signature)	Date	Time	Received By (Signature)
Relinquished By (Signature)	Date	Time	Received By (Signature)	Relinquished By (Signature)	Date	Time	Received By (Signature)
Relinquished By (Signature)	Date	Time	Received for Lab By (Signature)	Date	Time	Comments	
Method of Shipment							

Distribution Original to Lab Copy 1 to Field Files, Copy 2 to Project Manager

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5.7 Marking and Labeling

Each package of environmental samples known to contain hazardous materials must be appropriately marked and labeled.

Marking: Marking must be:

Durable
In English
On a background of a sharply contrasting color.
Unobscured by labels or attachments
Away from other markings which could substantially reduce their effectiveness (49 CFR 172,300)

For non bulk packages, markings should include

The proper shipping name
The ORM marking where applicable (49CFR 171.8)
The UN or NA Number
The name and address of the shipper, the consignee or both, and additional marking appropriate to material being shipped.

For non bulk packages where liquids are being shipped. The following applies (49 CFR 172.312):

- Packaged with closures upward
- Legibility marked with package orientation marking that conform pictorially to ISO standard 780-1985, on two opposite vertical sides of the package with arrows pointing in the correct upright direction.

Environmental samples that are liquids such as water from a monitoring well must be packaged and legibly marked with the correct upward orientation. Samples of any liquid, known hazardous or not known hazardous must be packaged as such.

Labeling. Environmental samples are usually not hazardous due to their unknown characteristics. Label requirements are listed for each regulated substance in the 49 CFR 172.101 table. In some cases such as benzene both the primary hazard and a subsidiary hazard must be labeled. For example, benzene is regulated by the DOT as both a flammable liquid and poison.

When a sample of a high toxic material such as dioxin (2,3,7,8-Tetrachlorodibenzo p-dioxin) or a high concentration PAH is the environmental sample to be shipped, then the labels must indicate the extra hazard or a special precaution to be taken during transportation. An example is the "Cargo Aircraft only" label used for hazardous materials shipped by air which are prohibited from passenger aircraft. The labels must be properly affixed to the cooler as per 49 CFR 172. 400

5.8 Shipping Papers

It was said previously, that shipping papers are the Chain-of-custody for environmental samples. Since all environmental samples originating from a hazardous waste site or suspected hazardous sites must be shipped as hazardous materials then the shipping papers must comply with 49 CFR 172.200. Each overnight express company has their own set of regulations that are normally more restrictive than those in 49 CFR 172-179. The sampler must have those requirements in hand to properly plan a field episode. All environmental samples going by air to the laboratory must use a hazardous airbill (see Figure 3) that is properly prepared. If environmental samples are

shipped over land, delivered by courier, or delivered by sampling personnel, then a bill of lading or shipping order is required. Environmental samples no matter what mode of transportation, must have shipping papers. When a description of hazardous material is required to be included on a shipping paper that description must conform to the requirements in 49 CFR 172.202. General entries include:

- Contents
- Name of Shipper
- Continuation page
- Emergency response telephone number

The dangerous goods air bill in Figure 1 describes the proper name, hazard class, UN number, quantity and packing group. The airbill details the shipper, and the destination as well as the type of cargo aircraft. The title and name of the shipper, the shippers signature and an emergency telephone number are also included.

5.9 Placards

When hazardous or dangerous samples are shipped by carrier over the highway or delivered by sampling personnel then the vehicle must be placarded. Refer to 49 CFR 172.500 to determine the requirements. Most shipments of environmental samples are below the 1000 lb rule and thus do not require placarding. Shipping papers and proper packaging, marking and labeling are required.

5.10 Incidents and Emergencies

Any incident or emergency involving the shipment of hazardous material sin transit must be immediately reported to the next level of management, AWT Corporate Health and Safety Officer, AWT Corporate Legal, AWT Corporate Insurance Officer and AWT Corporate Communications. Notifications to the DOT (Emergencies 49 CFR 171.15; Incident 49 CFR 171.16), and EPA ("Hazardous Substance" Releases 40 CFR "SARA;' releases 40 CFR 355 and other agencies may be necessary.

6.0 RECORDS

All Chain-of-Custodies, airbills, bills of lading, shipping paper must be retained in the project file and must be available for inspection at any time.

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