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FINAL SAMPLING AND ANALYSIS PLAN AND FINAL QUALITY ASSURANCE PLAN SITE 8

NCBC GULFPORT MS

10/1/2004

ENVIRONMENTAL CHEMICAL CORPORATION

FINAL
SAMPLING AND ANALYSIS PLAN

Site 8 – Herbicide Orange Storage Area and Off-Base Area of Contamination

Naval Construction Battalion Center
Gulfport, Mississippi

October 2004

Prepared for:

Southern Division
Naval Facilities Engineering Command
2155 Eagle Drive
North Charleston, SC 29406

Prepared under:

Contract Number N62467-02-D-0468
Contract Task Order 002

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FOR

SITE 8 – HERBICIDE ORANGE STORAGE AREA

AND

OFF-BASE AREA OF CONTAMINATION

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LIST OF ACRONYMS AND ABBREVIATIONS

AOC	Area of Concern
BSTS	Bench Scale Treatability Study
BSTSWP	Bench Scale Treatability Study Work Plan
CAS	Columbia Analytical Services, Inc
CCI	CH2M Hill Constructors, Inc.
CEGS	Corps of Engineers Guide Specifications
CIH	Certified Industrial Hygienist
COC	Contaminants of Concern
CQC	Contractor Quality Control
cy	cubic yards
DI	Distilled Water
DQCR	Daily Quality Control Report
DQI	Data Quality Indicator
DQO	Data Quality Objective
ECC	Environmental Chemical Company
EPA	United States Environmental Protection Agency
ER	Engineer Regulation
FSP	Field Sampling Plan
HLA	Harding Lawson Associates
HO	Herbicide Orange
IATA	Industrial Air Transport Association
IDW	Investigation Derived Waste
LQAM	Laboratory Quality Assurance Manual
MDEQ	Mississippi Department of Environmental Quality
MS/MSD	Matrix Spike/Matrix Spike Duplicate
µg/kg	microgram per kilogram
Navy	United States Navy, Southern Division, Naval Facilities Engineering Command
NCBC	Naval Construction Battalion Center
ng/kg	nanograms per kilogram
OSHA	Occupational Safety and Health Administration
pg/kg	picograms per kilogram
PHSM	Project Health and Safety Manager
PM	Project Manager
PPE	Personal Protective Equipment
PRM	Program Manager
QA	quality assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QC	quality control
QCM	Quality Control Manager
QCP	Quality Control Plan
QCSR	Quality Control Summary Report

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

RA	Remedial Action
SAP	Sampling and Analysis Plan
SHSP	Site Health and Safety Plan
SOP	Standard Operating Procedure
SOUTHDIV	Southern Division, Naval Facilities Engineering Command
SPLP	Synthetic Precipitation Leaching Procedure
SRT	sediment recovery trap
SSHO	Site Safety and Health Officer
TCDD	2,3,7,8-tetrachlorodibenzo-p-dioxin
TRG	target remediation goal
TEQ	total toxicity equivalents
TtNUS	Tetra Tech, Inc. NUS
USAF	United States Air Force
WP	Work Plan

1.0 INTRODUCTION

This Field Sampling Plan (FSP) is prepared by Environmental Chemical Corporation (ECC) for the United States Navy, Southern Division, Naval Facilities Engineering Command (Navy), Contract Number N62467-02-D-0468, Contract Task Order 002, for the Remedial Action (RA) of the Site 8 Herbicide Orange (HO) Storage Area and Off-Base Area of Contamination located at the Naval Construction Battalion Center (NCBC) in Gulfport, Mississippi (Figure 1-1).

The FSP is Part I of the Sampling and Analysis Plan (SAP). The FSP is intrinsically related to the Quality Assurance Project Plan (QAPP) and, as such, is considered a companion document to the QAPP. The QAPP is Part II of the SAP. All work described in the FSP will be performed in compliance with federal, state, and local regulatory requirements to the extent applicable. The purpose of the FSP is to provide a comprehensive description of field sampling and associated activities to be conducted during this project. The procedures required to ensure that data obtained during sampling activities are of acceptable and verifiable quality and attain project data quality objectives (DQOs) are discussed in Section 4.0 of the QAPP.

This SAP provides sufficient detail regarding the field sampling requirements so that it may be used as an audit guide for field and laboratory work. Copies of the SAP will be provided to the analytical laboratory and made available to the appropriate field personnel.

As part of the RA being performed at Site 8, ECC will:

- Perform a bench-scale treatability study to identify treatment parameters capable of achieving the site physical and chemical criteria for remediation;
- Excavate dioxin-contaminated sediment from on-base drainage channels contiguous to Site 8 and from an associated off-base area of concern (AOC) located north of the base and excavate soil ash located at Site 8;
- Perform verification sampling at the excavations;
- Consolidate, homogenize, and stabilize soil ash and contaminated sediment within a portion of Site 8;
- Perform confirmation sampling of the stabilized material;
- Construct a rolled-concrete cap over the stabilized material;
- Restore the on-base drainage channels and off-base AOC affected by excavation activities;
- Implement land-use controls; and
- Perform long-term monitoring.

1.1 Project Background

NCBC Gulfport is located in the southeastern corner of Mississippi, approximately two miles north of the Gulf of Mexico. The base is located in the western part of the city of Gulfport in Harrison County (Figure 1-1). The off-base AOC is located north of NCBC, across 28th Street near Outfall 3. The base occupies 1,100 acres with an average elevation of approximately 30 feet above sea level, the only significant exceptions being two rectangular piles

of bauxite (aluminum ore) stored on the surface that are approximately 45 feet higher than the adjacent ground. A map of NCBC Gulfport is provided as Figure 1-2.

Site 8 consists of three contiguous storage areas (areas A, B, and C) located in the north-central portion of NCBC Gulfport (Figure 1-2). The main former HO drum storage area, Site 8A, encompasses approximately 13 acres, has an undulating surface due to previous remedial activities, and is covered with light vegetation. The surface soil in non-stabilized areas is typically fine to medium sand. Approximately one-third of Site 8A consists of stabilized areas where HO drums were stored. Site 8A includes the upper reaches of the drainage areas for the eastern two-thirds of the base. Surface drainage from Site 8A flows to the northwest, exiting the base at Outfall 3 into a drainage system that feeds Canal No. 1. Canal No. 1 flows north to Turkey Creek (TtNUS, 2003a).

Prior to 1995, the surface water that exited the base via Outfall 3 discharged to wetlands (the off-base AOC) that are a part of the Turkey Creek drainage basin (HLA, 2000). Sites 8B and 8C encompass approximately 18 acres, are relatively flat, and have very little vegetation. Sites 8B and 8C were also used for storage of drums containing HO. The Site 8B and 8C surface soils consist of fine to medium sand, and approximately one-third of these areas are stabilized with Portland cement. Sites 8B and 8C are also located at the head of local drainage basins. Surface water from Site 8B flows north and exits the base at Outfall 4, discharging to the Turkey Creek drainage basin. Surface water from Site 8C drains to the southeast, exiting the base at Outfall 2 into Brickyard Creek (TtNUS, 2003a).

1.2 Site History and Contaminants

Prior to 1968, Site 8 was used as an equipment storage and staging area. Around 1961, the surface soils were stabilized with Portland cement to provide a hardened surface for heavy equipment operation and storage. Between 1968 and 1977, Site 8 was used by the United States Air Force (USAF) as a storage area for drums containing HO. In 1977, the HO drums were removed from Site 8, transported to port by railroad, and placed on a ship for destruction by incineration in the South Pacific. The release of dioxins at Site 8 was confirmed in 1977, and the site was fenced and left inactive until 1985 (TtNUS, 2003a). Approximately 850,000 gallons of HO was thought to have been originally stored on the 13 acres of Site 8. This 13-acre area is currently referred to as Site 8A (HLA, 2000).

In 1985, the USAF began operations to clean up the dioxin-contaminated soils that remained on site following the removal of the drums of HO. Contamination of soils resulted from spills and leaks during the 10 years that HO was stored at Site 8. A Research, Development and Demonstration permit obtained through the United States Environmental Protection Agency (EPA) Region IV, allowed the USAF to conduct test burns and, thus, demonstrate that incineration was capable of reducing dioxin concentrations in site soils to less than 1.0 microgram per kilogram (µg/kg), which was the EPA criterion in 1985. During the test burns, two additional areas outside the original 13 acres were identified and verified as previous storage locations for drums containing HO. These two areas were designated as Sites 8B and 8C.

Following EPA acceptance of the test burn data, full-scale incineration of dioxin-contaminated soils from Sites 8A, 8B, and 8C was conducted. The incineration process was conducted within the boundaries of Site 8A and was completed in 1988. The ash that remained from the incineration process was stored and currently remains on Site 8A. Although the soils within Sites 8A, 8B, and 8C were incinerated, the drainage channels that carry surface water and sediment from these sites to the lower reaches of the local drainage basins were not addressed during this remedial effort (HLA, 2000).

Between 1987 and January 2001, access to Site 8A was restricted and operations were not conducted within site boundaries. Since January 2001, activities conducted within Site 8 include the construction of a new loading ramp in anticipation of using the site as a storage and staging area (TtNUS, 2003a) and the performance of a pilot-scale treatability study for remediating soil ash and contaminated on-base and off-base AOC sediments (TtNUS, 2001b). In August 2002, the Navy performed a sediment removal action in the drainage channels of Sites 8B and 8C. Approximately 2,600 cubic yards (cy) of sediments were excavated from 3,800 linear feet of drainage channels and were transported to Site 8A.

Remedial activities were also conducted within the off-base AOC. As part of pilot-scale activities in November and December 2001, dioxin-contaminated sediment located within the off-base AOC was excavated and transported to Site 8A. Approximately 1,000 cy of material located on property owned by Mr. H. A. Edwards (the Edwards property) were excavated. A sediment recovery trap (SRT) was installed at the western extent of the excavation to prevent recontamination of this area from upgradient sources. Verification sampling demonstrated that dioxin concentrations (total toxicity equivalents [TEQ] of 2,3,7,8-tetrachlorodibenzo-p-dioxin [TCDD]) were below the Mississippi Department of Environmental Quality (MDEQ) unrestricted Tier 1 target remediation goal (TRG) of 4.26 nanograms per kilogram (ng/kg) (TtNUS, 2002). In April 2003, 30 cy of sediment contaminated with dioxin were excavated from an area adjacent to a culvert that runs beneath Canal Road. The excavation was performed to enable the city of Gulfport to perform a culvert replacement project. An SRT was installed at the eastern extent of the excavation (TtNUS, 2003c).

For additional details on site history, refer to Section 1.3 of the Remediation Planning Document (HLA, 2000).

1.3 Summary of Existing Site Data

For an overview of previous investigations performed at Site 8, refer to Section 2.3 of the 100% Remedial Design (TtNUS, 2004). A complete list of references for reports generated from previous studies is included in Section 11.0 of this FSP.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The ECC project organization chart identifying key personnel, responsibilities, and lines of authority is provided in Figure 2-1. A discussion of the roles of the key project personnel is provided in the sections that follow. If changes to the key quality control staffing are required, ECC will discuss the proposed change(s) with the Navy prior to making the change(s).

2.1 Program Manager

Mr. Raghu Arora will be the Program Manager (PRM) for this project. Program Manager duties and responsibilities include the following:

- Overall contract conformance to Navy and NCBC requirements and specifications, including technical, cost, and schedule;
- Overall responsibility for the success and proper execution of the project;
- Review of all required submittals;
- Designation of the Project Manager (PM) and Quality Control Manager (QCM); and
- Allocation of sufficient resources to ensure successful completion of the project.

2.2 Project Manager

Mr. Prashant Khanna will be the PM for this project. The PM reports directly to the PRM and will officially represent ECC in all project-related activities. The PM duties and responsibilities include the following:

- Initiation of project planning and implementation of project;
- Management of the project budget and schedule, with concurrence from the PRM, thereby, ensuring project requirements are satisfied;
- Management of all field construction activities, including the direction of project staff and subcontractors in accordance with project requirements;
- Tracking proposed changes to the project;
- Communicating directly with the Navy and the NCBC regarding project execution and accountability;
- Coordination with the QCM to ensure compliance with standard protocols and procedures and implementation of the ECC Work Plan (WP) and Quality Control Plan (QCP);
- Coordination with the Project Health and Safety Manager (PHSM) and the Navy to ensure implementation of the Site Health and Safety Plan (SHSP);
- Maintaining a project logbook of removal activities; and
- Procurement of equipment, material, and supplies.

2.3 Quality Control Manager

The ECC QCM for this project is Mr. Bob Anderson. The QCM will report directly to the PRM and have the following responsibilities:

- Supervising all quality control (QC) aspects of the project to ensure compliance with project plans and the scope of work as defined in the QCP;
- Overall management of the Quality Control Program and the authority to act independently in all QC matters;
- Coordinating and supervising field sampling activities;
- Approval of all submittals and supervision of all QC procedures; and
- Maintenance of communication between project management and project team members and designation as the primary spokesman on quality matters when interfacing with external organizations.

2.4 Site Health and Safety Specialist

The Site Health and Safety Specialist (SHSS) for this project will be responsible for the following:

- Implementation and enforcement of the SHSP;
- Ensuring site compliance with Federal, State and Occupational Safety and Health Administration (OSHA) safety and health regulations;
- Coordinating modifications to the SHSP with the ECC Safety and Health Manager, the Certified Industrial Hygienist (CIH), the Field Superintendent, and the Navy; and
- Maintaining the Health and Safety Logbook.

2.5 Field Personnel

Potentially contaminated soil and other hazardous and non-hazardous waste removal will be performed by HAZWOPER trained technicians, equipment operators, and field crews. ECC employees and sub-contractors will be certified as per federal, state, and local statutes.

2.6 Analytical Laboratory

Columbia Analytical Services, Inc (CAS) will be used during the project to perform laboratory analytical services. State laboratory certifications with reference to this project will be provided. The address and phone number for CAS are:

10655 Richmond Ave., Suite 130A
Houston, Texas 77042
Phone: 713-266-1599
Fax: 713-266-0130

3.0 PROJECT SCOPE AND OBJECTIVES

In the course of the Remedial Actions at the Site 8, ECC will complete the following tasks outlined in the WP for the site:

- Completion of work plans;
- Pre-mobilization and mobilization;
- Site-setup;
- Preparation of Site 8A;
- Field scale demonstration;
- Stabilization and compaction of soil;
- Offsite wetland area and on-base sediment excavation;
- Backfilling and site restoration;
- Roller compacted concrete cap;
- Wetland restoration; and
- Site restoration and demobilization.

A description of each task planned for this project is given in Section 7.0 of the project WP. Applicable regulations and/or standards that will be followed in completing this project also are outlined in Section 4.0 of the WP.

3.1 Chemical Data Quality Objectives

The quality assurance (QA) objective for this project is to develop and implement procedures for obtaining and evaluating data that meet the DQOs defined in the Section 4.0 of the QAPP. QA procedures are established to ensure field measurements, sampling methods, and analytical data provide information that are comparable and representative of actual field conditions, and that the data generated are technically defensible. Specifically, chemical data will be generated to determine if remediation goals and treatment criteria are achieved.

The measurement quality objective and the laboratory QC acceptance criteria are defined in terms of Data Quality Indicators (DQIs), as described in Section 4.0 of the QAPP. Data that meet the QA objectives and goals will be deemed acceptable. Data that do not meet objectives and goals will be reviewed on a case-by-case basis to ascertain its usefulness. Section 8.0 (data recording, reduction, etc.) of the QAPP describes how DQIs will be evaluated. When possible, corrective actions will be taken to bring data within the QA acceptability goals.

Adherence to the procedures in the QAPP also will ensure that data of sufficient quality are collected during the sampling programs. The use of the QAPP allows the development and implementation of procedures for obtaining and evaluating data in a manner that will result in a quantitative and qualitative representation of the DQIs. The precision, accuracy, and completeness DQIs provide a quantitative measure of the statistical significance of the data collected in this field program. The representativeness and comparability DQIs utilize documentation of the field and laboratory procedures to qualitatively evaluate the data.

3.2 Project Schedule

A detailed project schedule for the anticipated sequence of remediation activities is provided in Section 13.0 of the QCP.

4.0 FIELD ACTIVITIES BY AREA OF CONCERN

This section presents the sampling strategy and technical approach to be used in the collection of the following types of samples:

- Bench-scale testing samples;
- Excavation verification samples;
- Soil stabilization samples;
- Roller-compacted concrete cap samples; and
- Borrow material and top soil samples.

Information provided for each of these activities includes a brief description of the field activity, media to be sampled, number and location of samples to be collected, sampling method, and field and laboratory analytical methods to be performed. Sampling activities will be performed in accordance with ECC's Corporate Standard Operating Procedures (SOPs). The following SOPs are included in this FSP as Appendix A.

- E-302 Equipment Decontamination
- E-601 Sample Handling and Custody Requirements
- E-603 Field Documentation
- E-604 Data Management
- E-605 Data Assessment
- E-801 Soil Sampling
- E-1101 Field Quality Control Samples

Table 4-1 provides a summary of sampling activities with estimated quantities for all type of samples to be collected including required QA/QC samples. Duplicate laboratory samples will be obtained during each sampling activity for QA/QC analysis as described in Section 5.0. Details of the analytical requirements for each sampling activity are presented in Table 4-2. Table 4-3 presents quantitation limits and remediation goals for all parameters.

4.1 Bench-Scale Testing

Following the approval of the Bench-Scale Treatability Study Work Plan (BSTSWP), ECC will collect soil samples from off-base and on-base areas to perform bench scale testing. The sample collection locations will be based on the existing concentration contours available from the 100% design report. Areas with the highest concentrations of dioxins will be sampled.

Six samples from the off-base area, six samples from an on-base drainage location, and six samples from a soil ash pile will be collected using 5-gallon buckets and sent to the laboratory for both chemical and geophysical analyses as outlined in the BSTSWP.

Kemron Inc. will implement the BSTSWP and report on all finding in a Treatability Study Report. The report will present all findings related to the untreated material characterization, pre-treatment testing on several material blend combinations, mix ratio evaluations – screening phase, and mix ratio evaluations – verification phase

4.2 Excavation Verification Sampling

Post-excavation verification sampling refers to sampling of an excavation from which contaminated material has been removed. Post-excavation samples will be collected from excavation floors and sidewalls and analyzed in accordance with EPA SW-846 Methods 1312 and 8290 to verify that dioxin levels at the excavation boundaries are below the project cleanup levels.

The number, density, frequency, locations, and depths of verification samples will be in accordance with the Verification Sampling and Analysis Plan for Site 8 (TtNUS, May 2003c). Prior to the collection of verification samples for laboratory analysis, field screening of soil samples for dioxin will be conducted to determine the locations of laboratory samples. ECC personnel will collect samples from the excavation floors and sidewalls for field screening of dioxins, also in accordance with the Verification Sampling and Analysis Plan for Site 8 (TtNUS, May 2003). Root material and other debris will be excluded from both field screening and laboratory samples to the extent possible. Samples collected for laboratory analysis will be homogenized in a decontaminated stainless steel bowl and placed into the appropriate sample container(s).

Discrete samples will be collected from the excavation floor. Samples also will be collected from excavation sidewalls, if appropriate. A sample represents the particular area of the excavation from which it is collected (i.e., the floor, the north sidewall, the east sidewall, etc). Fewer samples will be collected from smaller excavations; however, at least one floor sample and one sidewall sample for each sidewall will be collected from each excavation.

For excavations of ditches, discrete samples will be collected from the excavated ditch sidewalls. Each sample will represent that particular section of the ditch.

If analytical results indicate that more excavation is required in an area, additional excavation will be performed and additional verification samples will be collected.

4.3 Soil Stabilization Sampling

Using the results achieved through the BSTS, ECC will perform soil blending and will add a stabilization agent as described in the RA/CA WP prior to spreading of the stabilized blend. Throughout the blending, stabilization, and placement of stabilized material in the landfill, field testing will be performed as described in Specification 02160A. This section presents the sample collection and analytical requirements for each step in the process.

4.3.1 Field Demonstration Material Blend Sample Collection

In preparation for the field demonstration, representative samples of soil ash and contaminated soil and sediment will be collected at random locations within the limits of disturbance. The samples will be analyzed in accordance with EPA SW-846 Methods 1312 and 8290 to verify that on-base sediments have a dioxin concentration of at least 500 ng/kg and off-base sediments have a dioxin concentration of at least 250 ng/kg.

4.3.2 Field Demonstration Stabilized Material Sample Collection

After the contaminated material has been processed for the field demonstration, a random representative sample of the stabilized material will be collected and analyzed in accordance with EPA SW-846 Methods 1312 and 8290 to verify that the dioxin concentration of the SPLP extract is less than 30 picograms per kilogram (pg/kg).

4.3.3 Full-Scale Operations Material Blend Sample Collection

ECC will collect samples by taking three incremental specimens at random from source material to make a proportional composite sample of the blend components. Samples will be a minimum of 150 pounds. Thereafter, during the course of the project, one random sample will be collected from each 7,000 tons of material blend created. The samples will be taken at random to make a composite sample of not less than 50 pounds. Repeat the sampling when the source of the material is changed or when unacceptable deficiencies or variations from specified grading of materials are indicated by testing.

4.3.4 Full-Scale Operations Stabilized Material Sample Collection

Representative samples of the stabilized material will be collected and analyzed in accordance with EPA SW-846 Methods 1312 and 8290 to verify that the dioxin concentration of the SPLP extract is less than 30 picograms per kilogram (pg/kg). One random sample will be collected for every 5,000 cy of blended material, immediately after the material has been blended.

4.4 Roller-Compacted Concrete Cap Sampling

If not prequalified, any aggregate, additives, and cement used for the roller-compacted concrete cap will be sampled and tested for the parameters and characteristics detailed in Specification 02755A. Results of the tests, if required, will be provided to the Contracting Officer prior to using the materials for preparing the concrete.

After operations begin, aggregate samples will be collected at the mixing plant for the following tests:

- Sieve analysis (ASTM C 136 and ASTM C 117) – one prior to start of work and one for every 500 cy, or portion thereof, of concrete placed each shift;
- Quality parameters (Los Angeles abrasion, magnesium sulfate soundness, clay lumps, friable particles, lightweight pieces, soft particles, specific gravity) – one for every 5,000 cy of concrete placed or when a visual change is observed in the aggregate; and
- Moisture content (ASTM C 566) – one at the beginning of the day.

Samples of the concrete mixture will be tested for the following:

- Moisture-density (ASTM D 1557) – two for initial lot (test section) and at least one for each lot thereafter or whenever the mixture proportions change; samples will be collected from plant mixer discharge;
- Field density and moisture content (ASTM C 1040 and ASTM D 3017) – at least one for every 100 feet of pavement of each lift and at least one for every 100 linear feet of longitudinal and transverse construction joint;
- Pavement thickness (ASTM C 174/ C 174M) – at least three cores per lot within one to three feet of density test holes randomly selected by the Contracting Officer;
- Flexural strength (ASTM C 31/ C 31M) – in accordance with Paragraph 3.10.2.9, Section 02755; and
- Compressive strength (ASTM C 1176) - in accordance with Paragraph 3.10.2.10, Section 02755.

4.5 Borrow Material and Top Soil Sampling

Backfilling and site restoration will involve the following set of operations:

- Sample collection of borrow material and topsoil for chemical analyses;
- Geotechnical testing of borrow material;
- Backfilling of the excavated areas with borrow material;
- Placement of topsoil and seeding of the restored areas; and
- Placement of erosion control blanket in the ditch swales.

Fill material for backfilling excavations will be analyzed for chemical parameters and tested for geotechnical parameters as specified in Section 1.6 of the Specification 02315N. One composite sample from each structural fill, common fill, and topsoil source will be analyzed or tested for the following:

- TPH using EPA Method 1664 (EPA Method 418.1 is no longer recommended by the EPA);
- BTEX using EPA Method 5030/8020;
- Ignitability, corrosivity, and reactivity using EPA Method 1311 for extraction and SW846 7.3.3.2 for analysis;

- Sieve analysis using ASTM C 136 (for structural fill);
- Liquid limit and plastic limit using ASTM D 4318; and
- Moisture-density using ASTM D 698 or ASTM D 1557.

At each location, sample material sufficient to complete all chemical analyses and geotechnical tests will be collected using a decontaminated stainless spoon. The material will be placed into a decontaminated stainless steel bowl and thoroughly mixed. Each sample container will then be filled with the soil mixture. The sample containers will be sealed, packaged, and transported to the laboratory following the procedures outlined in Table 4-2 and in Section 5.0 of this FSP.

5.0 FIELD QUALITY CONTROL

A series of quality samples will be collected in the field and submitted to the laboratory for analysis. These samples are analyzed for the purpose of assessing the quality of the sampling effort and the analytical data.

5.1 Quality Control Samples

QC samples are those samples collected in duplicate that will be submitted to the project laboratory as blind samples and/or blank samples collected to assess the quality of the sampling effort. QC samples will be analyzed for the same parameters as the field sample. Results from the QC duplicate samples will be compared to data from the appropriate field sample to assess the consistency and quality of data produced from the laboratory.

QC field duplicate samples will represent at least 10% of the field samples. Every tenth sample will be collected in sufficient volume to provide a minimum of two (2) portions. One (1) portion will be identified as the field sample, and one (1) portion as the QC duplicate sample. A third portion may be required as a QA sample. QC blank samples are generally collected once per day of sampling and/or once per cooler shipped to the laboratory.

The following QC samples will be collected during each day of sampling at GAFB:

- 10% QC samples (field duplicates) for all parameters;
- 5% of the samples will include sufficient volume (3X) of material for the required matrix spike/matrix spike duplicate (MS/MSD) aliquots;
- One (1) rinsate or equipment blank collected per day; and
- One (1) temperature blank for each cooler containing field samples.

5.1.1 Field Duplicate Samples

Field duplicate samples are samples collected in quantity at the same time and location and under the same conditions as the original sample. Field duplicate or triplicate samples are used to estimate the overall precision of a data collection activity. Replicate samples are collected at the same time and homogenized in the field so they are equally representative of sample conditions at a given point in space and time. The homogenized sample is split into a field sample, a QC sample, and, if required, a QA sample.

5.1.2 Rinsate and Equipment Blanks

Rinsate or equipment blanks are samples of distilled (DI) water poured over decontaminated sampling equipment. The rinsate blank is collected during the final rinse of the sampling equipment after decontamination procedures are performed. Rinsate blanks are analyzed for the same parameters as field samples to evaluate the effectiveness of field decontamination procedures. Equipment blanks will be collected at a rate of one (1) per day when non-disposable field sampling equipment is decontaminated.

5.1.3 Temperature Blanks

Temperature blanks are bottles of water packaged in each sample cooler, allowing the laboratory to determine the temperature of the shipment without disturbing the field samples. One (1) temperature blank is placed in each cooler with the samples.

5.2 Sample Containers and Preservation Procedures

All samples collected will be preserved according to U.S. Environmental Protection Agency (EPA) protocols established for the parameters of interest. Appropriate measures will be taken to ensure that requirements with respect to temperature are maintained during transport to the laboratory, and prior to log-in and storage at the laboratory.

Sample preservation, container, and holding time requirements were identified based on EPA-SW846 protocols. These requirements are summarized in Table 4-2.

The analytical laboratory will supply sample bottles, containers, and preservatives to field personnel. Sample bottles and containers will be free of target analytes and of known quality (i.e., I-Chem 200 series or equivalent), as documented by the container manufacturer.

5.3 Equipment Decontamination

Equipment decontamination will be performed before and after soil sample collection at each sampling location. All sampling equipment will be decontaminated as follows:

- Clean with non-phosphate surfactant using a brush if necessary to remove particulate matter and surface films, and rinse in distilled water;
- Rinse a final time with DI water and allowed to air dry;
- Completely wrap sampling equipment in aluminum foil, shiny side out, to prevent contamination during transportation;
- Clean, disposable gloves will be worn while handling sampling equipment during the final stages of decontamination. DI water will be stored in glass or Teflon containers and applied via Teflon squeeze bottles; and
- Equipment or materials not used immediately after decontamination will be placed on a plastic sheet, covered with plastic, and secured to avoid potential contamination.

5.4 Personnel Decontamination

Personnel decontamination procedures will be followed during sampling to prevent cross-contamination between samples and to protect sampling personnel from potential dangers associated with the contaminants present at the site. Please refer to Section 10.0 of SHSP for complete personnel decontamination procedures.

6.0 FIELD OPERATIONS DOCUMENTATION

A stringent program of custody procedures will be utilized to ensure that each sample is accounted for from the time of collection to analysis. Documentation in logbooks and Chain-of-Custody Records will be employed to maintain a comprehensive record of sample collection, transfer between personnel, shipment, and receipt by the laboratory. This section outlines the procedures that will be followed to document sample history and integrity.

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict chain-of-custody procedures. To maintain and document sample possession specific procedures are to be followed. A sample is considered to be in an individual's custody if the sample is:

- In the physical possession or view of the responsible party;
- Secured to prevent tampering; or
- Placed in a restricted area by the responsible party.

6.1 Daily Quality Control Reports

Daily Quality Control Reports (DQCRs) will be prepared from field sampling notes, observations, and audit reports for each day of the project in which field activity occurs. The following information will be noted on the DQCRs:

- Job and site numbers;
- Location of work;
- Weather conditions including temperature, wind speed and direction, barometric readings and significant wind changes, etc.;
- Description of work performed including Quality Assurance/Quality Control (QA/QC) samples collected and calibration information;
- Problems encountered and associated corrective action;
- Description of field tests performed, including the individuals performing the tests, test results, QC check results, and calibration procedures;
- Results of audits or inspections, including problems identified and corrective actions taken;
- Signature of responsible authority and initials of all persons conducting changes or corrective actions; and
- General comments.

If necessary, the laboratory will provide ECC with reports concerning any out-of-control analytical events. These reports will be attached to the DQCRs and contain the following:

- Holding time violations;
- Chain-of-Custody discrepancies;
- Sample storage and preservation errors;
- Out of control QA/QC sample results; and
- Corrective actions taken as a result of the above problems.

DQCRs and any laboratory reports will be assembled and provided to the Navy CO on a weekly basis.

6.2 Field Logbook and/or Sample Field Sheets

A bound field logbook will be used to document all field operations and will contain sufficient data and information to reconstruct field activities for a specific day. Pages in the logbook will be bound and numbered. All entries will be recorded legibly in indelible ink. At the end of each day, the last page will be signed and dated by the author(s) and a line drawn through the remainder of the page. At a minimum, the daily log will contain:

- Date and time the field work started;
- Names and titles of sampling personnel;
- Purpose of the sampling;
- Location and description of the sample and sample site;
- Date and time each sample was collected;
- Any deviations from the FSP;
- Meteorological conditions at the start of sampling and changes in these conditions;
- Record of any field measurements observed;
- The number and type of samples collected and the sample numbers;
- Packaging information; and
- Sample destination.

Errors on field documents will be corrected by drawing a line through the error and entering the correct information. Errors on a field document should be corrected by the person who made the original entry, and the erroneous information should not be obliterated. All corrections will be initialed and dated.

In addition to the field logbook, as site map will be maintained indicating the locations of all post-excavation samples. The sample designation will correspond with the sample location number on the map. The site map, Chain-of-Custody Records and corresponding analytical data packages will be submitted to the Navy.

6.3 Photographic Records

Color photographs will be taken of site activities to record important features of the site prior to the commencement of work, during activities, and after work is complete. A camera-lens system with a perspective similar to the naked eye will be used; telephoto or wide-angle photographs are not suitable for legal purposes. Before work begins, a minimum of 10 exposures will be taken. After site activities have started, the project will be photographically recorded as it progresses. In addition, at least one photograph will be taken from the same overall view at successive periods during the project.

After completion of work, a minimum of 10 photographs will be taken including an overall view of the site. The following information about each photograph will be recorded in the field logbook:

- Photographer's name;
- Date and time of the photograph;
- General camera direction;
- Brief description of the subject and the field work portrayed in the photo; and
- Film roll number of the photograph.

6.4 Sample Documentation

This section describes the sample numbering system, the sample labels, the custody seals, and the Chain-of-Custody Records to be used in the collection of the project samples.

6.4.1 Sample Numbering System

Every sample will be given a unique sample designation for identification purposes. The numbering system will be coordinated with the project chemist and the Navy chemist to ensure that the proposed sample identifiers are discrete. The sample identification will be a series of letters and numbers consisting of three (3) or four (4) character strings, identifying the specific sample as follows:

The following is an example sample numbering system.

NN-HH-LL-dd-aaa or WT-Nu-HH-LL-dd-aaa

- NN is the grid, material stockpile, or excavation from which the sample is collected,
- HH is the sample type identifier as follows;
  - PE = Pre-Excavation Soil
  - PV = Post-Excavation Verification Soil
  - PB = Pre-Treatment Material
  - PS = Post-Treatment Solidification Material
  - BS = Borrow material
- LL describes the sampling location as follows;
  - IS = in-situ
  - SW = excavation sidewall
  - FL = excavation floor
  - SP = soil stockpile
- "dd" describes the depth from which the sample was collected:
- "aaa" represents the sample number for a specific sample location.

For example: Sample No. 40-PE-IS-04-003 indicates that the sample is:

- Excavation 40
- Pre-excavation soil collected in-situ at a depth of 4 feet bgs; and
- Sample 3.

All logbooks, sample labels or tags, custody seals, representative sampling documents, and chain-of-custody documents will be completed using these sample designations. QC samples will be assigned unique sample designations in the same way as field samples. A two (2)-digit designation will be added at the end of the sample number to identify the QC samples in the field logbook as described below:

- QC = Quality Control sample;
- RB = Rinsate Blank sample;
- FB = Field Blank; and
- TM = Temperature Blank.

The additional two (2) digit QC designation will not be placed on the sample jars or the Chain-of-Custody Record; therefore, the QC samples will not be identified as such to the laboratory.

6.4.2 Sample Labels and/or Tags

A sample label, as presented in Appendix B, will be attached to each sample container and completed legibly with indelible ink. The sample labels will be affixed to the sample bottle and covered with clear tape. The labels will identify the name and initials of the sample collector, date and time of sample collection, place of collection, sample number, analysis required, preservatives added; and designation between grab and composite samples. Grab samples are individual soil samples and duplicate samples collected from one (1) location at one (1) time. Composite samples are grab samples that are combined and homogenized representing several locations collected at different times.

6.4.3 Custody Seal and Chain-of-Custody Records

The custody seal, as presented in Appendix B, will be attached to the outside of the shipping container in such a manner that the seal must be broken to allow access to the container. The custody seal will provide the sample collection date and the sampler's signature or initials.

All sample shipments will be accompanied by the Chain-of-Custody Record (Appendix B) identifying its contents. This record will be used to document sample custody transfer from the sampler, to other sampling team members (if necessary), to the courier, and finally to the analytical laboratory. The Chain-of-Custody Record ensures that samples can be traced from the time of field collection until they are received and analyzed by the analytical laboratory. The original custody record is shipped along with the samples, while the initiator of the record retains a copy.

The information required for the Chain-of-Custody Record includes:

- Type of sample (grab or composite) and matrix;
- Analytical method numbers and parameter names;
- Sample number;
- Signature of sampler;
- Date and time of sample collection;
- Project name, location and address; and
- Signatures of persons involved in the chain of possession.

When responsibility for a group of samples changes several times, each custodian is not required to retain a copy of the Chain-of-Custody Record, as long as the original custody record indicates that each person accepting the samples has subsequently relinquished custody appropriately. Chain-of-custody forms will be completed according to the following protocol:

- The originator fills in all requested information from the sample labels;
- The originator signs the "Relinquished by" box and keeps the copy;
- The original record sheet is shipped with the samples. A plastic shipping envelope will be taped to the inside of the cooler top and the remaining two (2) copies of the Chain-of-Custody Record will be filed with the representative sampling documents;
- The person receiving custody checks the sample label information against the custody record. He/she also checks sample condition and notes anything unusual under "Remarks" on the custody form;
- The person receiving custody signs in the adjacent "Received by" box and keeps the original;
- The Date/Time will be the same for both signatures, since custody must be transferred between two (2) individuals. However, when samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures;
- When samples are shipped via common carrier, the original custody form is shipped with the samples and the shipper (e.g. Field Sample Custodian) keeps the copy. The shipper also keeps all shipping paper, bills of lading, etc.;
- In all cases, it must be readily seen that the person receiving custody has relinquished it to the next custodian; and
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the Chain-of-Custody Record.

6.5 Field Analytical Records

For this project, ECC will record:

- Calibration procedures and frequency for the field tests;
- The source of calibration materials;
- Equipment decontamination procedures and schedules;
- The quality control materials and frequency for the field tests;
- The quality control limits and acceptance criteria for the quality control materials;

- The acceptance criteria for calibration procedures;
- The corrective actions for out-of-control events for both calibration and quality control samples;
- the actions required by field personnel in the event that control parameters exceed the acceptance criteria; and
- The form used to document exceedence of criteria and subsequent corrective actions.

6.6 Documentation Procedures/Data Management and Retention

The ECC will maintain field records with sufficient data to recreate all sampling and measurement activities. The requirements listed in this section apply to all measuring and sampling activities. Requirements specific to individual activities are listed in the section that addresses each activity. The information will be recorded with indelible ink in a permanently bound notebook with sequentially numbered pages. These records will be archived in an easily accessible form and made available to the Navy upon request.

The following information will be recorded for all field activities:

- Location;
- Date and time;
- Identity of people performing the activity; and
- Weather conditions.

For field measurements:

- The numerical value and units of each measurement; and
- The identity of and calibration results for each field instrument will also be recorded.

The following additional information will be recorded for all sampling activities:

- Sample type and sampling method;
- The identity of each sample and depth(s), where applicable, from which it was collected;
- The amount of each sample;
- Sample description (e.g., color, odor, clarity);
- Identification of sampling devices; and
- Identification of conditions that might affect the representativeness of a sample (e.g., Refueling operations, damaged casing).

7.0 SAMPLE PACKAGING AND SHIPPING REQUIREMENTS

This section outlines how the project samples will be packaged, shipped and labeled after they have been collected.

7.1 Packaging of Samples

Standard samples for laboratory analysis will be placed in containers and preserved as described in Table 4-2. ECC will follow the procedures described in SOP E-601 (see Appendix A) for sample packaging as follows:

- Samples will be placed in glass jars with Teflon lids;
- Each sample container will be placed inside a self-sealing polyethylene bag. Sample containers will be kept upright in the cooler, with a minimum one (1) inch space between sample containers;
- Three (3) inches of inert, absorbent, packing material will be placed at the bottom of the cooler;
- Samples will be placed in the water proof metal or insulated plastic coolers;
- An additional layer of inert packing material will be placed in the cooler to partially cover the sample containers;
- A temperature blank will be placed in the cooler immediately adjacent to the sample containers;
- Double-bagged ice packs will be placed around the sample container to provide uniform cooling during shipping. At least three (3) ice packs will be used per cooler;
- All remaining space in the cooler will be filled with a packing material to provide stability during transport;
- The Chain-of-Custody Record will be placed in a self-sealing polyethylene bag and taped to the inside lid of the cooler;
- The shipping container will be closed and taped shut with duct tape or strapping tape. The drain of the cooler will be shut and sealed with duct tape;
- Custody seals will be placed over the seam at the front and rear of the cooler lid and covered with clear tape;
- The completed shipping label will be placed on the top of the cooler (see Section 7.3) along with any other required labels ("Environmental Samples", "This Side Up", "Fragile", etc.); and
- The shipper's hazardous certification form will be completed prior to shipping.

7.2 Shipping Containers for Standard Samples

All samples sent to the laboratory for analysis will be shipped overnight via Federal Express. Samples will be properly classified to assure the protection of personnel involved in the shipment of samples and to maintain the integrity of the samples. The packaging, labeling, and shipping of hazardous substances are regulated by the International Air Transport Association (IATA), Resolution 618, effective January 1, 1992. Samples shipped from the site will comply with IATA requirements.

The PM (or designated representative) will contact the laboratory as necessary to inform them of samples being shipped from the site, arrival time, and special handling or analytical procedures required. Samples will be delivered to the laboratory within 48 hours of sample collection.

7.3 Marking and Labeling of Shipping Containers

Clearly print in indelible ink the following information in unabbreviated form on a label attached to the shipping container:

- Laboratory name and address; and
- Return name and address.

Each shipping container will be labeled with "Environmental Samples" and "This Side Up" stickers on the top, upward arrows on all four (4) sides, and “Fragile” stickers on at least two (2) sides.

8.0 INVESTIGATION DERIVED WASTES

Investigation derived wastes (IDW) from the remedial activities will consist of used personal protective equipment (PPE), and disposable sampling equipment. IDW will be managed in accordance with EPA guidance for Management of IDW during Site Inspections (1992) and other applicable guidance.

IDW will be stored in an on-site location designated by the Navy. The IDW staging area will be inspected daily by ECC personnel during the field program. IDW will be treated, or otherwise disposed of in accordance with applicable Federal, state, or local regulations.

9.0 FIELD ASSESSMENT

The contractor is required to ensure that quality is maintained throughout all field work by means of a three-phase control process (Engineer Regulation (ER) 1180-1-6 and Corps of Engineers Guide Specifications (CEGS) 01450 and 01451). Contractor Quality Control (CQC) phases (preparatory, initial, and follow-up) are performed onsite by the QCM, who will summarize the activities of each CQC phase in the DQCR. The CQC phases are performed for each definable feature of work.

9.1 Three Phase Inspection Procedures

Chemical QC consists of three (3) phases (preparatory, initial, and follow-up) of control that will be performed for sampling activities. ECC also conducts a "fourth phase" or completion inspection which consists of a data review of each sampling data package.

9.1.1 Preparatory Inspection

The QCM and the sampling team will discuss sampling requirements for the project as described in Section 5.0 of this FSP. Each sampler will be given copies of Tables 4-1 through 4-3, which summarize the required sampling activities. The preparatory session will include a discussion of the following:

- Review of site hazards, both chemical and physical;
- Review of the personal protective equipment (PPE) required during sampling;
- Review of the SAP;
- Review of the sampling techniques to be used;
- Examination of the required sampling equipment and materials; and
- Review of the decontamination procedures.

The following Sampling Apparatus and Field Instrumentation Checklist will be used during the sampling activities:

- Field notebook;
- Chain-of-Custody Records;
- Stainless steel spoon or trowel;
- Stainless steel bowl;
- Sampling jars and bottles, as indicated in Table 4-2;
- Disposable gloves;
- Tyvek suits;
- DI water stored in glass or plastic containers for decontamination and equipment blanks;
- Non-phosphate liquid surfactant for decontamination;
- Bagged ice for sample preservation;

- Shipping labels for sample containers;
- Custody seals for sample coolers;
- Packing material; and
- Aluminum foil for wrapping sampling equipment for storage.

To conclude the preparatory inspection, the QCM will conduct an orientation walk-through of the sampling areas with personnel performing remediation and sampling activities. This phase will be repeated for new sampling personnel joining the team.

9.1.2 Initial Inspection

During the initial inspection, the QCM will supervise the initial sampling of each definable feature of the sampling activities to ensure that required sampling procedures are followed.

9.1.3 Follow-up Inspection

The follow-up inspection requires the QCM to review the progress of sampling activities to ensure compliance with all sampling methods. Supervision of the sample packaging and shipping, as described in Section 7.0, is part of the follow-up phase of chemical QC.

9.1.4 Completion Inspection

The QCM will perform completion inspections for each sampling event by conducting data review of each sampling data package. The data review is a systematic approach for review of the laboratory data as the first level of data evaluation. At this level of data evaluation, only the sample results and limited project documentation are typically reviewed. The data review includes examination of the following elements of the laboratory data package:

- Case Narrative;
- Data Completeness;
- Holding Times;
- Chain-of-custody form;
- Laboratory QC samples; and
- Sampling results.

The data review is performed to ensure that the data generated is complete and that there are no obvious quality problems. The completion inspection ensures that the data generated meets the requirements of the FSP and QAPP. A “punch list” of items that do not conform to the approved sampling plans and project specifications will be developed. This list of data deficiencies will be included in the QC documentation with the planned corrective actions and an estimated date of completion.

The QCM will submit any questionable data package for a second level of data evaluation, data verification. Also, as specified in the QAPP, 100 percent of all laboratory data will be submitted for data verification. The final project completion inspection will provide the following:

- Summary of the analytical results, including detection limits, in tabular format to be included in the Quality Control Summary Report (QCSR);
- Synopsis of project data evaluation reports summarizing the review process used and any specific comments pertaining to a sample or group of samples;
- An outline of QC practices employed, including problems encountered and corrective actions taken; and
- Conclusions and recommendations describing the impact of analytical results on disposal or treatment of material removed from or treated on the project site.

9.2 Contractor Quality Control

Information on Contractor Quality Control is included in a separate plan, the QCP.

10.0 NONCONFORMANCE/CORRECTIVE ACTIONS

The following sections briefly discuss field and laboratory corrective actions in response to detected deficiencies.

10.1 Field Corrective Actions

Any sampling problems or deficiencies (i.e., improper sampling, decontamination, or packaging and shipping procedures) detected during the initial or follow-up phases of quality control will be corrected immediately. After corrective actions are taken, the follow-up phase of quality control will be intensified until the QCM is satisfied that the problem is permanently corrected. The Navy will be notified as soon as possible concerning sampling problems or deficiencies and any corrective actions taken.

10.2 Laboratory Corrective Actions

In accordance with the laboratory SOPs and the project specific QAPP (see Section 7.0, Corrective Actions), any problems or potential system problems detected through the calibration check samples, QC samples, daily performance audits, and/or the Quality Assurance (QA) audits performed by the ECC QA Manager or his designee, will precipitate immediate action to find and correct the problem. Failure to meet calibration criteria, record keeping omissions, improper sampling technique, and improper storage or preservation of samples are conditions that affect data quality and require investigation and correction.

Laboratory corrective actions associated with the project are documented in the laboratory maintenance book. After the corrective actions are instituted, the system's performance is rigorously checked before continuing sample analysis.

If more than 10% of the QC values associated with a sample determination are unacceptable, the Laboratory Quality Assurance Manager (LQAM) will notify the project QCM.

11.0 REFERENCES

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Tetra Tech, Inc. NUS (TtNUS), 2001. Report for Pilot-Scale Soil/Sediment Treatability Study, Site 8, Herbicide Orange Storage Area at NCBC Gulfport, Mississippi. December, 2001.

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TtNUS, 2003a. Focused Feasibility Study, Revision 2, Site 8 Herbicide Orange Storage Area at NCBC Gulfport, Mississippi. March, 2003.

TtNUS, 2003b. Draft Site Characterization Report, Off-base Area of Contamination, NCBC Gulfport, Mississippi. April, 2003.

TtNUS, 2003c. Verification Sampling and Analysis Plan for Site 8 – Herbicide Orange Study Area and off base Area of Contamination, Naval Construction Battalion Center, Gulfport, Mississippi. May, 2003.

TtNUS, 2004. 100% Remedial Design for Site 8 – Herbicide Orange Storage Area and Off-Base Area of Contamination, Naval Construction Battalion Center, Gulfport, Mississippi. May, 2003.

TABLES

Table 4-1
Summary of Sampling Activities
Site 8 – Herbicide Orange Storage Area and Off-Base Area of Contamination
Naval Construction Battalion Center
Gulfport, Mississippi

Sampling Activity	Matrix	Estimated Number of Field Samples Per Analyte			
		Dioxins [a]	TPH [b]	BTEX [c]	TCLP [d]
Verification	Soil	400	N/A	N/A	N/A
Soil Stabilization	Soil	17	N/A	N/A	N/A
Borrow Material and Topsoil	Soil	N/A	4	4	4
Field QC Duplicates [1]	Soil	42	1	1	1
MS/MSD [1]	Soil	21	1	1	1

Notes:

a Extraction (by Synthetic Precipitation Leaching Procedure) - EPA Method 1312; Analysis – EPA Method 8290
b EPA Method 1664
c Extraction – EPA Method 5030; Analysis – EPA Method 8020
d EPA Method 1311 (Includes Ignitability, Corrosivity, and Reactivity)

[1] QC Requirements: 1 field duplicate sample per 10 field samples collected
1 matrix spike (MS) sample per 20 field samples collected
1 matrix spike duplicate (MSD) sample per 20 field samples collected
1 temperature blank per cooler
1 rinsate blank per day

Table 4-2
Analytical Requirements
Site 8 – Herbicide Orange Storage Area and Off-Base Area of Contamination
Naval Construction Battalion Center
Gulfport, Mississippi

Matrix	Parameters	Analytical Methods EPA Methods SW-846	Container *	Preservative	Holding times
Soil/Rock/Debris	Dioxins	EPA 1312/8290	4 oz glass jar	Cool to 4° C	14 days
	Total Petroleum Hydrocarbons (TPH)	EPA 1664	8 or 9 oz glass jar	Cool to 4° C	28 days
	Benzene, Toluene, Ethylbenzene, and Xylenes (BTEX)	EPA 5030/8020	4 oz glass jar	Cool to 4° C, no headspace	14 days
	Waste Characterization (TCLP with Ignitability, Corrosivity, and Reactivity)	EPA 1311	4 oz glass jar	Cool to 4° C	14 days

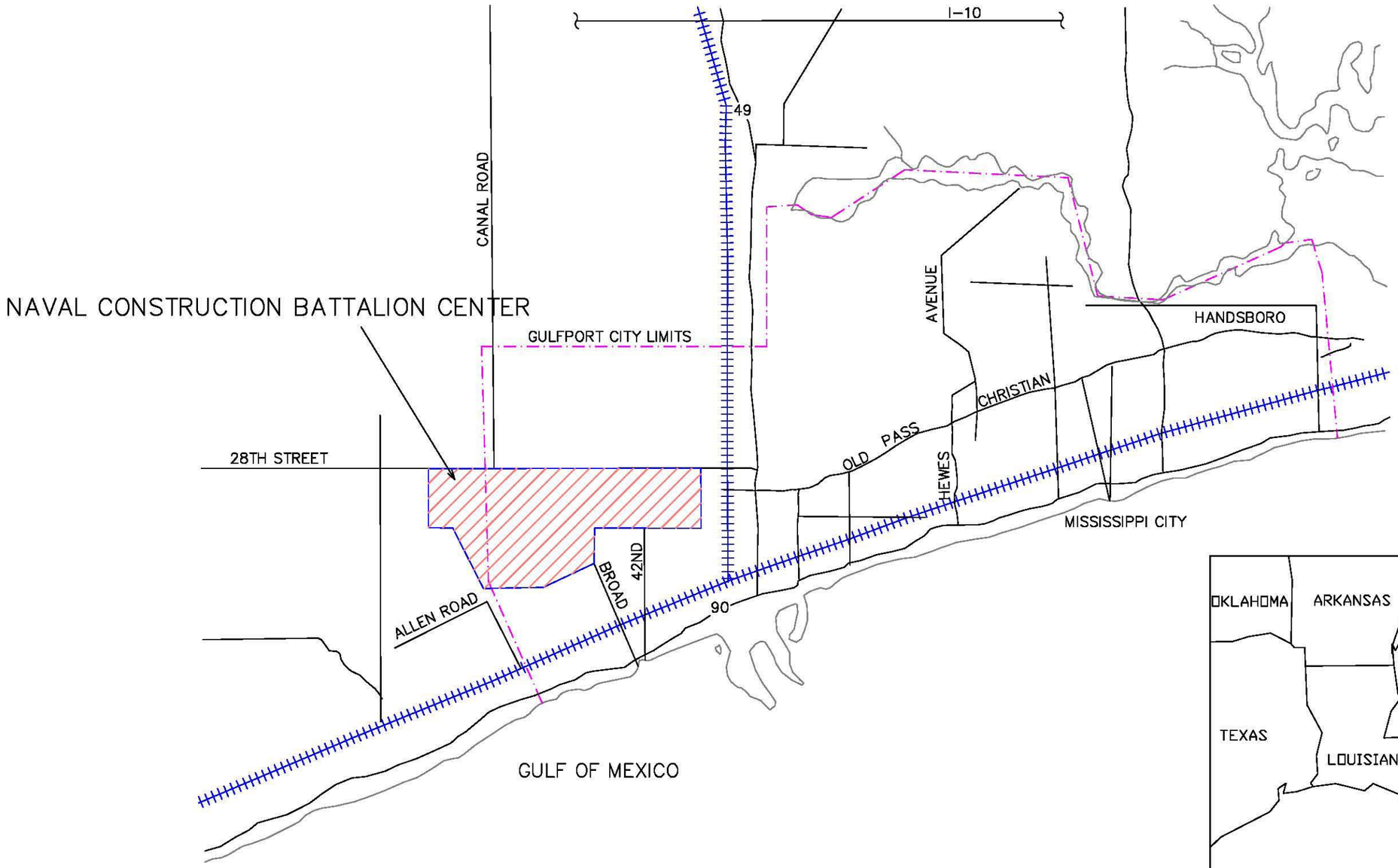
Notes:
^{o}C = degrees Celcius
TCLP = Toxicity Characteristic Leaching Procedure

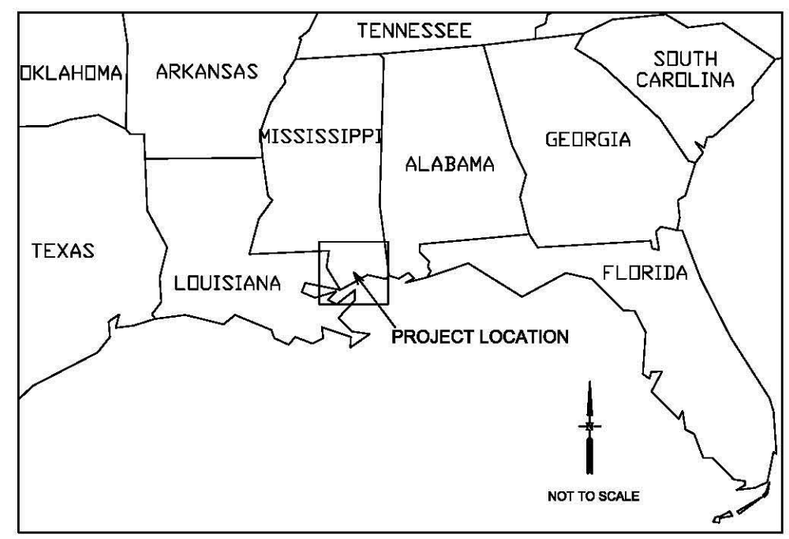
Table 4-3
Quantitation Limits and Remediation Goals
Site 8 – Herbicide Orange Storage Area and Off-Base Area of Contamination
Naval Construction Battalion Center
Gulfport, Mississippi

Matrix	Parameters	Analytical Methods	Reporting Limits	Remediation Goals: Maximum Allowable Concentration
Soil	2,3,7,8-tetrachlorodibenzo-p-dioxin	SPLP EPA 1312 SW 846 8290	0.001 ug/kg	< 0.038 ug/kg
	Total Petroleum Hydrocarbons (TPH)	SW 846 418.1	TBD	< 50 ppm
	Benzene, Toluene, Ethylbenzene, Xylenes (BTEX)	SW 846 8020	Benzene – TBD Toluene – TBD Ethylbenzene – TBD Xylenes – TBD	< 1 ppm
	Waste Characterization	TCLP EPA 1311 RCRA Hazardous Characteristics SW 846 7.3.3.2	NA	Shall not fail

Notes: mg/kg – milligrams per kilogram
mg/L – milligrams per liter
ppm – parts per million
TBD – To be determined upon final selection of the laboratory

FIGURES

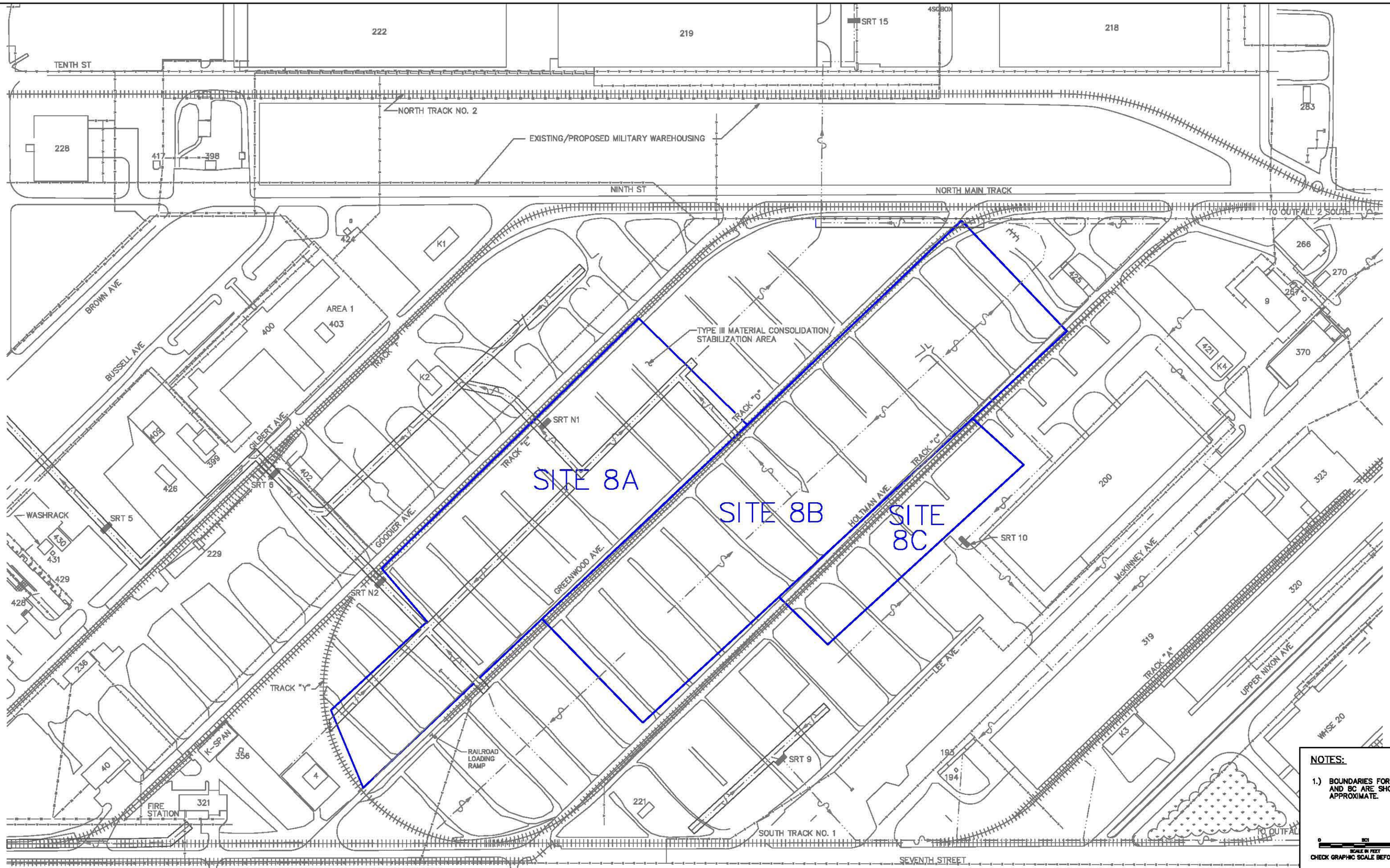




Environmental Chemical Corporation

FIGURE 1-1

SITE LOCATION MAP
NAVAL CONSTRUCTION
BATTALION CENTER
GULFPORT, MISSISSIPPI



DRAWN BY DM	DATE 8/05/04	FIGURE 1–2 – SITE LAYOUT PLAN SITES 8A, 8B, AND 8C HERBICIDE ORANGE STORAGE AREA NAVAL CONSTRUCTION BATTALION CENTER GULFPORT, MISSISSIPPI	CONTRACT NO.
CHECKED BY	DATE		NAVFAC NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE AS SHOWN			DRAWING NO. / REV. 0

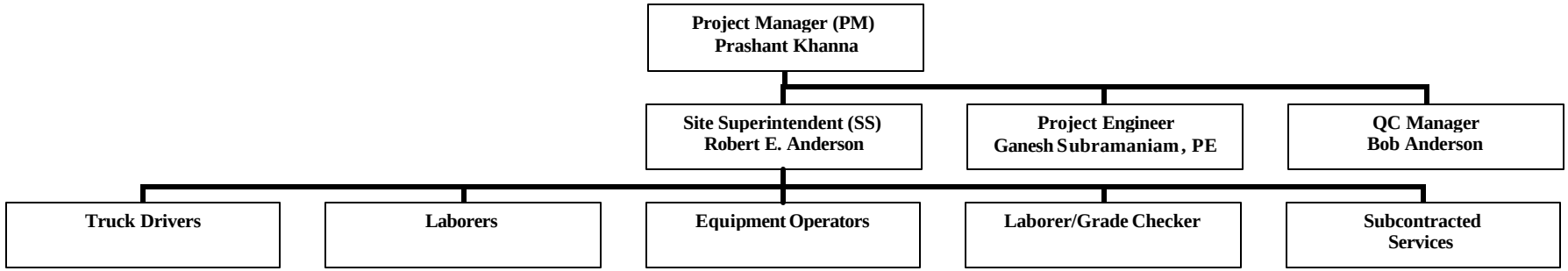


Figure 2-1
ECC Organization Chart

APPENDIX A

SOP E-302
EQUIPMENT DECONTAMINATION

1.0 OBJECTIVE

The objective of this Standard Operating Procedure (SOP) is to establish safe, standardized methods for decontaminating equipment during field sampling operations.

2.0 BACKGROUND

An important aspect of quality control is the decontamination of field sampling equipment. It is imperative that sampling devices and equipment be carefully cleaned prior to collecting each sample, between sample collection, and before removal from the site, thereby avoiding cross-contamination. Cross-contamination results in samples which are not representative of the sampled matrix.

Equipment decontamination minimizes the risk of cross-contamination between individual samples and assists in the reduction of possible hazardous material migration to other areas within the site.

3.0 EQUIPMENT REQUIRED

- Plastic drop cloths;
- Scrub brushes in various sizes;
- Disposable dishwashing gloves;
- Potable water (avoid water obtained from the site unless it has been tested for the Constituents of Concern);
- Deionized (DI) water;
- Wash tubs and/or buckets; (one for detergent, one for potable water, and one for DI water);
- Poly squeeze bottles (wash bottles);
- Manually operated pressure sprayers (one for detergent, one for rinse);
- Aluminum foil;
- Plastic wrap;
- Phosphate-free laboratory grade detergent (Alconox or equivalent);
- Dilute hydrochloric or nitric acid;
- Steam cleaner or high-pressure water cleaner; and Gloves - latex or vinyl (powderless); and
- Personal Protective Equipment (PPE) (wear the PPE as prescribed in the Safety and Health Plan, when handling contaminated materials).

4.0 PROCEDURE

4.1 Decontamination of Sampling Equipment Items

All sampling equipment must be carefully cleaned and dried prior to the start of sampling and between each sample collected. The following decontamination sequence will be utilized prior to sampling for all sampling equipment:

- Wash and scrub with a solution of Alconox detergent in water;
- Triple rinse with tap water;
- Rinse with DI water;
- Air dry;
- Rinse with dilute hydrochloric or nitric acid;
- Triple rinse with water;
- Rinse with DI water;
- Air dry; and
- Wrap in plastic for protection.

Sampling equipment will be cleaned following the collection of each sample in the field to avoid cross-contamination. A minimum of three tubs will be used – Detergent tub, Potable water gross rinse, and DI final rinse. Decontamination personnel shall wear PPE in accordance with the Safety and Health Plan. The field decontamination sequence will include the following steps:

- Scrape and then clean with tap water and soap using a brush if necessary to remove particulate matter and surface films. Equipment may be steam cleaned (detergent and high-pressure hot water) as an alternative to brushing. PVC or plastic items should not be steam cleaned;
- Wash equipment thoroughly with a solution of Alconox detergent in water and scrub to remove any particulate matter or surface film;
- Rinse equipment thoroughly with tap water;
- Rinse equipment thoroughly with DI water;
- Allow to air dry; and
- Wrap item in plastic for protection.

After the equipment is decontaminated between sampling episodes, it will be kept on clean plastic sheeting until needed.

When sampling equipment is used to collect samples containing oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane, or petroleum ether to remove the materials before proceeding with the first step. In extreme cases, it may also be necessary to steam clean the field equipment before proceeding with initial decontamination. If the equipment cannot be cleaned utilizing these procedures, it should be discarded.

4.2 Decontamination of 55 Gallon Bulk Sample Drums

Drums and lids will be decontaminated according to the following procedure:

Wash and scrub the drums with an Alconox detergent solution utilizing a pressure sprayer;
Triple rinse with water;
Rinse with DI water; and
Air-dry in an inverted position.

After drying, the drums will be resealed and taken to the sampling site. The lids will be removed just prior to actual sampling. When the drum is full, the lip of the drum will be wiped clean with paper towels to ensure a good seal when the lid and closing ring are attached. The outside of the drum will be washed down to remove clinging contaminants.

SOP E-601
SAMPLE HANDLING AND CUSTODY REQUIREMENTS

1.0 OBJECTIVE

The objective of this Sample Handling and Custody Requirements Standard Operating Procedure (SOP) is to establish procedures for the proper handling and custody of field samples. Sample handling refers to how samples are labeled, packaged, shipped, and stored.

2.0 BACKGROUND

Custody refers to how the samples are tracked and kept in possession by authorized parties (i.e. sample collectors, laboratory sample custodians, etc.), and of how the transfer of sample possession is documented. Custodial possession is critical to show that samples have not been tampered with and remain representative of the site.

A sample is physical evidence collected from a site or facility. Sample handling and custody serves to maintain sample representativness (i.e. the degree to which samples reflect the matrix sampled), and serves as a means of tracking samples. Every sample will be given a unique field sample designation for identification purposes. Proper sample handling and custody maintains sample representatives by controlling and preserving samples to prevent sample degradation and preventing cross-contamination by proper packaging. Sample handling and Custody entails selection of proper sample container, sample preservation, shipping, sample custody, chain of custody, and sample packaging.

Due to the possible evidentiary nature of the samples collected during enforcement investigations, a stringent program of custody procedures will be utilized to assure that each sample is accounted for from the time of collection to completion of analysis. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, documentation in logbooks and a Chain-of-Custody Record will be employed.

3.0 SAMPLE IDENTIFICATION

The method of sample identification used depends on the type of sample collected. Samples collected for specific field analyses or measurement data will be recorded directly in bound field logbooks and/or recorded directly on the Chain-of-Custody Record, with identifying information, while in the custody of the samplers.

All sample identification, chain-of-custody records, receipts for sample forms, and field records will be recorded with waterproof, non-erasable ink. If errors are made in any of these documents, corrections will be made by crossing a single line through the error and entering the correct information. All corrections will be initialed and dated. If possible, all corrections will be made by the individual making the error. If information is entered onto sample labels, logbooks, or

sample containers using stick-on labels, the labels will not be capable of being removed without leaving obvious indications of the attempt. L abels will never be placed over previously recorded information. Corrections to information recorded on stick-on labels will be made as stated above.

Sampling locations will be numbered sequentially and a sketch provided showing sample numbers and their corresponding locations. Every sample will be given a unique sample designation for identification purposes. The numbering system will be coordinated with the on-site representative to ensure that the proposed sample identifiers are discrete. The sample identification will be a series of letters and numbers consisting of three or four character strings, identifying the specific sample as follows:

PPPP-hhhh-xaaa

X PPPP is the 4 digit project code number (5 37 9)
X " hhhh" is the unique identification assigned to each sampling location:

EA = Excavation Area
DC = Drum Contents
DS = Debris Sampled
IF = Imported F ill
PT= Post Treatment
MS = Metals contaminated soil
F T = Water samples from frac tanks
DT = Demonstration Testing
OT = Operational Testing
IW = Incineration Waste
UW = Upwind (air monitoring)
DW = Down wind (air monitoring)
SC = School Z one (air monitoring)
WZ = Work Z one (air monitoring)
SW = Source Water
EF = L TTD Effluent
F D = F inal Decontamination
EW = Excavation Water
WW = Waste Water
IL = Inline Air Monitoring

X " x" describes the sample matrix:
1 = soil
2 = water/liquid
3 = debris
4 = Air

X " aaa" represents the sample number

F or example: Sample No.5 37 9 -EA01-1001 indicates that the sample is:

X F or Project No. 5 37 9 ;
X Collected at the IL SS, excavation area 01; and

X Soil sample 1.

All logbooks, sample labels/tags, custody seals, representative sampling documents, and COC documents will be completed using these sample designations. Certified, commercially clean sample containers shall be used for submitting samples for analysis. Sample jars or bottles should have a certificate of analysis from the manufacturer the bottles or jars, which shows the bottles or jars are contaminant free. Bottles or jars with preservatives added at the laboratory may lack these certificates. Check with the laboratory to determine that preservatives were added to certified commercially clean containers. Clean containers ensure samples are not cross-contaminated. The bottle or jar with the proper septum should be specified in the Field Sampling Plan.

4.0 SAMPLE PRESERVATION

Sample preservation ensures the samples are representative of the matrix sampled, by preventing sample degradation, volatilization, or precipitation. Samples are preserved by addition of chemical preservatives to the sample and/or by cooling the samples to 4 degrees Centigrade immediately upon sample collection. Typically, chemical preservatives are added to the sample bottle by the contract laboratory before the bottles are shipped to the site, and the bottles should indicate the added preservative. Generally, preservatives are not added to sample containers used for solid samples. Reference the Field Sampling Plan to ensure the correct preservatives were added to the bottles by the laboratory for the types of analyses needed. Contact the laboratory for more containers if the containers are not preserved or if preservatives must be added in the field. Check the preservative levels in the bottles least once during the beginning of the project. Determine if the preservative pH is within required limits by sample preservation as specified in the Field Sampling Plan. Some sample matrices may require additional preservative. If the pH is not in the proper range notify the contract laboratory.

5.0 SAMPLE LABELS

Sample labels are used to identify samples collected for laboratory analysis. A sample label is securely attached to each sample container. Some labels may not adhere to chilled sample bottles. Clear tape may be used to secure the labels in place. In some cases, particularly with biological samples, the sample labels may have to be included with or wrapped around the samples. The sample labels are sequentially numbered and are accountable documents after they are completed and attached to a sample or other physical evidence. The following information, at a minimum, will be included on the sample label using waterproof, non-erasable ink:

- Project number;
- Unique sample number and sample location;
- Date and time of sample collection;
- Sample description and location;
- Designation of the sample as a grab or composite;
- The signature of either the sampler(s) or the designated sampling team leader and the field

sample custodian (if appropriate);
- Whether the sample is preserved or unpreserved; and
- The general types of analyses to be performed.

6.0 CUSTODY SEAL

After collection, separation, identification, and preservation, the sample will be maintained under chain-of-custody procedures. Due to the evidentiary nature of the samples collected, possession must be traceable from the time the samples are collected until valid analytical results are obtained. The custody seal will be attached to the outside of each sample container in such a manner that it is necessary to break it in order to open the container. The following information, at a minimum, will be written on the custody seal:

- Date; and
- Signature of sampler(s).

7.0 SAMPLE CUSTODY

A sample is considered under custody if any one of the following conditions is met:

- It is in the actual possession of an investigator;
- It is in the view of an investigator, after being in their physical possession;
- It was in the physical possession of an investigator and then they secured it to prevent tampering; and/or
- It is placed in a designated secure area with limited or controlled access.

8.0 CHAIN-OF-CUSTODY RECORD

All sample shipments to on-site or off-site laboratories will be accompanied by the Chain-of-Custody (COC) record identifying its contents. The original custody record will be shipped along with the samples, while the person who initiates the record (the originator) will retain a copy. Chain-of-Custody procedures are comprised of the following elements: 1) maintaining sample custody and 2) documentation of samples for evidence. The Chain-of-Custody record ensures that samples are traced from the time of field collection until valid analytical results have been obtained.

When responsibility of a group of samples changes several times, each custodian is not required to retain a copy of the custody record, as long as the original custody record indicates that each person accepting the samples has subsequently relinquished custody appropriately. Chain-of-Custody forms will be completed according to the following protocol:

- The originator will fill in all requested information from the sample labels;

- The originator will sign and date the " Relinquished by" box and keep the copy;
- The F ederal Express Tracking number or other reference shipping number, if any, will be included in the sample shipment documents;
- The original record sheet will be shipped with the samples. A plastic shipping envelope will be taped to the inside of the cooler top and the remaining two copies of the chain-of-custody will be inserted;
- The person receiving custody will check the sample label information and the custody record, and will also check sample condition and note unusual observations under " Remarks" on the custody form;
- The person receiving custody will sign in the adjacent " Received by" box and keep the original;
- The Date/Time will be the same for both signatures, since custody must be transferred between two individuals. However, when samples are shipped via common carrier (e.g.,), the date/time will not be the same for both signatures;
- When samples are shipped via common carrier, the original custody form will be shipped with the samples and the shipper (e.g., F ield Sample Custodian) will keep the copy. The shipper will also keep all shipping paper, bills of lading, etc.;
- In all cases, it must be readily apparent on the custody form that the person releasing custody has relinquished it to the next custodian; and
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the custody record.

8.0 REPRESENTATIVE SAMPLE DOCUMENT

A representative sample document will be completed for each sample collected. The representative sample document will record the complete history of each sample, including the sample source, date collected, sample methodology, sample size and container, sample number, and the analysis required. This document will be signed by the sampler and a witness, and will remain with the project records.

9.0 SAMPLE HANDLING AND SHIPPING

Place samples into appropriately pre-labeled and preserved sample bottles or jars and immediately store the samples on ice at 4 degrees Centigrade in an insulated cooler. Keep the cooler under observation and in a shaded area and maintain custody over the samples. Document the samples that are collected in the field logbook.

Wipe excess soil or water off of the sample bottles or jars before storing or shipping the samples. Wear appropriate PPE as described in the Health and Safety Plan while handling samples.

Prior to shipping the samples, prepare the COC documentation. Ensure the COC and sample label information agree and that the COC accurately indicates all of the samples in the cooler. Place the samples in Z iploc storage bags and wrap in bubble wrap. Place the completed COC in a plastic bag and tape the COC to the inside top of the cooler used for shipping. Keep a copy of

the COC in the field notebook.

Prior to placing samples in the cooler, place 3 inches of packing material (waterproof Styrofoam " peanuts") on the bottom of the cooler. Place samples upright into the cooler, and do not let the samples touch each other. Place ice (double-bagged in Z iploc bags) around the samples and on top of the samples. F ill in voids with packing material, but do not overpack too tightly. Tape shut the cooler drain port. Use strapping tape to secure the cooler lid. Place two custody seals on the cooler – one on the front and one on the back, as described before. Ensure samples reach the analytical laboratory and are analyzed well before the holding time expires.

Notify the laboratory of sample shipments on the weekend to ensure a sample custodian is present to receive the samples, and notify the laboratory if the number of samples per day or type of analysis is greater than that specified in the laboratory contract.

If required in the F ield Sampling Plan or if sent by the laboratory, add a temperature blank to the sample cooler. The laboratory measures the temperature of the temperature blank to determine the temperature of the samples upon receipt at the laboratory.

SOP E-603
FIELD DOCUMENTATION

1.0 PURPOSE

The purpose of the Field Documentation Standard Operating Procedure (SOP) is to describe the procedures for maintaining sample control through proper sample documentation. When samples are collected for chemical or physical characteristic analysis, proper documentation needs to be completed. The information presented in this SOP enables maintenance of sample integrity from time of collection through transportation and storage. It is this documentation that will verify that the samples were properly handled.

1.1 Documentation

The following discussion outlines standard practices and procedures to be used when documenting a sampling episode. All project-specific documentation requirements must be presented in the Sampling and Analysis Plan (SAP). This includes identification of procedures required for field documentation, sample labeling, and the maintenance of chain-of-custody (COC). For United States Corps of Engineer's (USACE) projects, the Contractor is required to obtain a tracking number (e.g., Laboratory Information Management System (LIMS) number) to be used in conjunction with the government quality assurance (QA) sample shipments. The tracking number should be specified in the SAP. Proper completion of the logbook and supporting paperwork with indelible ink is necessary to support potential enforcement actions that may result from the sample analysis. Therefore, maintaining sample integrity through proper documentation is essential.

1.1.1 Field Logbooks

A field logbook will be used to record data collection activities performed at the site. Each logbook will be bound and contain consecutively numbered, water-resistant pages. The first page of each logbook will contain the project number. All entries will be in indelible ink. Notations should be made in logbook fashion, noting the time and date of all entries. All corrections will be made using lineout deletions and will be initialed by the person making the correction. At the end of each field day, the end of the last page used will be initialed and dated by the author(s), and a line will be drawn through the remainder of the page. The cover of each logbook will contain the project name and number, log start date, and log end date. Entries to the logbook will be sufficient to recreate the activities of the day and must include at least the following information:

- Name and title of author;
- Name(s) and title(s) of the field crew;
- Date and time the field work started;
- Location and description of the work area;
- Field instrumentation and equipment used and purpose of use (i.e., health & safety screening, sample selection for laboratory analysis), calibration methods used, field results, and quality control (QC) information;

- Meteorological conditions at the initiation of fieldwork and changes during the sampling event; and
- Personal and equipment decontamination activities.

F or each sample collected, the following information must be entered into the logbook:

- Type of sample;
- Date and time of sample collection;
- Sample collection or measurement method;
- Sequential number and volume of sample taken;
- L ocation and description of sampling point;
- Sample identification number;
- Sample containers (size/type) and preservatives used in sample;
- Sample manipulations (i.e., filtration, compositing, etc.);
- Destination of samples after collection;
- Description of the number of shipping coolers packaged with corresponding COC numbers and shipping method employed (noted applicable tracking numbers);
- Name and address of all receiving laboratories (include L IMS number for QA laboratory);
- Reference entries for all maps and photographs of sampling site;
- F ield observations/comments;
- F ield measurements taken (pH, temperature, etc.);
- Serial numbers for field instruments;
- Time and date of last calibration of field instruments; and
- Any deviations from the SAP.

No field logbooks will be destroyed or thrown away, even if illegible or containing inaccuracies requiring a replacement document. Replacement documents will be so noted and referred to the original.

2.0 DOCUMENTING SAMPLING POINTS

The exact locations of sampling points should be documented for purposes of generating an accurate representation of the site conditions using the data generated to date, defining data gaps, and identifying potential future data needs. This is accomplished through the use of a monument and compass. A monument should be chosen at each site to act as a stationary reference point from which all sampling points can be measured using a compass and measuring tape. If a building or other stationary structure exists, its corner may act as this reference point. If no monument exists, it will be necessary to create one using a piece of wood, approximately 2 inch by 2 inches, hammered into the ground almost to ground level. The stake should then be marked with flagging tape or fluorescent paint. When establishing a sampling point, the following procedure should be used:

- Standing at the monument, facing the sampling point, use the compass hairlines to determine the degree of direction;
- Ensure that the line of sight runs from the monument, through both hairline needles on the compass, to the sampling point; and

- When first establishing the sampling point, record in the field logbook the degree and compass directional reading, along with the distance measurement from the monument to the sampling point, sampling stake or surveyed pin flags.

3.0 PHOTOGRAPHS

All sampling points should be documented on film to allow for positive identification of the sampling points. Photographs are the most accurate and convenient records of field personnel observations. Photographs taken to document sampling points should include two or more reference points to facilitate relocating the point at a later date. Keeping a record of photographs taken is crucial to their validity as a representation of an existing situation. Color photographs will be taken of the field activities using a camera-lens system with a perspective similar to the naked eye; telephoto or wide-angle shots can not be used for enforcement purposes. Photocopies will be made of the photographs as required for reporting purposes. The following information concerning each photograph will be recorded in the field notebook:

- Photographer's name;
- Name of Site;
- Date and time of the photograph;
- General direction the camera was facing;
- Brief description of the subject and field work portrayed;
- Sequential number of the photograph as indicated on the camera counter;
- Site photo map; and
- The film roll number on which the photograph appears.

4.0 SAMPLE COLLECTION PAPERWORK

4.1 Sample Labels

Sample labels are required for the proper identification of the samples. All samples must be properly labeled with the label affixed to the container prior to transportation to the laboratory. It is also recommended that samples be photographed so the labels are clearly legible for later identification. Information on sample labels should include, but not be limited to, the following:

- Project Code;
- Sample Identification Number;
- Sampler's name and signature or initials;
- Preservatives used;
- Analysis requested;
- Date/Time of sample collection;
- Type of sample (discrete or composite); and
- Federal Express tracking number.

4.2 Sample Numbering System

A sample numbering system should be used to identify each sample collected and submitted for

analysis. The purpose of the numbering system is to assist in the tracking of samples and to facilitate retrieval of analytical results. The sample identification numbers for each sampling effort should be used on sample labels, sample tracking matrix forms, COC forms, field logbooks, and all other applicable documentation. A listing of all sample identification numbers should be recorded in the field logbook. The sample numbering system may vary depending on the number and type of samples that will be collected at the site.

Each sample collected must be assigned a unique sample number. Sample numbers should change when the media or location changes. However, sample numbers should not change because different types of analysis are requested.

4.3 Chain-of-Custody Record

All sample shipments will be accompanied by the chain-of-custody record (Appendix B) identifying its contents. This record will be used to document sample custody transfer from the sampler, to other sampling team members (if necessary), to the courier, and finally to the analytical laboratory. The chain-of-custody record ensures that samples can be traced from the time of field collection until they are received and analyzed by the analytical laboratory. The original custody record is shipped along with the samples, while the initiator of the record retains a copy. The information required for the chain-of-custody record includes:

- Type of sample (grab or composite) and matrix;
- Analytical method numbers and parameter names;
- Sample number;
- Signature of sampler;
- Date and time of sample collection;
- Project name, location and address; and
- Signatures of persons involved in the chain of possession.

When responsibility for a group of samples changes several times, each custodian is not required to retain a copy of the chain-of-custody record, as long as the original custody record indicates that each person accepting the samples has subsequently relinquished custody appropriately. chain-of-custody forms will be completed according to the following protocol:

- The originator fills in all requested information from the sample labels;
- The originator signs the " Relinquished by" box and keeps the copy;
- The original record sheet is shipped with the samples. A plastic shipping envelope will be taped to the inside of the cooler top and the remaining two copies of the chain-of-custody record will be filed with the representative sampling documents;
- The person receiving custody checks the sample label information against the custody record. He/she also checks sample condition and notes anything unusual under " Remarks" on the custody form;
- The person receiving custody signs in the adjacent " Received by" box and keeps
- the original;
- The Date/Time will be the same for both signatures, since custody must be transferred between two individuals. However, when samples are shipped via common carrier (e.g.,

F ederal Express), the date/time will not be the same for both signatures;

- When samples are shipped via common carrier, the original custody form is shipped with the samples and the shipper (e.g. F ield Sample Custodian) keeps the copy. The shipper also keeps all shipping paper, bills of lading, etc.;
- In all cases, it must be readily seen that the person receiving custody has relinquished it to the next custodian; and
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the chain-of-custody record.

In addition to the COC record, a COC seal is used to ensure that no sample tampering occurred between the field and the laboratory analysis. The COC seal is an adhesive seal placed in areas such that if a sealed container were opened, the seal would be broken.

SOP E-604
DATA MANAGEMENT

1.0 PURPOSE

The purpose of the Data Management Standard Operating Procedure (SOP) is to describe steps that will be used at Environmental Chemical Corporation (ECC) project sites to ensure field and laboratory data is recorded, verified, and filed.

2.0 BACKGROUND

This SOP applies to data generated during any field-related sampling events performed at an ECC project site and encompasses the data handling process from the point of collecting, filing to data transmission. This SOP describes the field data collection, data entry, data verification, data archiving, and filing that will be implemented to attain acceptable standards of accuracy, precision, representativeness, comparability, and completeness. In addition, this SOP describes laboratory data entry, data verification, data archiving, and filing.

3.0 RESPONSIBILITIES

Project Manager: The Project Manager (PM) will be responsible for ensuring the implementation of this SOP. The PM will select the project personnel to implement this SOP and will ensure that the subcontractor personnel follow the SOP procedures. In addition, the PM will provide general oversight of the project data management according to the SOP required procedures.

Project Engineer: The Project Engineer (PE) will be responsible for:

- Oversight of the field data collection to ensure that proper data management procedures are followed in accordance with this SOP;
- Preparation and maintenance of the project database; and
- Selection of personnel to verify the project data.

4.0 PROCEDURES

The following procedures will be used to facilitate project field and laboratory data management.

4.1 Field Data Collection

All data collected from the field will be recorded in the PE's project logbook (SOP E-603) or a separate sample-tracking logbook. To the extent possible, the format of the logbook data entry will coincide with the electronic form in the project database. This will assist the PE in entering data into the

database efficiently and accurately.

4.1.1 Data Completeness Check

At the end of each day, the field data generated will be checked for completeness by determining if all the data required was produced for each field sample or test. The data completeness check will initiate the data verification process by providing an initial review of the data. Preliminary verification of the field data will be conducted at the end of each sampling event. This task ensures that the data meets the completeness criteria before being entered into the database. If the data is not complete, the PE will confer with the PM before deciding how to meet the completeness criteria.

4.1.2 Technical Data Verification

After the data completeness is verified, a technical verification will be performed on the data to ensure that the data is consistent with known chemical and physical properties of the media sampled. All calculations and reported units will be checked; if an error is detected, the PE will confer with the PM prior to changing any information. Any change made must be noted in both the PE and the PM's logbook.

4.1.3 Data Entry

After verification of the data completeness, the data and its field tracking information will be placed into the project database. The field tracking information is documented on the chain of custody (COC) form and includes:

- Sample Number
- Sample Location
- Date Sampled
- Parameter
- Date Shipped
- Laboratory
- Date Results Received

4.2 Data Verification

Data verification ensures that the electronic database and the data originally recorded is the same.

4.2.1 Field Data

After the field data is entered into the database (MS Access database is recommended), a hard copy will be printed from the database and evaluated against the logbook entries. If possible, the data verifier will not be the person who originally entered the data. The printed database will be compared for

accuracy. If the verifier finds transcription errors, the errors will be highlighted on the printed report and returned to the person who entered the data for correction. The errors will be corrected and a new copy of the data will be generated. This process will be repeated until the printed report matches the data from the field logbook. Both the verifier and the PE will sign the acceptable printout of the final data report prior to filing.

4.2.2 Tracking Data

The sample tracking information (Section 4.1.3) also will be entered into the project database. After the tracking information is entered, it will be printed and verified as described in Section 4.2.1.

If the PE is responsible for the functions of data entry, data verification, and data tracking, a weekly check of the logbook versus the electronic database (as described in Section 3.2.1) remains the required data verification procedure.

4.3 Laboratory Data Verification

Verification of the project laboratory data will consist of an evaluation of completeness, correctness, consistency, and compliance of the laboratory data package against the contract requirements. The Project Engineer or another competent, designated person will perform the data verification upon receipt of the laboratory data package and prior to entering the laboratory results into the project database.

After the laboratory results are entered into the database, a hard copy will be printed from the database and verified as described in Section 4.2.1.

4.4 Data Archiving and Filing

Upon completion of data verification for either field data or laboratory data, the hard copy of the final data reports will be signed by both the verifier and the PE and filed in the project files. A copy of the printed data information will be presented to the client if requested. Two copies of the electronic files of the database information will be backed up on diskettes each week and appropriately labeled with the contractor company name, the date, and the type of data contained (i.e., well data, soil boring data, etc.). One diskette will be delivered to the client and one diskette will be archived with the project files.

SOP E-605
DATA ASSESSMENT

1.0 PURPOSE

The purpose of the Data Assessment Standard Operating Procedure (SOP) is to describe procedures for ensuring that project data meet the project Data Quality Objectives (DQO) outlined in the Sampling and Analysis Plan (SAP). This SOP specifically addresses data assessment of the following:

- Holding Times;
- Method Blanks;
- Field Blanks;
- Trip-Blanks;
- Laboratory Control Samples (LCS);
- Surrogate Recovery;
- Matrix Spike/Matrix Spike Duplicates (MS/MSD);
- Field Duplicates; and
- Continuing Calibration Verification.

2.0 BACKGROUND

Project objects are developed during the project's DQO process, which encompasses sample locations, sample depths, error confidence limits, method of collection, constituents of concern, analytical methods, applicable or relevant and appropriate requirements (ARARs), and determining project decision-making levels. All of these concerns culminate in the development of the project DQOs. Understanding the DQO process is critical in evaluating the quality of the chemical data.

Laboratory specific elements of project DQOs are expressed as Method Quality Objectives (MQO) or Quality Control (QC) acceptance criteria. Data Quality Indicators (DQI) are the analytical results of QC samples or spikes and are compared to the MQOs as part of the data assessment process. Other DQIs are obtained from field QC sample results. Correct evaluation of the project data and the assessment of its usefulness is a critical tool in determining the success of the project.

Project data must be of sufficient quality to support the project decisions that are based on the chemical data. Assessing data quality and communicating the data quality to the client or regulatory authority is necessary to build confidence in data based project decisions. The data quality process starts with the development of the project work plans, determination of decision-making levels, selection of a laboratory, representative sample collection strategies, and assessment and presentation of quality data. Data assessment also is performed to ensure contractual compliance by the laboratory.

laboratory; analytical batches with marginal data quality may be released. As a general rule, the more data that is qualified or flagged on the LCS, MS/MSD, and surrogate recoveries singularly or in conjunction, the greater the need for a more in depth review of the data by a chemist.

Data assessment is not the same in-depth evaluation of chemical data as data validation. Either a chemist or other personnel with knowledge of the data validation process and requirements should evaluate any discrepancies uncovered in a data assessment.

4.1 Data Assessment Report Introduction

Review the project's DQOs and MQOs. If the laboratory QC acceptance parameters differ from the project MQOs, use the project MQOs to review the analytical data. If the laboratory uses Reporting Limits (RL) which differ from the projects RLs or Action Limits, obtain a copy of the project RLs.

Prepare an introduction for the Data Assessment Report describing the sample locations, depths, and sample rationale. The data assessment will discuss the Precision, Accuracy, Representativeness, Comparability, Completeness, and Sensitivity (PARCCS parameters). The PARCCS parameters describe the required analytical precision, analytical accuracy, representativeness of samples, comparability to past and future analysis, completeness of sampling, and analytical sensitivity required to ensure that the MQOs fulfill the project DQOs.

4.2 Preparation and Analytical Method

Review the extraction and analytical methods to ensure they agree with the project Field Sampling Plan (FSP) portion of the SAP. Prepare a table for the DATA ASSESSMENT REPORT presenting the extraction method number and analytical method number for each analyte. Present any other method deviations or changes listed in the laboratory narrative.

4.3 Analytical Sensitivity

Compare the laboratory Method Detection Limit (MDL) and Method Quantitation Limit (MQL) to the project decision making levels or action levels. Generally, this comparison is performed during the laboratory selection process, but for major constituents of concern perform the comparison. For analytes of interests, confirm that the project's MQL is at least 3X the MDL. For some projects, the laboratory reports a Method Reporting Limit (MRL). A MRL is an arbitrary limit that is not based upon the MDL or a multiplier of the standard of deviation. If an MRL is reported, the data must be evaluated based upon comparison to the MDL and MQL.

The Data Assessment Report should discuss the following information concerning the analytical sensitivity:

- Whether the analytical sensitivity was adequate for all analyses and present or reference a data table showing a comparison between the project action levels and

the laboratory MDL and MQL to demonstrate the laboratory's quantitative effectiveness;
- Whether the project decision-making levels are below the laboratory's MQL;
- Whether the laboratory was able to quantitatively detect analytes at levels below the project decision making levels; and
- What analytes lacked adequate sensitivity and the impact on data quality?

4.4 Sample Chain of Custody

Review the sample Chain of Custody (COC) copies returned by the laboratory to determine how the laboratory received the samples. Note any discrepancies in sample packing, containers, preservatives, broken custody seals or signs of tampering, broken sample bottles or jars, and the sample temperature.

Evaluation Criteria

The evaluation criteria should include a report of any discrepancies in the condition of the samples when they are received. The following sample conditions should be noted:

- The temperature of the samples when received at the laboratory;
- Whether the samples were properly preserved;
- Whether the custody of the samples were maintained; and
- Whether the laboratory narrative addressed the major discrepancies.

The following types of sample conditions may require sample recollection:

- Samples received by the laboratory at an elevated temperature;
- Samples received with broken custody seals; and
- Broken sample containers.

If the samples received at an elevated temperature are analyzed, the data may be qualified as estimated. Since broken sample containers may cause cross-contamination, the Method Blanks and other sample blanks should be closely evaluated.

4.5 Holding Times

Sample holding times are the maximum time that a sample or sample extract may be kept in storage and still meet method guidelines. The holding time starts from the date of sample collection. Information to evaluate holding time compliance can be found on the COC (time of sample collection) or on the analytical data sheet that provides the date of sample collection, date of extraction, and date of analysis. Check several of the sample data sheets versus the information on sample collection dates on the COC and the field logbooks. Check the length of time the samples were held on-site, if the samples were preserved during on-site storage, and the time of sample transport to the laboratory.

Evaluation Criteria

Samples exceeding holding times must be rejected (R) or qualified as estimated (J). If sample holding times are not provided in the DQOs, the default sample holding times presented in the following table will be used:

Default Sample Holding Times

Analyte	Liquid	Solid
VOC	14 days	14 days
SVOCs	7 days / 4 0 days (extract)	14 days / 4 0 days (extract)
Metals [except Cr(VI), Hg, F e(II)]	6 months	6 months
Explosives	7 days / 4 0 days (extract)	14 days / 4 0 days (extract)
Total Petroleum Hydrocarbons	7 days / 4 0 days (extract)	14 days / 4 0 days (extract)
PCB/Pesticides/Herbicides	7 days / 4 0 days (extract)	14 days / 4 0 days (extract)

F or a Data Assessment Report, provide a short narrative stating that the samples were analyzed within the holding times and describe any samples analyzed outside of holding times.

4.6 Field Quality Control

All field sampling events require the collection of additional samples to provide quality control (QC) for the field and laboratory procedures. F ield QC samples determine the precision and accuracy of samples by checking the quality of the field sample collection methods and the possibility of cross-contamination during sample collection.

F ield QC samples are analyzed by the laboratory; however, the laboratory may not use these results to assign data qualifiers to other samples in the sample batch or sample cooler.

4 .6 .1 Equipment Rinsates, F ield B lanks, and Tripblanks

Equipment (Rinsate) B lanks, F ield (B ottle) B lanks, and Trip B lanks check for potential cross-contamination during the sampling program. Contaminated equipment rinsates may indicate improper equipment decontamination or contamination in the source water.

Evaluation Criteria

The quality of the F ield QC sample is initially determined by examining the surrogate recoveries and the overall analytical sample batch quality. If the surrogate recoveries are adequate and the method is in control, the results of the F ield QC will be used to determine cross-contamination in the samples associated with the data batch and the sample cooler by

- Determining the analytes and levels of contaminants found in F ield B lanks and Tripblanks; and

- Determining if similar analytes are found in samples of that same data batch and in sample shipped in the same sample cooler.

Analyte levels found in environmental samples at levels less than 5 X or 10X greater (for common laboratory contaminants) than those found in the Field Blanks or Tripblanks should be reported as Non-Detect (U) or Non-Detect Estimated (UJ).

Equipment Blanks indicate the effectiveness of equipment decontamination by

- Determining the analytes and levels of contaminants found in the Equipment Blanks; and
- Determining if similar analytes are found in samples obtained from equipment decontaminated during the same day or at the same decontamination station.

Analyte levels found in environmental samples at levels less than 5 X or 10X greater (for common laboratory contaminants) than those found in the Equipment Blanks should be reported as Non-Detect (U) or Non-Detect Estimated (UJ). Blank concentration levels will not be subtracted from the sample levels.

The following Field QC areas should be examined:

- The rate of Field QC sample collection;
- Whether Trip Blanks were added to sample coolers containing aqueous VOC samples;
- Describe the Field Quality Control samples with contaminants;
- Describe any samples requiring new data qualifier assignments and the basis of the assignment (i.e. 5 X or 10X rule);
- Describe the contaminant level detected in the environmental samples;
- Determine the source of cross-contamination; and
- Examine the Method Blanks for similar contaminant levels;

If the Method Blank and Field Quality Control sample exhibit the same levels of contamination, the contamination may be attributable to the laboratory. If the Method Blanks yield no similar contaminants as the Field QC samples, the cross-contamination may be attributable to field or shipping conditions.

4.6.2 Field Duplicates

Field Duplicates are used to assess the precision of the field sampling effort through a review of the analytical results. Field Duplicate results are not a measure of laboratory precision or accuracy. Field Duplicates are sent "blind" to the laboratory, so the Project Engineer must compare the sample and Field Duplicate sample. The comparison is made by determining the Relative Percent Difference (RPD) between the sample and Field Duplicate by using the equation below:

$$RPD = (A - B) \times 100 / (A + B)/2 \quad \text{(take the absolute value)}$$

Where "A" is the level of an analyte in the sample and "B" is the level of an analyte in the Field Duplicate sample.

Evaluation Criteria

The field sampling effort can be evaluated through a review of the Field Duplicates as follows:

- Determine that at least one Field Duplicate was performed per sample batch;
- Determine the RPD of all detected analytes in between the sample and Field Duplicate; and
- Compare the Field Duplicate RPD with that provided in the Field Sampling Plan or use a default value of 20% for liquid samples and 40% for solid samples.

For a Data Assessment Report, present the sample, Field Duplicate, and RPD results for all detected analytes in tabular format. Provide an assessment of the field sampling precision based upon the number of duplicates failing RPD acceptance parameters.

Reasons for failing RPD acceptance parameters may be the sample heterogeneity, especially for solid samples; insufficient mixing of solids in the field; and collecting co-located but not homogenized samples. Liquid samples are typically not mixed, but co-located.

For analysis near the lower limits of quantitation (i.e. near the MQL) the possible statistical error in analysis increases. Caution should be exercised in evaluating estimated (J) data and any low-level data near the MQL.

The frequency of Field Duplicate collection, the number of sample / Field Duplicate RPD failures, and the degree of magnitude of any failures should be discussed in the Data Assessment Report. In addition, provide any suggestions for corrective action to increase field sampling precision.

4.7 Laboratory Quality Control

Laboratory Quality Control samples indicate the precision and accuracy of the analytical method by determining the following:

- Whether the samples were cross contaminated in the laboratory;
- Whether the instrument performance is stable and in control; and
- Whether extractions were performed within MQOs.

Laboratory Quality Control samples are used to evaluate the QC of the analytical batch (i.e. about 20 associated samples) and determine the quality and the data qualifiers assigned to all analytes in the sample batch. Therefore:

- The Laboratory Quality Control samples should be analyzed in conjunction with the associated environmental samples;

- The data qualifiers applied to Laboratory Quality Control samples should be applied to associated environmental samples.
- Any Laboratory Quality Control sample performed as part of a sample batch should be evaluated. The laboratory practice of running multiple Laboratory Quality Control samples and choosing the one that is in control is not acceptable.

4.7.1 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)

The LCS measures the accuracy of the analytical method by measuring the recovery of spiked compounds. The LCS is an interference free matrix spiked with analytes of interest or with compounds prescribed by the method. If performed, the LCSD is used to determine the precision of the laboratory method by determining the RPD between the LCS and the LCSD. The LCS and LCSD data should be presented on a data sheet from the laboratory.

<u>Evaluation Criteria</u>

To evaluate the effectiveness of the LCS

- Determine that at least one LCS was performed per sample data batch;
- Determine that the LCS compounds agree with those in the Field Sampling Plan or the MQOs;
- Determine if the Recovery (%R, expressed as a percentage of the spike level) meets the MQOs. Generally, the %R range is between 50% – 120%.

If the %R range exceeds 120%, call the laboratory for an explanation of the range. A LCS acceptance range that is too large may mask poor laboratory performance and method accuracy.

If the LCS recovery is out of the MQO acceptance range, sample data with the same specific spike compounds must be qualified as estimated (J) data. If more than 1/2 of the LCS compounds exceed %R MQOs, than all of the analytes must be qualified as estimated (J). LCS %R failures may require re-extraction and batch analysis, if the laboratory narrative doesn't provide an adequate explanation. Although some LCS failures may be due to poor LCS injection or degraded spike solutions, there must be some explanation about the laboratory performance to warrant not re-running the sample batch. For sensitive sites, re-running the sample batch will avoid quality issues. If the number of LCS %R failures is low (i.e. one or two depending upon the number of spiked compounds) and the MQO exceedence is low, then the sample batch data may still be used with appropriate qualification.

If an LCSD sample was analyzed, the RPD between the LCS and the LCSD should be compared to the project MQOs or at least 30% or less. Recovery failures in an LCSD performed as part of the sample batch must be given the same weight as the LCS. If the LCS/LCSD RPD is provided, it must be used to evaluate the laboratory precision. LCS/LCSD RPD exceedence will require that the sample batch be re-extracted and re-analyzed, because of poor performance.

Include the following information in the Data Assessment Report:

- The frequency of LCS and LCSD (if performed) analysis;
- Which LCS and/or LCSD samples and which spike compound(s) failed the %R MQO; and
- Which samples and analytes were qualified on the basis of the LCS %R failure?

The laboratory narrative accompanying the data set may help explain the %R failures and the impact on the data quality. Generally, a %R failure for compounds not detected in the samples will have minimal impact. LCS spike compounds %R failures may have a high or low bias and must be stated as such.

4.7.2 Matrix Spike/ Matrix Spike Duplicate

Matrix Spike/ Matrix Spike Duplicate samples should be performed on the site matrix (i.e. site groundwater or site soils) to determine if the matrix interferes with the extraction or analysis. Matrix interferences are common with SVOC and TPH analysis, as compounds in this family are native to many sites. The recovery (%R) of the spiked compounds determines the accuracy of the laboratory in determining compounds in the site-specific matrix, and the RPD between the MS and MSD determines the laboratory performance for the matrix. The MS/MSD data should be presented on a data sheet from the laboratory.

<u>Evaluation Criteria</u>

To evaluate the MS./MSD perform the following checks:

- Determine that at least one MS/MSD was performed per sample data batch.
- Determine that the MS/MSD spike compounds agree with those in the Field Sampling Plan or the MQOs.
- Determine if the Recovery (%R, expressed as a percentage of the spike level) meets the MQOs.
- Determine the level of spike compounds relative to the native concentration. If the spike level is not 2X greater than the native concentration then data qualification based upon %R or RPD may not be warranted.

Generally the %R range is between 35 % – 130%. For some compounds in a soil matrix, the %R range may be greater. If the %R range exceeds 100%, call the laboratory for an explanation of the range. For accurate MS %R interpretation, the spike levels must be at least 2X greater than the levels of the spike compound that may already exist in the site matrix.

If the MS or MSD recovery is out of the MQO acceptance range, sample data with the same specific spike compounds must be qualified as estimated (J) data. If a subset of the target analytes are present in the %R failed matrix spike (i.e. same family of chemical), than all of the non-spiked compounds may be qualified as estimated (J) based upon professional judgment. Some MS failures may be due to poor spike injection or degraded spike solutions, but there must be some explanation about the laboratory performance. Poor spike recovery, assuming the LCS sample shows the method is in control, may indicate a low or high matrix bias. Contact the laboratory to determine if matrix cleanup needs to be performed to remove matrix interferences.

The MS and the MSD RPD should be compared to the project MQOs, and the RPD should be at least 4 0% or less. The MS/MSD RPD may be used to evaluate the laboratory precision if the L CS/L CSD pair is not analyzed. F or a spiked analyte, assuming no matrix interference or native analyte levels less than 2X the spike level, an RPD failure causes the analyte in the MS and all associated samples to be qualified as estimated (J).

The Data Assessment Report should include:

- The frequency of MS and MSD analysis;
- Which MS and/or MSD samples and which spike compound(s) failed the %R MQO; and
- Which samples and analytes were qualified on the basis of the MS %R failure?

The laboratory narrative accompanying the data set may help explain the %R failures and the impact on the data quality. MS spike compounds %R failures may have a high or low bias and may indicate a matrix interference or analytical variation. If the L CS is in control and the MS recoveries are out of MQO then a bias may exist.

F or the report, state for which samples an MS/ MSD RPD failure causes sample qualification as estimated (J).

4 .7 .3 Surrogates

Surrogates are compounds spiked to samples before the sample extraction is performed. Surrogates determine the extraction efficiency and matrix interferences. The Surrogate information should be presented on the same data sheet as the sample results.

<u>Evaluation Criteria</u>

To evaluate the surrogate information, determine that the proper surrogates were spiked into each sample. The surrogates may be specified in the DQOs or listed in the QAPP. Surrogates are typically isotopic or halogenated compounds not typically considered contaminants.

Surrogate recovery is expressed as %R. Typical surrogate recovery ranges are 7 0-120, but vary for each analyte or method. A surrogate recovery range greater than 110 is unusual. Surrogate recovery less than 10% qualifies the data an unusable (R). High or low surrogate recovery may indicate extraction problems or matrix interference. In samples with high concentrations, the surrogate may be diluted out. Examining the MS/MSD and L CS samples will help determine matrix interference and if the method is in control. Samples with recoveries out of MQOs must be qualified as estimated (J).

The Data Assessment report should include which samples had surrogate recoveries outside of acceptable limits.

4.7.4 Method Blank

Method Blanks are contaminant free reagent water or sand samples spiked with surrogates and internal standards. Method blanks are used to determine if cross contaminants are entering the environmental samples in the laboratory. Analysis of high level samples may leave residual traces of contaminants in the analytical instrument that, in turn, may contaminant later samples. Laboratory contamination also may occur by infiltration of gaseous contaminants through the septum of sample containers. Some contaminants may be leached out of or off-gas from plastic tubing or containers. Information about Method Blanks will be found on data sheet provided by the laboratory.

<u>Evaluation Criteria</u>

To evaluate the Method Blank information:

- Determine if at least one Method Blank was performed per sample batch; and
- Ensure the laboratory has properly qualified data based upon the Method Blanks.

A Method Blank should be completely free of constituents of concern. Any compound detected, other than a surrogate or internal standard may cause the other samples in the sample data group to be qualified as estimated (J).

Environmental samples in the same sample batch with analyte levels 5X or 10X the level of the contaminant found in the method blank will not require qualification. If the sample level is less than 5X or 10X the Method Blank contaminant level (for common laboratory contaminants), the sample should be qualified (B) for blank contamination. Other qualifications may be JB (estimated concentration but blank contamination present) or UB (non-detect but blank contamination present). Blank concentration levels will not be subtracted from the sample levels.

The Data Assessment Report should state what samples were impacted by Method Blank contamination, name the samples the 5X or 10X rule was applied to, and document any changes to sample qualifications.

4.8 Representativeness of Samples.

Representativeness is the degree to which the samples and sample results accurately portray the media and indicate the levels of contaminants in the sampled media. By its nature, sampling is an intrusive act, which disrupts the environmental media and may produce sampling artifacts that are not representative of the true media. Laboratory contamination is another means whereby samples are rendered unrepresentative of the true levels of contamination in the environmental media. Method Blanks (one per sample data group) are analyzed to determine if laboratory contamination occurred or if samples were contaminated during shipping and storage. Sample loss due to volatilization or degradation before analysis also can produce sample results that are not representative of the true levels of contamination in the environmental media. To minimize sample degradation or volatilization, all samples will be preserved according to the Sampling and Analysis Plan and all samples will be shipped and stored at or about 4 C.

The Data Assessment Report will describe how sample representativeness was maintained by determining:

- If Method Blanks for all sample analyses yielded non-detect results;
- Whether any laboratory contamination has biased the sample results;
- Whether the samples were subjected to the proper chemical preservation;
- Whether the samples were chilled to the required 4° Centigrade after collection;
- Whether the cooler receipt forms show that the samples in the sample shipments were received at or about 4 C; and
- Whether the required chain of custody conditions were met.

4 .9 Comparability.

Comparability is a qualitative characteristic that defines the extent to which a chemical parameter measurement is consistent with and may be compared to, values from other sampling events. The Data Assessment Report should make a statement concerning the comparability of the data. To determine the comparability, determine if the laboratory and analytical methodology used for this sampling effort was the same laboratory or same standard methodology used for other investigation or sampling activities.

4 .10Completeness.

Completeness is a quantitative measure of what percent of the chemical measurements are successfully accomplished. Completeness is expressed as a percentage and calculated based on the number of samples successfully analyzed by the laboratory compared to the number of samples that are planned to be collected. The Data Assessment Report should state the degree of completeness. This information may be provided as a project DQO. The completeness goal for the project and whether the required samples were successfully collected and analyzed also should be addressed.

SOP E-801
SOIL SAMPLING

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to discuss equipment and appropriate collection methods for soil sampling. This SOP also provides general sampling instructions for composite sampling and the sampling of specific volatile organic compound (VOC) analytes.

2.0 BACKGROUND

Prior to conducting a soil sampling investigation, a sampling strategy will be developed based on the objectives of the investigation. After designing a soil sampling strategy, the appropriate equipment and techniques must be used to conduct the investigation. This SOP details the following soil sampling methods:

- Surface soil sampling;
- Sampling of soil wastepiles;
- Sampling soil from backhoes;
- Sampling soil from excavations;
- Split-spoon and direct push sampling for collection of soil samples at targeted depths below surface;
- Continuous core auger sampling; and
- Bulk sample collection for large soil sample quantities.

The techniques employed to obtain a soil sample depend on the physical properties of the material to be sampled, the depth from which the sample is collected, the analyte, the quantity, and the location to be sampled. Manual techniques and equipment, such as stainless steel scoops/spoons or hand augers, are usually used for surface or shallow subsurface soil sampling. Power operated equipment is usually associated with collecting deep samples. Power equipment can be used for collecting shallow samples in the event the auger hole begins to collapse, or if soil is so resistant that manual augering is not practical. Samples may be either grab or composite samples depending upon the project sampling objectives. Additional factors to consider during sample collection procedures are the time required to fill a sample container after exposure of the soil to the atmosphere, and the degree of acceptable soil disturbance.

3.0 EQUIPMENT REQUIRED

A copy of the Sampling and Analysis Plan shall be available for review at the site. The following is a list of equipment that may be used for surface soil sampling:

- Stainless steel scoop or spoon or Stainless steel hand-trowel;
- Stainless steel mixing bowl;
- Sample location stakes – PVC or wooden;

- Back hoe;
- Disposable gloves;
- Field notebook;
- Sample description forms;
- Camera and film;
- Pens, pencils,and indelible markers;
- Labels;
- Clinometer;
- Magellan global positioning system (GPS);
- Slide Hammer – Brass sleeves for VOC sample collection;
- Shovels;
- Post-hole hand auger with a 5 foot extension;
- Casing;
- Polyethylene sample bags with fasteners (Ziploc type);
- Sample containers (wide mouth jars or Method 5 035 VOC containers – 6 0 ml and 4 0 ml screw cap PTFE lined septum seals - laboratory preserved as necessary);
- Plastic wrap and clean plastic sheeting;
- Cooler and ice packs (for samples);
- Vermiculite packing material;
- Triplicate Chain-of-Custody forms;
- Representative sample documents;
- Custody seals;
- 5 5 -gallon drums with ring-type closures;
- PID with a lamp with an Ev setting as specified in the Health and Safety Plan;
- Appropriate safety equipment (Heat Stress Monitoring, CGI, PID, etc.) and PPE as specified in the Health and Safety Plan; and
- First aid kit.

In the event that soil VOC samples are to be obtained, the following equipment may be utilized:

- Soil VOC samples by Method 5 035 – top loading balance accurate to 0.01 grams and calibration weights; and
- Soil VOC samplers by Method 5 035 - EnCore™ or Purge and Trap Soil Sampler™ Devices.

4.0 SAMPLING PROCEDURES

The two primary types of surface and subsurface samples are composite and grab samples. The samples are defined on the basis of their sampling origin:

Grab Sample – an individual sample collected from a single location at a specific time or period of time.

Composite Sample – a sample collected over a temporal or spacial range that typically

consists of a series of discrete, equal samples (or " aliquots") that are combined or " composited" .

4.1 Surface Soil Sampling

Sample locations shall be marked by a stake and surveyed or otherwise recorded with reference to local landmarks in such a manner that the location can be re-located.

4 .1.1 Surface Soil Sampling Procedures

All sampling will be performed with properly decontaminated tools and equipment. Samples will be placed into properly labeled sample jars or vials. The proper field book and chain-of-custody entries will be made immediately after sample collection, and the sample will be placed on ice in a cooler chest at a temperature of approximately 4°C. Proper custody of the samples shall always be maintained. All sampling equipment will be decontaminated in accordance with standard decontamination procedures before moving to another sample location.

Non-VOC grab samples

Remove debris, grass, roots, rocks or other material leaving only the bare soil. When sampling from a backhoe bucket or a spoil pile, remove the upper most layer of soil, as this soil may consist of nonrepresentative adjacent soil layers. Prior to entering an excavation to sample the sidewalls, determine if the excavation presents a confined space or collapse hazard. F or confined space or other excavations with forbidden entry, sample from the surface using a slide hammer. When using the slide hammer to sample an excavation sidewall, discard the upper quarter inch of soil, as this soil may be smeared from adjacent soils. If entry into the excavation is possible, other sampling instruments (hand trowels, spoons, etc.) may be used, once the sidewall surface has been scraped to the original matrix. Use a stainless steel spoon, slide-hammer, hand trowel, or hand auger to collect a sample from the depth specified in the F ield Sampling Plan. Collect the soil from the specified depth and place the soil in a stainless steel mixing bowl. Avoid collecting oversized rocks, roots, or other non-soil debris unless specified in the F ield Sampling Plan. Collect enough soil volume from the desired depth to fill the required sample jar(s). Homogenize the soil in the mixing bowl, by the quartering method – divide the sample into quarters – mix each quarter thoroughly, and then remix all four quarters. F rom the homogenized soil, fill the sample jars as full as possible.

Non-VOC Composite Samples

Perform the sample collection procedures as described above for non-VOC grab samples. Obtain the required number of discrete samples from various depths in an excavation, locations in an area, or sidewall locations, as described in the F ield Sampling Plan. A composite sample shall be comprised of not more than 8 discrete samples.

1.2 Bulk Soil Sample Collection

Bulk samples consist of large volume samples from shallow pits generally collected using a backhoe, and loaded into 55-gallon drums. Prior to sampling, all equipment will be decontaminated in accordance with standard decontamination procedures.

First scrape off the upper few inches of the area to be sampled with the backhoe and place the material to the side. Decontaminate the backhoe bucket. Place a suitable number of 55-gallon drums (estimate 500 pounds per drum) onto pallets near the backhoe for filling. After each drum is filled, seal it with the lid and ring closure. Label each drum with an appropriate sampling label with the following information:

- Date;
- Project;
- Location;
- Contents;
- Point of Contact Telephone number;
- Drum Tracking Number or Sample Designation;
- Depth; and
- Number of drum in total (Example: 1 of 3; 2 of 3; 3 of 3).

Before moving the filled drums, clean off any material spills that may have occurred during the sampling process.

1.3 Subsurface Soil Sampling

The method used for subsurface soil sampling is generally a function of the depth of the sample. The interval to be sampled may be determined by the results of PID or Headspace Analysis. However, once the sample is brought to the surface, the method of transferring the sample to the sample container is generally the same, with the exception that special requirements exist for VOC samples. The following procedures are based upon the method of subsurface soil sampling, and the most common method of transferring a grab sample to sample containers is discussed. For soil VOC sampling, very unique sampling methodologies must be employed which are described in Section 5. VOC samples shall not be homogenized or collected as composite samples.

1.4 Soil Sampling by Hand Auger Borers or Core Borers

Auger borings provide a simple method of soil investigation and sampling. Auger boring will be placed on a predetermined spacing grid with samples collected at predetermined depths, as outlined in the site Field Sampling Plan. The depth measurement will be marked on the shaft of the auger. A post-hole auger will be utilized for this procedure since this type of auger has a means of blocking the escape of soil from the auger (refer to ASTM D4547).

Make the auger boring by rotating and advancing the auger into the soil until the auger is full (generally just less than the length of the auger coring bit). Remove the auger and clean off and dump excess boring material when not at sampling depths. Before a sample is collected from a targeted sampling depth, remove all residual material from the auger and decontaminate the auger. To sample, carefully insert the auger into the borehole and rotate and advance until the auger is full. Collect the soil from the specified depth and place the soil in a stainless steel mixing bowl. Avoid collecting oversized rocks, roots, or other non-soil debris unless specified in the Field Sampling Plan. Collect enough soil volume from the desired depth to fill the required sample jar(s). Homogenize the soil in the mixing bowl, by the quartering method – divide the sample into quarters – mix each quarter thoroughly, and then remix all four quarters. From the homogenized soil, fill the sample jars as full as possible.

Obtain the required number of individual discrete samples from locations described in the Field Sampling Plan. A composite sample shall be comprised of not more than 8 discrete samples. For a composite sample over varying depth intervals, obtain discrete samples from determined intervals. In unstable soil a casing with an inside diameter slightly greater than the auger will be required. Advance the casing to a depth not greater than the top of the next intended sample depth, and clean the casing out with the auger. The auger can then be inserted into the borehole and rotated below the bottom of the casing to obtain the sample. This technique may be used when ground water is encountered, but consideration must be given to the loss of water while withdrawing the sample.

1.5 Soil Sampling by Split Spoon and Direct Push Methods

Advance the augers to just above the desired depth. Retract the auger and replace with a split spoon sampler. Record the number of blows to advance the split spoon. Alternatively, by Direct Push sampling, advance the stem with a sample barrel to the desired depth and obtain a soil sample. Collect enough soil volume from the desired depth to fill the required sample jar(s). For composite sampling, the individual discrete samples will be placed into a mixing bowl, and the material will be homogenized by using the method of quartering: divide the sample into quarters, mix each quarter thoroughly, and then remix all four quarters. From the homogenized composite soil, fill the sample jars as full as possible.

A composite sample shall be comprised of not more than 8 discrete samples. For a composite sample over varying depth intervals, obtain discrete samples from pre-determined intervals to be combined.

1.5.1 Composite Sample Collection

Composite samples are a means to gather data over a large area with minimal sample costs; however, lower resolution of contaminate delineation may result. Composite samples will not be obtained for VOC analysis. Composite samples are comprised from 2 but not more than 8 discrete samples. The individual discrete samples are collected in the same manner as grab samples. The individual discrete samples will be placed into a mixing bowl, and the material

will be homogenized by using the method of quartering: divide the sample into quarters, mix each quarter thoroughly, and then remix all four quarters. F rom the homogenized composite soil, fill the sample jars as full as possible.

If the total sample volume is too large for a mixing bowl, then blending and sample size reduction will follow the standard procedures for " coning and quartering" described in ASTM C7 02.

5.0 SOIL VOC SAMPLES

Soil VOC samples must be obtained as grab samples in order that VOC volatilization is minimized. VOC samples shall not be homogenized or collected as composite samples. Excess disturbance or exposure to atmosphere of soils to be sampled for VOCs will cause any VOCs present to be volatilized, which will reduce the representativness of collected samples. Soil VOC samples should be collected as soon as possible after the soil has been exposed to the atmosphere and with as little disturbance of the soil as possible.

5.1 Soil VOC Sample Collection

The F ield Sampling Plan will describe the method of Soil VOC collection. Many States are now requiring SW-8 4 6 Method 5 035 which requires the use of hermetically sealed vials preserved with Sodium B isulfate (for low anticipated VOC concentration) or Methanol (for high anticipated VOC concentration).

A small coring device EnCore^{tm} or Purge and Trap Soil Sampler^{tm} or a cut plastic syringe is used to obtain a soil plug (2-5 grams) from undisturbed soil. These devices are used to minimize soil disturbance and to trap VOCs in the soil during transfer to the soil vials. The soil face will be scraped clear of debris, smeared soil, rust from core-borers or split spoons, or other material that would interfere with obtaining a soil plug by the corer. The core will be obtained as quickly as possible (within a few minutes) from the soil after it has been brought to the surface from boring activities, slide hammer, or from a backhoe bucket.

Once the soil plug is obtained within the corer, the soil plug will be immediately transferred to the appropriate preserved soil vial, as described in the F ield Sampling Plan or in the instructions supplied by the analytical laboratory. Gloves will be worn whenever handling the sample vials. The sample vials will be filled as completely as possible and wiped clean of dirt and moisture. The sample vials will be weighed to the nearest 0.01 grams. Alternatively, if prescribed in the F ield Sampling Plan, trials to determine the weight versus length of soil column in the syringe will be performed, and the length of soil corresponding to 5 .0 + /- 0.5 grams will used for future soil collections. The soil vials will never be opened in the field once soil has been deposited into the vials. Always handle the soil vials with gloves, before and after sample collection. More complete Method 5 035 instructions can be obtained from the analytical laboratory or the manufacturer. Upon collection, VOC sample containers will be placed into plastic bags and placed upon ice in a cooler at 4°C.

5.2 Headspace Analysis

Headspace analysis is a means of determining which portion of soil within a split spoon or core borer soil sample core has the highest VOC concentration. Headspace analysis is a crude field screening method and there are several variations of this field screening method. Variations include simply passing a PID or F ID probe up and down the exposed soil sample core, while another method utilized for extruded sand from a Sonic Rig is to obtain a reading from the punctured plastic sample liner. The Headspace analysis method described below is a standard methodology.

If high humidity conditions exist during the time period when field activity is to be performed, the F ID is recommended since a PID is not a reliable screening instrument under these conditions. The ionization potential of the lamp for the PID shall be optimal for the contaminants of concern.

Immediately upon opening the split-spoon (or other sample retrieval device) and after collecting the volatile organic sample (if required), a representative portion of the sample shall be collected and placed in a clean, contaminate-free jar. The sample may be placed in a new, clean, plastic sandwich bag inside a jar to minimize the number of new jars required. Additional headspace readings must be taken inside the plastic bag or sample jars prior to their use to ensure no external contamination is present.

If the volume of sample recovered is insufficient for all analytical requirements, then the material used in the headspace readings could be included with any nonvolatile sample matrix. Seal each jar with at least one continuous sheet of aluminum foil, using the jar lid to secure the foil. Vigorously agitate the sample jar for at least fifteen seconds - and then allow a minimum of ten minutes (or as the environmental conditions dictate) for the sample to adequately volatilize.

During cold weather, warm the samples to near room temperature prior to taking the headspace measurement. Re-agitate the jar and then remove the jar lid. Quickly insert the PID or F ID probe through the aluminum foil and record the maximum instrument reading (which should be within the first two to five seconds). Erratic responses should be evaluated in terms of high organic vapor concentrations or conditions of elevated headspace moisture.

Record the headspace screening data on the boring log and any other appropriate documentation (e.g. sample transmittals, field notebooks, etc.) as appropriate.

Calibration of the screening instrument is performed with the appropriate calibration gas and shall be calibrated at least twice daily, and before use after a long shut down period (i.e. lunch breaks, equipment breakdowns, weather caused breaks, etc.).

If sample jars are to be re-used in the field, jars must be cleaned according to field decontamination procedures for the cleaning of sampling equipment.

SOP E-1101
FIELD QUALITY CONTROL SAMPLES

1.0 OBJECTIVE

The objective of this Field Quality Control Sample Standard Operating Procedure (SOP) is to establish procedures for the preparation and implementation of field quality control samples.

2.0 BACKGROUND

Field quality control samples are prepared as a check of field sample collection methods and as a check of cross-contamination of sample in the field or during shipment. Equipment Rinsate, Field Blank, and Trip Blank samples check for potential cross-contamination, which impacts sample representativness. Field duplicates measure the precision and reproducibility of the field sampling efforts. Matrix Spike/Matrix Spike Duplicate (MS/MSDs) samples determine the potential for site matrix interferences in determining the true level of contaminants in the sampled matrix. Field quality control samples consist of the following:

Field Duplicate/Replicate samples are independent samples of a homogeneous matrix collected at the same time from the same location and are analyzed to check the field variability of sampling. Field Duplicates are generally required QC samples according to many analytical methods.

Equipment Rinsate samples are samples of analyte free (deionized) water which are rinsed over decontaminated equipment, collected, and submitted for analysis. Equipment rinsate samples are analyzed to determine if contaminants from one sampling activity are being transferred to another sample. The primary purpose of running Equipment Rinsate samples is to determine if the decontamination procedures are adequate.

Field Blanks are samples that are prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example: air-borne dust or organic vapors).

Trip Blanks are required only when samples are collected for analysis of volatile organic compounds (VOC)s. Trip Blank samples are prepared prior to the sampling event in the actual sample container and stored continuously with the investigative samples throughout the sampling event. Trip Blanks are used to determine if VOC samples were contaminated during storage and/or transportation back to the laboratory (a measure of sample handling variability resulting in positive bias in contaminant concentration). Trip Blanks are collected as a quality control check on aqueous VOC samples submitted to the laboratory, and to determine if the aqueous VOC samples were contaminated by either sample containers or during sample shipment.

Matrix Spike/Matrix Spike Duplicates are samples collected from the site and are used by the

laboratory to determine the potential for matrix interferences, which may bias results. Matrix interferences are site specific, so site samples should be obtained for MS/MSD samples. Some laboratories will use soils or waters from an environmental sample for the MS/MSD sample. MS/MSD samples, whether collected as samples or obtained from environmental samples, are generally required QC samples according to many analytical methods.

3.0 EQUIPMENT REQUIRED

- Labels;
- Custody seals;
- Chain-of-Custody forms;
- Appropriate sampling containers;
- Indelible ink pens; and
- Field book.

4.0 PROCEDURE

Field QC samples shall be collected, containerized, preserved, stored, and shipped in the same manner as environmental samples. Generally, MS/MSDs, Equipment Rinsates, Field Blanks, and Trip Blanks are designated on the Chain of Custody and sample designation, as prescribed in the Field Sampling Plan or as in SOP –102 Sampling Handling and Custody Requirement.

4.1 Sample Handling and Mixing

After collection, all sample handling should be minimized. Investigators should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, investigators should ensure that the sample containers do not become submerged in melted ice, as this may result in sample cross-contamination. Plastic bags, such as Zip-Lock® bags or similar plastic bags sealed with tape, should be used when small sample containers (e.g., VOC vials or bacterial samples) are placed in ice chests to prevent cross-contamination.

It is extremely important that soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

1. The material in the sample pan should be divided into quarters and each quarter should be mixed individually;
2. Two quarters should then be mixed to form halves; and
3. The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4.2 Field Duplicate/Replicate Samples

Field Duplicate/Replicate samples will be collected at the same sampling location and immediately placed into the proper sample containers. Each duplicate sample will be analyzed for the same suite of analytes as the original sample. A minimum of one duplicate will be collected per twenty samples, but no less than one duplicate per sampling shift.

Duplicate samples will be collected by initially collecting a minimum of twice as much material as normally collected for a sample. After mixing, the material will be apportioned into two sets of sample containers. Both sets of containers will be submitted for analyses with one set designated with an appropriate sample identification number; the second possessing a fictitious sample number to mask the identity of the duplicate sample from the laboratory.

4.3 Equipment Rinsate Samples

Rinsate samples are samples of deionized (DI) water poured over decontaminated sampling equipment. The Rinsate Blank is collected during the final rinse of the sampling equipment after decontamination procedures are performed. Rinsate samples are analyzed for the same parameters as field samples to evaluate the effectiveness of field decontamination procedures. One rinsate Blank will be collected each day that sampling is performed.

4.4 Field Blanks

Field Blanks are collected in the field during sampling activities by pouring analyte-free water directly into the sample containers at the predetermined field sampling location. Field Blanks are analyzed for the same analytes as the related samples. At a minimum, one Field Blank will be collected during each sampling day. Field Blanks should be collected in dusty environments and/or in areas where volatile organic contamination is present in the atmosphere that originates from a source other than the source being sampled. Field Blanks are analyzed for the same analytes as the related samples. Field Blank samples will be collected with no headspace present within the sample container.

4.5 Trip Blanks

Trip Blanks generally are collected as a quality control check on volatile organic samples. When volatile organic samples are shipped to the recipient laboratory, trip Blanks will be provided with each shipment, but not for each cooler. They are packaged for shipment with the other samples and submitted for analysis. At no time after their preparation are trip Blanks to be opened before they reach the laboratory. Trip Blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-of-Custody Records as Trip Blanks. A minimum of one Trip Blank shall be transported per one to ten samples collected.

The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site:

Water Sample VOC Trip Blank - A water sample VOC trip Blank is required for every study where water samples are collected for VOC analysis. Two sealed preserved (or unpreserved if appropriate) 40-ml VOC vials will be transported to the field. For routine studies the samples will be prepared by lab personnel. Investigators shall request that these samples be provided at least one week in advance of scheduled field investigations and inspections and never less than two days in advance of scheduled field investigations and inspections (except in emergency situations). These samples should not be picked up earlier than the morning of departure for the scheduled inspection/investigation.

Soil Sample VOC Trip Blank - A soil sample VOC trip Blank is required for every study where soil samples are collected for VOC analysis. The preparation and pick up of this sample will be the same as for the water sample VOC trip Blank. One two-ounce soil VOC vial will be transported to the field. This Field Blank will be handled and treated by personnel in the same manner as the soil samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-Of-Custody Records as trip Blanks.

MS/MSDs - MS/MSDs are collected from the same sample interval or location as environmental samples. The MS/MSDs are required batch QC samples. If MS/MSD samples are not sent samples volume from environmental samples from the site or another site will be analyzed by the laboratory.

2.1 Project Laboratory

Ensure the selected laboratory has the proper certification or validation requirements to satisfy the project DQOs. In the SAP, present the point of contact, full name of the laboratory, laboratory address, phone number, and fax number. State the laboratory's certifications or validation status (i.e., USACE, AF CEE…). Acquire the Quality Assurance Manual (QAM) of the selected laboratory prior to completion of the SAP. After review and insertion of applicable laboratory information into the SAP, present the QAM as an appendix to the SAP.

3.0 RESPONSIBILITIES

Project Manager: The Project Manager will be responsible for ensuring compliance with the project's DQOs by

- Selecting a laboratory able to meet the required sensitivity;
- Providing oversight of sample collection, custody, and handling;
- Providing oversight of the project data assessment; and
- Providing oversight of the final submission of project required data reports and/or data assessments.

The Project Manager will understand the DQO development process and how project decisions may impact the quality of the project data.

Project Engineer: The Project Engineer will be responsible for

- Oversight of the sample collection team to ensure proper sample handling and custody procedures are followed and samples are collected in accordance with the SAP.
- Performing routine data assessment to determine if the data supports the project's DQOs; and
- Prepare as part of a Final Report or other interim reports a written data assessment, based upon the format described in the following section.

4.0 DATA ASSESSEMENT PROCEDURES

Data assessment is performed by reviewing the DQIs and applicable non-laboratory criteria. The data assessment may be performed on the complete project or each discrete sampling site. The items to be reviewed in the data assessment and the format of the Data Assessment Report are presented in the following subsections.

Each analytical method for all matrixes must be assessed. The following guidance is intended for a review of data sets performed with reasonable accuracy and precision. The contract laboratory must evaluate problems with the its instrumentation, out of control situations, or matrix interferences. Analytical batches with sever QC problems should be re-analyzed by the

APPENDIX B

CHAIN-OF-CUSTODY RECORD

Customer Name:_____

Address:_____

Contact:_____

Phone:_____

Fax:_____

Customer Project Name:_____

Customer Project Number:_____

P.O. Number:_____

Date	Time	Type	Client Sample Identifer	Tests	Container(s)	Matrix	Sample No.

Notes:

Requested Turnaround Time:

Relinquished by:	Recieved for Laboratory by:
(Signature)	(Signature)
Date:	Date
Time:	Time

DAILY QUALITY CONTROL REPORT

Site: _____
Project No: _____
Date: _____
Crew No: _____
Crew Mem: _____

Weather (circle)

	Bright Sun	Clear	Overcast	Rain	T-storm	Snow
Temp:	to 32	32-50	50-70	70-85	85+	
Wind:	Still	Gusty	Moder.	High	**Direction:**	
Humidity:	Dry	Moder.	Humid			

Subcontractors and Equipment on Site: _____

Health and Safety Levels: (circle)

D	Mod. D.	C	B	A

Summary of Health and Safety Activities: _____

Instrument Used: (circle)
Calibrated: (check)

PID	LEL	pH	Cond.	Therm.	Turbidity	DO	ORP

For actual calibration results, see field calibration forms.

Summary of Work Performed: _____

All Samples Were Collected According to Procedures Outlined in the Work Plan?
Yes _____ No _____

Problems Encountered/Corrective Action Taken: _____

Time Project Manager Contacted: _____

Tomorrow's Expectations: _____

Name: _____ Signature: _____

Custody Seal

Environmental Chemical
Corporation
999 18th Street, Ste 2350
Denver, CO 80202
303-298-7607

CUSTODY SEAL

Date: _____

Signature:_____

Environmental Chemical
Corporation
999 18th Street, Ste 2350
Denver, CO 80202
303-298-7607

CUSTODY SEAL

Date: _____

Signature:_____

Environmental Chemical
Corporation
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Denver, CO 80202
303-298-7607

CUSTODY SEAL

Date: _____

Signature:_____

Environmental Chemical
Corporation
999 18th Street, Ste 2350
Denver, CO 80202
303-298-7607

CUSTODY SEAL

Date: _____

Signature:_____

Environmental Chemical
Corporation
999 18th Street, Ste 2350
Denver, CO 80202
303-298-7607

CUSTODY SEAL

Date: _____

Signature:_____

FINAL
QUALITY ASSURANCE PROJECT PLAN

Site 8 – Herbicide Orange Storage Area and Off-Base Area of Contamination

Naval Construction Battalion Center
Gulfport, Mississippi

October 2004

Prepared for:

Southern Division
Naval Facilities Engineering Command
2155 Eagle Drive
North Charleston, SC 29406

Prepared under:

Contract Number N62467-02-D-0468
Contract Task Order 002

FINAL
QUALITY ASSURANCE PROJECT PLAN

FOR

SITE 8 – HERBICIDE ORANGE STORAGE AREA

AND

OFF-BASE AREA OF CONTAMINATION

Naval Construction Battalion Center
Gulfport, Mississippi

Submitted to:

Southern Division
Naval Facilities Engineering Command
2155 Eagle Drive
North Charleston, SC 29406

Submitted by:

Environmental Chemical Corporation
1040 Bayshore Highway
Burlingame, CA 94010

Contract Number N62467-02-D-0468
Contract Task Order 002

October 2004

PREPARED UNDER THE SUPERVISION OF:

_____
Prashant Khanna
Project Manager
Environmental Chemical Corporation

APPROVED FOR SUBMITTAL BY:

_____
Robert W. Anderson
Quality Control Manager
Environmental Chemical Corporation

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LIST OF ACRONYMS AND ABBREVIATIONS

AOC	Area of Concern
BTEX	benzene, toluene, ethyl benzene, and xylene
CAS	Columbia Analytical Services, Inc.
CLP	Contract Laboratory Program
DQI	Data Quality Indicators
DQO	data quality objectives
ECC	Environmental Chemical Corporation
EPA	Environmental Protection Agency
FSP	Field Sampling Plan
HO	Herbicide Orange
J	qualified as estimated
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LQAM	Laboratory Quality Assurance Manual
MDL	method detection limit
MQL	method quantitation limit
MQO	method quality objectives
MS	matrix spike
MSD	matrix spike duplicate
Navy	United States Navy, Southern Division, Naval Facilities Engineering Command
NCBC	Naval Construction Battalion Center
PARCC	precision, accuracy, representativeness, completeness, comparability
PE	performance evaluation
PM	project manager
QA	quality assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	quality control
QCM	Quality Control Manager
RA	remedial action
RL	reporting limit
RPD	relative percent difference
SDG	sample delivery group
SOP	standard operating procedure
SPLP	synthetic precipitation leaching procedure
TCLP	toxicity characteristic leaching procedure
TPH	total petroleum hydrocarbons
U	non-detect

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) was prepared by Environmental Chemical Corporation (ECC) for the United States Navy, Southern Division, Naval Facilities Engineering Command (Navy), Contract Number N62467-02-D-0468, Contract Task Order 002, for the Remedial Action (RA) of the Site 8 Herbicide Orange (HO) Storage Area and Off-Base Area of Contamination located at the Naval Construction Battalion Center (NCBC) in Gulfport, Mississippi [Field Sampling Plan (FSP) Figure 1-1].

The QAPP provides a consistent and detailed framework of policies, procedures, functional activities, and organization to be used during execution of the Remedial Action at the Site 8 NCBC. The QAPP outlines the quality assurance (QA) program and quality control (QC) procedures that will be used to verify and maintain the level of performance required to meet the project objectives. The QAPP will define the procedures required to ensure that data obtained during sampling activities are of acceptable and verifiable quality and attain project data quality objectives (DQOs) as discussed in Section 4.0 of the QAPP. In addition, the QAPP provides specific descriptions of how the QA program will be implemented at the laboratory.

Project history including the site description is presented in Section 1.0 of the FSP. The project scope of work is discussed in Section 3.0 of the FSP.

The purpose of a QAPP is to ensure that the project analytical data produced is of known quality that satisfies the project objectives. To meet this data quality requirement, the QAPP provides the following:

- Mechanism for the ongoing control and evaluation of data quality; and
- Methods to measure the data quality in terms of precision, bias (accuracy), sensitivity, representativeness, completeness, and comparability [formerly precision, accuracy, representativeness, completeness, comparability (PARCC)] to assess whether the data meet the project objectives and can be used for their intended purpose.

The objective of the chemical measurement data is to generate sufficient information to determine the presence or absence of chemical contamination within the media of each site and evaluate the effectiveness of each treatment system. To meet this objective, data acquired during the sample collection phase must be defensible. The quality objectives for the chemical data specify the quality of the data needed to enable project personnel to make project decision (i.e., the decision to determine the effectiveness of contaminant removal, stabilization, etc). As such, the objectives determine the type and quantity of data needed to make a decision, as well as the measurement objectives (precision, accuracy) for each type of measurement data collected.

The objectives for the Site 8 NCBC Remedial Actions analytical data are:

- To collect samples necessary to attain the data required for project decisions;
- To collect and analyze samples under controlled situations using standard methods;
- To obtain usable and defensible analytical results; and
- To achieve a completeness goal of 90%.

The following sections discuss the steps that will be taken to ensure the validity of the data acquired during the Site 8 NCBC Remedial Actions. The representativeness of the measurement data is a function of the sampling strategy and will be achieved by following the procedures discussed in the FSP. The quality of the analytical results is a function of the analytical system and will be achieved by using standard methods and the quality control system discussed in the QAPP. The basis for assessing the data quality indicators (DQIs) of precision, bias (accuracy), sensitivity, representativeness, completeness, and comparability (formerly PARCC) of the data; the specific calculations for these data quality measurements; and the data assessment procedure are presented in Section 8.0 of this QAPP.

The QAPP is based on and is substantially consistent with the project specifications and with the following documents to the extent applicable:

- U. S. Environmental Protection Agency (EPA), 2000a. Requirements for Quality Assurance Project Plans for Environmental Data Operations (EPA QA/G-4 HW, January 2000);
- EPA, 2001a. Guidance for Quality Assurance Project Plans (EPA QA/G-5, March 2001);
- EPA, 2001b. Data Quality Objectives for Quality Assurance Project Plans (EPA QA/R-5, March 2001);
- EPA, 2000b. Test Methods for Evaluating Solid Waste (SW-846); and
- EPA, 2000c. Guidance for Data Quality Assessment - Practical Methods for Data Analysis (EPA QA/G-9, July 2000).

2.0 PROJECT LABORATORY ORGANIZATION

The following sections describe the laboratory organizational structure of Columbia Analytical Services, Inc. (CAS) of Houston, Texas. CAS Houston will be used to perform the project dioxin analyses using EPA Method 1312/8090. The following sections present the responsibilities of key personnel who will ensure the data collected meet the data quality control and quality assurance requirements outlined in the QAPP.

The CAS certifications are listed in Appendix F of the Laboratory Quality Assurance Manual (LQAM), which is provided in Appendix A of this QAPP. Appendix B of the LQAM depicts the laboratory organizational structure. A brief description of the roles and responsibilities of laboratory personnel with specific QC or QA responsibilities are listed in the following subsections.

2.1 Laboratory Director

The role of the Laboratory Director is to provide technical, operational, and administrative leadership through planning, allocation, and management of personnel and equipment resources at the laboratory. The Laboratory Director has the following duties:

- Provide leadership and support for the QA program;
- Responsible for overall laboratory efficiency;
- Provide resources for implementation of the QA program, and reviews and approves the Laboratory Quality Assurance Manager (QAM);
- Review and approves standard operating procedures (SOPs);
- Identify and respond to QA needs; and
- Summarize and report overall unit performance, including round-robin programs, certification and accreditation activities, and blind and reference sample analysis.

2.2 Laboratory Quality Assurance Director

The Laboratory Quality Assurance Director is responsible for the overall QA program at all the CAS laboratories. The QA Director has the following duties:

- Perform an annual on-site audit at each CAS laboratory and prepare a written report;
- Maintain a database of information about state certifications and accreditation programs;
- Write laboratory-wide SOPs;
- Maintain a database of CAS-approved subcontract laboratories;
- Provide assistance to the laboratory QA staff and laboratory managers;
- Prepare an annual QA activity report;
- Identify and respond to organizational QA needs; and
- Summarize and report overall CAS performance, including round-robin programs, certification and accreditation activities and blind and reference sample analysis.

2.3 Laboratory Quality Assurance Manager

The responsibility of the Laboratory QAM is to oversee implementation of the quality program and to coordinate overall QA activities within the laboratory. The QAM has the following duties:

- Maintain the QA Manual and performing the annual QA Manual review;
- Review, approve, and control laboratory SOPs;
- Coordinate performance evaluation (PE) sample analyses and prepare corrective action reports for any missed PE sample results;
- Review chemical data;
- Maintain the laboratory's certifications and approvals; and
- Perform internal QA audits and preparing QA reports.

2.4 Laboratory Project Manager

The Laboratory Project Manager is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project manager has the following duties:

- Ensure that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements;
- Coordinate with laboratory technical staff and administrative staff to ensure that client-specific needs are understood; and
- Ensure the services CAS provides are properly executed and satisfy the requirements of the client.

2.5 Stop Work Authority

The Laboratory Director, Quality Assurance Director, QAM and each have the authority to stop work in the laboratory in case of quality problems. Each has the authority to request appropriate corrective actions be implemented immediately.

3.0 DATA ASSESSMENT ORGANIZATION

The following sections describe the ECC organization structure that will be implemented to ensure the data collected in support of the Site 8 NCBC remediation activities meet the project objectives and the specific requirements outlined in the QAPP. The overall project organization, including a list of key personnel and their responsibilities, is provided in Section 2.0 and depicted in Figure 2-1 of the FSP. A brief description of the roles and responsibilities of task personnel with specific chemical QC responsibilities are listed below.

3.1 ECC Quality Assurance Manager

In accordance with the ECC Quality Assurance Program, the Corporate QAM, Mr. Brady Bigelow, has overall responsibility and authority for development and management of the Contract Quality Control Program.

3.2 Quality Control Manager

The Quality Control Manager (QCM), Mr. Bob Anderson, is responsible for supervising the quality control aspects of the project and has the authority to act independently in all quality control matters. The QCM will report directly with the Program Manager. With regard to this QAPP the QCM duties will include the following:

- Maintain communication between project management and project team members;
- Ensure data packages received from the laboratory are complete;
- Recommend corrective action procedures to maintain project QA objectives;
- Ensure that corrective action is implemented when necessary; and
- Act as the primary spokesperson on quality matters when interfacing with external organizations;
- Be available for consultation with Navy and applicable ECC personnel regarding project chemistry concerns; and
- Ensure that all chemistry related goals for the project are attained.

The QCM will be available for consultation with Navy and the laboratory personnel during remediation activities. With respect to data evaluation, the QCM or his designee will have the following duties:

- Ensure that project requirements for data documentation and evaluation are attained;
- Evaluate chemical data results;
- Review QC results of each laboratory data package;
- Perform data validation on the EPA National Functional Guidelines for Chlorinated Dioxin/Furan Data review (EPA, 2002);
- Prepare sample result tables and figures; and
- Coordinate with Navy personnel regarding the chemical quality management of the project.

4.0 DATA QUALITY OBJECTIVES

Project DQOs are an integrated process used to define data quality requirements based on the intended use of the data. DQOs are qualitative and quantitative statements that:

- Clarify the project remediation objectives;
- Define the data required for the project;
- Determine the project decision-making levels; and
- Specify the level of decision errors acceptable for data to support the project decisions.

Understanding the DQO process is critical in evaluating the quality of the chemical data.

The overall project QA objective is to develop and implement procedures for obtaining and evaluating data that meet the DQOs to ensure or confirm that the required project criteria are accomplished. QA procedures are established to ensure field measurements, sampling methods, and analytical data provide information that is comparable and representative of actual field conditions, and that the data generated are technically defensible. Specifically, chemical data will be generated to determine if the remediation criteria at the site and the treatment criteria of the soil meets the proposed cleanup goals. Specifically, analytical data will be collected to indicate the effectiveness of the stabilization treatment technique to inhibit the leaching of dioxin in the interest of minimizing the threat of contamination to surrounding groundwater systems. Fill material will be brought in from off-site to backfill the excavated areas. The project cleanup goals/governing regulatory limits and required laboratory quantitation limits are presented in Table 4-3 of the FSP.

Chemical Data will be quantitatively and qualitatively validated using rigorous methods of analysis and QA according to the EPA National Functional Guidelines for Chlorinated Dioxin/Furan Data review (EPA, 2002). Substantiating the results of the data provides confidence in the project data so decisions can be made regarding physical and/or chemical properties of the samples, verification of contaminant identification, and other decisions relevant to the project.

4.1 Data Use Background

To determine the project DQOs, a series of planning steps are used as specified in the EPA Guidance for Data Quality Objective Process QA/G-4 (EPA, 2000a). The DQO development process is used to optimize the data collection necessary to meet the applicable decision criteria. The seven steps of the DQO process are presented in the following sections.

4.1.1 Step 1: State the Problem

Excavation and on-site treatment of contaminated soil at the site is being implemented to remove the human health and environmental risk that was identified during the site investigations and to prevent any leaching of the soil contaminants into the groundwater. Excavated soil will be subjected to cement stabilization treatment based on previously performed bench-scale study analytical results. Soil samples from areas of known contamination will be mixed with the cement stabilization agent. Subsequent to the mixing, the resulting matrix will be sampled and submitted to the analytical laboratory for dioxin leachate analysis by EPA Method 1312/8290 to assess the effectiveness of the stabilization treatment. After the sampling of the initial mixing is demonstrated to meet the project goals, field demonstration samples will be collected. The field demonstration samples will consist of a mixture of soils from the contamination streams (off-base excavated soils and wetland areas, on-base sediment, and ash) which have been subjected to the stabilization and compacting remediation processes. These samples will demonstrate the efficacy of the stabilization treatment for large-scale clean-up. Subsequent to the stabilization and compacting treatment of the excavated soils, treated material will be stockpiled at Site 8A (Figure 1-2 of the Work Plan). Confirmation samples will then be collected and submitted to the analytical laboratory for the verification of the attainment of project clean-up goals. Subsequently, the stockpiles will be capped with a roller-compacted concrete cap.

The ECC decision team will consist of the Project Manager, QCM, Project Engineer, and the Site Superintendent.

4.1.2 Step 2: Identify the Decision

A review of the applicable analytical results will enable the decision team of Navy and ECC to make the following decisions to determine if the remedial technologies are attaining the cleanup goals:

- Establish if the remediation technology is attaining the operation goals;
- Determine whether the cement solidification performed on the bench-scale samples effectively contains the dioxin to meet the project clean-up goals;
- Determine if the cement solidification remediation technique is effective at meeting project specifications on the mixed contamination stream of off-base excavated sediment and wetland soil, and on-base soil samples;
- Confirm the overall effectiveness of the remediation technique subsequent to the treatment of the excavated soil; and
- Determine whether the stockpiles of treated material meet project clean-up goals and can be affixed with a roller-compacted concrete cap.

4.1.3 Step 3: Identify Inputs to the Decision

Decisions will be made based on individual analytical data received from all sampling events conducted over the course of the Site 8 NCBC Remedial Actions. Sampling activities are described in Section 4.0 of the FSP. EPA methods will be used for all analyses.

The field samples of the cement stabilization treated soil will be analyzed for dioxin using EPA Method 1312/8290. Analytical results will be compared to the proposed project cleanup goals shown in Table 4-2 to assess the effectiveness of the cleanup effort.

4.1.4 Step 4: Define the Boundaries of the Project

NCBC Gulfport is located in the southeastern corner of Mississippi, approximately two miles north of the Gulf of Mexico. The base is located in the western part of the city of Gulfport in Harrison County (FSP Figure 1-1). The off-base area of concern (AOC) is located north of NCBC, across 28th Street near Outfall 3. The base occupies 1,100 acres with an average elevation of approximately 30 feet above sea level, the only significant exceptions being two rectangular piles of bauxite (aluminum ore) stored on the surface that are approximately 45 feet higher than the adjacent ground. A map of NCBC Gulfport is provided in the FSP (Figure 1-2).

The excavation areas are indicated on the project drawings.

4.1.5 Step 5: Develop a Decision Rule

Analytical results from the solid samples collected during the site remediation activities will be used to evaluate the effectiveness of the remedial technology. The results will be compared to proposed cleanup goals defined in Table 4-2 of the FSP. If the analytical results of the remediated material are less than the proposed cleanup goals the remedial technology is deemed to have met the operational goals of the project. If the contaminant concentrations are greater than the cleanup concentration listed in Table 4-2, then further assessment of the remediation technology is necessary and re-treatment of the excavated soil must be considered.

4.1.6 Step 6: Specify Tolerable Limits on Decision Errors

The sample data are subject to random and systematic errors during field collection and sample analysis. The combination of errors is referred to as “total study error.” The two contributors to the total study error are the sample design error and the measurement error. The sample design error occurs when the data collection plan fails to provide a limited variability within the decision unit necessary for an accurate decision. Measurement errors are introduced during processes the sample is subjected to; such as physical collection, sample handling, sample preparation, sample analysis, data reduction, and transmission. Since the total study error directly affects the possibility of making a decision error, the total decision error must be managed by minimizing the sample design and measurement errors.

Environmental decisions affecting human health and the environment are usually determined by baseline conditions, such as action levels and regulatory limits. The baseline conditions for this project are the project clean-up goals; therefore, the hypothesis testing that will be applied to any decision procedure is a choice between the applicable clean-up goal for the soil and an alternate position as the following hypotheses indicate:

Null Hypothesis: The dioxin soil concentrations do not exceed the applicable clean-up goal for treated material.

Alternative Hypothesis: The dioxin soil concentrations exceed the applicable remediation goal for treated material.

4.1.7 Step 7: Optimize the Design

The variability of data may have an effect on the sampling design. If necessary, the sample frequency or procedure may be changed to optimize the design. The design options will be evaluated based on cost and ability to meet the DQOs and presented to the Navy for approval before implementation.

To achieve the project DQOs, this QAPP is designed to ensure that a sufficient number of samples will be collected using technically valid scientific procedures. The DQOs for this project require the quantitative and qualitative validation of 100% of the final sample results according to EPA National Functional Guidelines for Chlorinated Dioxin/Furan Data review (EPA, 2002). Substantiating the results of the data provides the level of confidence in the data necessary to so decisions can be made regarding project remediation goals.

4.2 Measurement Quality Objectives

Measurement quality objectives (MQOs) are the laboratory QC acceptance criteria that ensure quality data are continuously produced. To determine the reproducibility of the analytical procedures and test results, systematic QC checks are used during the analytical process. The systematic checks include analysis of field and laboratory replicates, laboratory standards, surrogates, spiked samples, and blanks. MQOs (acceptance criteria or ranges) for the systematic QC checks are established to assess whether the DQIs support the usability of the data. The DQIs are measurable attributes that are defined in terms of precision, bias (accuracy), sensitivity, representativeness, completeness, and comparability (formerly PARCC) parameters. The assessment of the DQIs is necessary to determine data usability. Table 4-1 presents the MQOs and DQIs for this project.

Review of the data includes the laboratory analytical methods, minimum detection limits, decision criteria for clean-up goals, and error tolerance limits. SW-846 tolerance parameters in accordance with LQAM and the project FSP protocols will be used to assess analytical data. Data that meet the quality objectives and goals will be deemed acceptable. Data that do not meet objectives and goals will be reviewed on a case-by-case basis to ascertain their usefulness.

5.0 SAMPLE CUSTODY AND HANDLING

The FSP details the field procedures used to protect the integrity of project samples from collection to shipment of the sample containers in FSP Sections 5.0 through 7.0. A stringent program of custody procedures is utilized to ensure that each sample is accounted for from the time of collection to analysis. This section of the QAPP outlines the specific procedures followed at the laboratory to document sample history and maintain the integrity of the sample.

5.1 Transfer of Custody

The Chain-of-Custody Record accompanies each group of samples. When custody of samples is transferred, both the individual relinquishing the samples and the individual receiving the samples must sign, date, and indicate the time of transfer on the Chain-of-Custody Record. When responsibility for a group of samples changes several times, each custodian is not required to retain a copy of the Chain-of-Custody Record, as long as the original Chain-of-Custody Record indicates that each person accepting the samples has subsequently relinquished custody appropriately.

5.2 Receiving Samples

Samples delivered to the CAS sample management office are received by a sample custodian. The Chain-of-Custody Record is reviewed for completeness and accuracy and Cooler Receipt and Preservation Form (Appendix B) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity by the sample custodian includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc);
- Entries include sample identification, description, date, time and location of sample collection, the name and signature(s) of the sample collector and intermediate sample custodian(s), date and time of each sample transfer, and signature of the CAS sample custodian upon receipt;
- Sample containers checked for integrity (broken, leaking, etc);
- Sample clearly marked with the sample ID, date and time of collection;
- Appropriate containers (size, type) are received for the requested analyses;
- Sample container labels and/or tags agree with Chain-of-Custody Record entries (identification, required analyses, etc);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- Inspection of volatiles containers for the presence/absence of bubbles.

The laboratory will fax or email the completed Cooler Receipt and Preservation Form to the project site the same day the samples arrive at the laboratory (Appendix B).

5.3 Sample Storage

All samples are stored in the laboratory sample refrigerators at 4°C ± 2° C by the laboratory custodian. To prevent a preservation error the refrigerators and freezers are segregated according to matrix type and in some cases method of analysis. The temperature of each thermal storage unit used at CAS is monitored daily (business days), using a NIST traceable thermometer, and the data is recorded in a bound logbook.

The following is a list of standard storage procedures, but may not include all conditions:

- Samples are stored away from all standards, reagents, food, and other potentially contaminating sources; and
- Sample fractions, extracts, leachates, and other sample preparation products are stored according to the described protocol or according to specifications in the test method.

5.4 Sample Holding Times

CAS uses the sample preservation, container, and holding-time recommendations published in *Test Methods for Evaluating Solid Waste*, SW-846 (EPA, 2000b), Third Edition and Updates I, II, IIA, IIB, III for hazardous waste samples, and EPA 600/4-79-020,600/4-91-010 and Supplement I, 600/4-82-057, 600/R-93/100, 600/4-88-039 and Supplements I and II, and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. The container, preservation and holding time requirements are summarized in Table 4-2 of the FSP.

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6.0 LABORATORY ANALYTICAL PROCEDURES

The LQAM for CAS, included in the QAPP as Appendix A, presents the QA and QC procedures the laboratory utilizes during analysis and reporting to provide data with quality necessary for its intended use. The QA/QC objectives and procedures and calibration procedures are included in the LQAM.

6.1 Laboratory Analytical and Measurement Procedures

The laboratory analytical program in support of the Site 8 NCBC Remedial Actions will include:

- Synthetic Precipitation Leaching Procedure (SPLP) for dioxin using EPA Method 1312/8090.

Fill material brought in from off-site for backfilling excavations will be tested for the following chemical constituents:

- Total petroleum hydrocarbons (TPH) using EPA Method 1664;
- Benzene, toluene, ethyl benzene, and xylene (BTEX) using EPA Method 5030/8020; and
- Toxicity characteristic leaching procedure (TCLP) using EPA Method 1311, including ignitability, corrosivity, and reactivity by SW846 7.3.3.2.

Table 4-2 of the FSP presents each parameter, matrix, preparation method, and analytical method. The preparation and analytical methods referenced are standard EPA methods from *Test Methods for Evaluating Solid Wastes*, SW-846 (EPA, 2000b).

6.2 Preventative Maintenance

Preventative maintenance procedures, frequencies, etc. are available for each instrument used at CAS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase. Responsibility for ensuring that routine maintenance is performed lies with the laboratory director.

All instruments are operated and maintained according to the instrument operating manuals and technical SOPs. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at CAS contain all relevant information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on each instrument used at CAS before sample analyses may proceed. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can continue.

6.3 Calibration Procedures

Calibration of instruments and support equipment ensures that the analytical system is operating and functioning correctly. Before samples are analyzed, chemical calibration standards of each target analyte are analyzed to establish that the instrument is functioning properly with the desired sensitivity. Calibration procedures are described in Section 11.0, *Calibration Procedures and Frequency*, of the LQAM. All equipment and instruments used at CAS are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as according to criteria set forth in the applicable analytical methodology. Trained personnel perform operation and calibration. Documentation of calibration information is maintained in appropriate reference files.

6.4 Laboratory Quality Control Procedures

Laboratory QC procedures are used to monitor the laboratory performance by analyzing a set of control samples to generate precision and bias (accuracy) data that is compared to the method MQO criteria or range. This information in combination with method blank data is used to assess laboratory performance and to determine the effect the sample matrix has on the data.

6.4.1 Analytical Batch Quality Control

The basic unit for analytical quality control is the batch. The two types of batches in the laboratory are the preparation and the analytical batch. Samples of the same matrix are extracted in batches not to exceed twenty samples, and the analytical batch is composed of no more than twenty samples. The preparation batches are usually analyzed as a group. Each sample batch includes the appropriate number and type of blanks, quality control samples, and project analytical samples. All calibrations and QC samples analyzed are identified and traceable to the unique sample preparation batch and specific sample analytical batch. All the samples in an analytical batch (field samples and quality control samples) are to be handled and processed the same way, and all of the data from each analysis is to be handled the same way.

6.4.2 Matrix-Specific Quality Control

Matrix-specific QC is based on the analysis of a sample to generate precision and accuracy data from the analysis of method blanks, surrogates, matrix spikes, and matrix spike duplicates that are compared to the method MQOs. The MQOs are denoted as QC sample acceptance limits and ranges. This information and the field blank data are used to determine the effect the sample matrix and the field conditions have on the analytical data. Deviations from the MQO target ranges precipitate the implementation of appropriate corrective actions and a review of the usability of the data.

6.4.3 Laboratory Internal Quality Control Checks

The laboratory internal quality control checks evaluate the method control (batch QC), and the effect of the sample matrix (matrix-specific QC). The overall level of laboratory QC will conform to the minimum required QC protocols of the standard EPA methods, as amended by the specific procedures developed by the laboratory for these methods.

The laboratory method-specific SOPs will define the types of QC checks required (i.e., laboratory control samples (LCS), method blanks, matrix-spike/matrix-spike duplicate (MS/MSD), post digestion spikes, calibration standards, internal standards, surrogate standards, specific calibration check standards, sample dilutions, and/or laboratory duplicate analysis). Laboratory method-specific SOPs also will define the frequency of each QC analysis, the analytes and reference concentrations to be used as controls, and the QC acceptance criteria. The general types of laboratory internal QC checks are summarized in the following underlined subsections.

<u>Laboratory Control Sample</u>

The LCS is an interference free matrix spiked with known quantities of the analytes of interest or specific compounds. The LCS measures the accuracy of the analytical method by measuring the recovery of the spiked compounds. The laboratory control sample duplicate (LCSD) is an aliquote of the LCS. The results of a paired LCS/LCSD will provide precision information for the batch.

Acceptance criteria for LCS analyses are based on EPA methods. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

<u>Matrix Spike/Matrix Spike Duplicates</u>

MS/MSD samples are two aliquot of the same environmental sample created in the laboratory by adding known concentrations of target analytes or specific compounds into each aliquot immediately before extraction or analysis. MS/MSD samples provide information on matrix interferences encountered during extraction, digestion, and analysis (i.e., suppression or enhancement of instrument signal levels). MS samples are principally used to evaluate accuracy. When the MS samples are used together with an MSD sample, they yield information on the analytical precision.

For the appropriate methods, matrix spiked samples are prepared and analyzed at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per 20 samples. A sample identified as field blank, equipment blank, or trip blank is not to be matrix spiked.

Method Blanks

Method blanks are contaminant-free reagent water or sand samples spiked with all reagents, surrogates, and internal standards carried through the entire analytical procedure. Method blanks are used to detect system bias introduced in the laboratory. A method blank should have a value below the reporting limit of the constituents of concern. A method blank is included with the analysis of every analytical batch, every 20 samples, or as stated in the method, whichever is more frequent.

Surrogate Spikes

Surrogate spikes are prepared in the laboratory by adding a known amount of analytes, chemically similar to the target analytes, to every blank, sample extract, matrix spike, matrix spike duplicate and standard. Surrogate spike results are used to evaluate analytical efficiency and matrix interferences.

6.5 Performance and System Audits

QC audits are part of the CAS QA program. There are two types of audits used at the facility. System Audits are conducted to qualitatively evaluate the operational details of the field and laboratory QA program. Performance Audits are conducted by analyzing performance evaluation samples in order to quantitatively evaluate the outputs of the various measurement systems.

6.6 Performance Evaluation Samples

PE samples are commercial samples of the applicable matrix containing target analytes. PE samples may contain components that cause known interferences when the target analytes are measured. PE samples are used to evaluate the performance of the laboratory for specific parameter and matrix.

7.0 CORRECTIVE ACTION

Actual or potential system problems are detected through calibration check samples, QC samples, daily performance audits, and QA audits. These problems and possible solutions are immediately discussed with laboratory management personnel.

7.1 Laboratory Situations

The need for corrective action resulting from evaluation of QA/QC results will be initiated by the ECC QCM in consultation with the project QCM. Corrective action may include, but is not limited to:

- Re-analyzing the samples;
- Evaluating and amending sampling and analytical procedures;
- Accepting data with an acknowledged level of uncertainty; and
- Resampling and analysis, if the completeness of the data set or intended use of the data are recognized during a preliminary review to be insufficient to meet program DQOs.

If the above corrective actions are deemed unacceptable, an alternate laboratory may be selected to perform necessary or appropriate verification analyses. The Navy will be notified in writing of any changes in sampling or analytical protocol prior to implementation. Written acceptance by the Navy of any changes in sampling or analytical protocol will be kept in the project files.

7.2 Immediate Corrective Action

Any equipment and instrument malfunctions will require immediate corrective actions. The laboratory QC charts are working tools that identify appropriate immediate corrective actions to be taken when a control limit is exceeded. The actions taken should be noted in field or laboratory logbooks, but no other formal documentation is required unless further corrective action is necessary. These on-the-spot corrective actions will be applied daily by laboratory analysts as necessary.

7.3 Long-Term Corrective Action

The need for long-term corrective action may be identified by standard QC procedures, control charts, and/or performance or system audits. Any quality problem that cannot be solved by immediate corrective action falls into the long-term category.

The essential steps in a long-term corrective action system are:

- Identification and definition of the problem;
- Investigation and determination of the cause of the problem;
- Determination and implementation of a corrective action to eliminate the problem; and
- Verification that the corrective action has eliminated the problem.

Documentation of the problem is important in corrective action. The responsible person may be a laboratory analyst, the laboratory project manager (PM), the laboratory QAM or Director, the ECC QAM, the QCM, the CQAO or the PM. In general, the QCM will investigate the situation and determine who will be responsible for implementing the corrective action. For field activities, the required corrective action will be documented by the QCM. For chemical data, the required corrective action will be documented by the QCM or other designated personnel. The corrective action will be discussed with the Navy prior to implementation if the severity of the problem warrants such discussion. The QCM will verify that the corrective action was taken and appears effective.

7.4 Out-of-Control Situations

A value outside the control limits or classified as outlier by statistical testing is considered an out-of-control situation. Failure to meet calibration criteria, record keeping omissions, improper sampling technique, and improper storage or preservation of samples are all conditions that affect data quality and require investigation and correction. Immediate action is taken to find the problem, recalibrate, and re-analyze the samples.

7.5 Laboratory Corrective Procedures

When an out-of-control situation is detected, the laboratory analyst will investigate to determine the cause and document the actions taken. Data acquired concurrently with this condition are discarded and samples re-analyzed unless the investigation of the problem proves that the analysis was in control.

Corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. After the corrective actions are instituted, the systems performance is rigorously checked before continuing sample analysis. No analysis is started if the calibration check samples are outside of the EPA limits. The problem is diagnosed, the system fixed, and the calibration rechecked before analysis is resumed. Corrective actions associated with the project are documented and records are maintained in the laboratory maintenance book. If more than 10% of the QC values associated with a sample determination are unacceptable, the QCM will notify the Navy.

8.0 DATA REDUCTION/CALCULATIONS OF DATA QUALITY INDICATORS

The following sections discuss the data reduction procedures performed by the laboratory and the calculations of the DQIs of precision, bias, sensitivity, and completeness from the project data.

8.1 Data Reduction

Data reduction will be performed according to standard mathematical and/or statistical procedures. The laboratory will provide the analytical data as a hard copy and in an electronic format. The QCM or a designated third party will perform calculations, data evaluation, and QC review.

The electronic deliverable will be used to store data and to perform data manipulations (e.g., sorting, statistical tests), as required to interpret data, evaluate their quality, and provide data reports. The following parameters will be evaluated during this review:

- Comparing of analyses requested on the Chain-of-Custody Record with the analyses actually performed by the laboratory;
- Checking syntax to assess whether the correct types of data are in the appropriate fields (e.g., that the chemical data field contains data information rather than method names);
- Checking whether the data records provided by the laboratory contain the correct minimum entries (e.g., date of analysis, field sample number, sample delivery group (SDG) number, and test methods);
- Checking correctness of Chemical Abstract Service numbers in the database;
- Checking preparation and test method codes against a pre-approved list; and
- Checking laboratory QC sample codes against a pre-approved list.

8.2 Calculation of Data Quality Indicators

Data assessment procedures will be used by the QCM (or designated representative) for statistically assessing duplicate and external spike samples and checking field blank samples that are submitted blind to the analytical laboratories from the field and generated internally by the laboratories in accordance with this QAPP. Field blank and field duplicate samples are submitted blind to ensure that these QC samples are treated the same as other samples and not subjected to special treatment. In addition, all sample results will be evaluated to determine if reporting detection limit goals (e.g., analytical sensitivity) were met and if test sample dilutions were justified. The purpose of implementing these procedures is to verify that the chemical data generated during the investigation are of acceptable precision, bias, sensitivity, and completeness and are representative of site conditions.

8.3 Assessment of the Data Quality Indicators

The goals of the assessment of DQIs are the following:

- Determine site-specific DQIs;
- Use DQI results to identify the limits of data usability; and
- Evaluate the data limitations in achieving the program DQOs.

Chemical data derived from the investigation will be evaluated based on DQI assessment for both the laboratory analytical and field sample collection programs. To address these issues, a combination of qualitative evaluations and comparisons to project QA objectives will be used to check the quality of the chemical data. A quantitative evaluation will be made of precision, bias, and completeness.

A qualitative evaluation will be made of representativeness and comparability based on assessment of precision, bias, and completeness. The results of the evaluations will not be used to eliminate data. Comparisons of internal laboratory QC samples to project QA goals (e.g., precision and accuracy goals specified by the methods and this QAPP) will be used to validate the analytical procedures used by each laboratory. Comparisons of field QC samples to project QA goals will be used to examine the overall effects of the field sampling and handling procedures.

Laboratory and field QC samples will be used in combination to assess overall DQI parameters. DQI parameters will be used to determine if DQOs for the project were met. Contract compliance and performance will be assessed by comparing QA/QC results to the method-specific checklists and QC goals specified in this QAPP. If problems arise and the data are found to deviate from previous analyses or surrounding conditions, the data will be annotated. No changes will be made to the QC acceptability criteria without prior approval of the Navy representative.

Chemical data that do not meet relevant QC criteria will be labeled appropriately with data qualifiers. Data qualifier codes to be used are based EPA National Functional Guidelines for Chlorinated Dioxin/Furan Data review (EPA, 2002). The qualifiers are used during the data review process to indicate to what degree the sample data conform to QC requirements. Three types of control samples will be evaluated: spikes, blanks, and duplicates. The definitions of these types of samples can be found in Section 6.4.3 of this QAPP. For those review items applicable, data assessment will be performed. The following sections present procedures for evaluating the DQI of data derived from the investigation.

8.3.1 Precision

Precision for sample data will be calculated by evaluating data from field duplicate samples, LCS/LCSD, and MS/MSD duplicate samples, as follows:

- Tabulate duplicate data and calculate the absolute value difference, average, and Relative Percent Difference (RPD) as shown below for each duplicate pair:

$$RPD = \frac{(x_1 - x_2)}{\bar{x}} * 100$$

Where:

x_1 = concentration for Sample 1 of duplicate

x_2 = concentration for Sample 2 of duplicate

$\bar{x}$ = mean of Samples 1 and 2

RPDs will not be calculated in cases where one analyte of the duplicate pair was reported as a non-detect;

- Identify duplicates that exceed the project (method) precision goals; and
- Qualitatively evaluate the significance of data that fall outside the project goals for precision.

Usability of data outside of project goals for precision is dependent on the degree of QC exceedance, whether or not there is a potential high or low sample result bias, whether or not associated sample results are significant compared to action levels, and if the sample is critical to the investigative findings. If data quality problems arise, the analytical laboratory will be notified for corrective action, as appropriate. Data will not be removed from the database solely as a result of these procedures and QC samples will not be used to alter or correct analytical data. Instead, data will be flagged with appropriate notation.

8.3.2 Bias

Bias is the systematic or persistent error of a measurement process in one direction, either above or below the true value or mean. While bias is related to accuracy, the terms are not interchangeable. Accuracy is the degree of agreement of a measurement with an accepted reference or true value. Bias assessments are usually based on the analysis of spiked materials or samples, such as LCS, MS/MSD, and surrogates. Generally, bias values are expressed as percent recovery.

Percent recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{(x_s - x_u)}{k} * 100$$

Where:

x_s = measured value of spiked sample
x_u = concentration in the unspiked sample
k = known amount added to sample

When %R is calculated for LCS or other reference materials, x_u, could be set at zero.

The percent bias (%B) is related to the percent recovery as follows:

$$\% B = \%R - 100$$

8.3.3 Sensitivity

The term sensitivity is used to describe the project method detection/quantitation/reporting limits established to meet project-specific DQOs. The method detection limits (MDLs) are defined as the minimum concentration of a substance that can be reported with 99% confidence that the substance concentration is greater than zero. Due to the significant amount of error associated with results calculated at the MDL, the method quantitation limit (MQL) is determined as five to ten times the MDL for the majority of target analytes, but no lower than three times the MDL for any target analyte. The reporting limits (RL) are based on project-specific concentrations of concern, regulatory action levels, or sensitivity capability of the method and instrument. This is the threshold value below which the laboratory reports a result as non-detect (U); presented as "less than the RL value". Reporting limits are at least two times the MDL, but no greater than ten times the MDL.

8.3.4 Completeness

The completeness goal for this project is 90 percent. Overall completeness of the sample data collected will be calculated according to the following equation:

$$\text{Completeness (C)} = \frac{V}{T} \times 100\%$$

Where:

C = percent completeness of analytical effort
V = amount of valid data obtained
T = amount of samples collected and analyzed

Completeness calculations will use validated data. Data that are rejected by external validation processes will be counted against completeness criteria. QC parameters evaluated to assess completeness include holding times, surrogates, laboratory and field duplicates, RPD, MS/MSD for percent recovery and RPD, and LCS for percent recovery. Samples results that do not meet relevant QC criteria due to substantiated matrix effects, and/or are re-analyzed past holding time due to QC corrective action and/or are qualified as estimated (J) because the sample results are below the reporting detection limit, will be considered usable and will not count against the completeness assessment.

9.0 LABORATORY OPERATIONS DOCUMENTATION

The following sections present the laboratory data reporting procedures for the project, including the report content, schedule, data archival procedures, and records retention requirements.

9.1 Data Reduction and Data Review

This section presents the laboratory data reduction and review procedures. A description of the levels of review that data receives and data custody procedures and specific review procedures and criteria are presented.

All data is initially reviewed and processed by analysts using appropriate methods (e.g. chromatographic software, instrument printouts, hand calculation, etc.). A file of all raw data is printed, reviewed for completeness and quality criteria against an in-house checklist and signed by the analyst. The operations manager reviews all reported data against the raw data validating completeness and quality. The final report data package is reviewed by the project manager for compliance with previously established project requirements.

Assessment of the analytical data includes evaluation of data consistency as follows:

- Comparability of duplicate analyses;
- Comparability of previous data from the same sampling location (if available);
- Adherence to accuracy and precision control limits; and
- Investigation of anomalous low or high parameter values.

The results of the review are discussed with either the departmental supervisor or lab director for resolution prior to final release of the package.

After the data has been checked for accuracy and acceptability, the laboratory director or QA coordinator reviews the final report and raw data for acceptability and signs the report. Subsequently, the report is distributed.

9.2 Data Reporting Procedures

The laboratory will provide a Contract Laboratory Program (CLP)-like data package, including all appropriate summary forms necessary for data validation.

9.3 Data Management Procedures

Data management procedures describing the control of raw data through the archiving procedures are presented in the following sections.

9.3.1 Raw Data Management

The raw data is filed for approximately one year. After the one year period, the data is archived. All hard copy and electronic backups are archived in a secured file room for a period of at least five years from the date of the final report.

9.3.2 Data Archival and Retention Requirements

Data will be accurately transferred from the analytical workstation to archive media. Protocols and procedures will be in place to provide custody tracking of the files and to ensure the quality of the archived data. All archived material will be maintained for a minimum of five years.

10.0 DATA ASSESSMENT PROCEDURES

Data assessment addresses the activities that are performed after the completion of the collection phase of the project to ensure the validity of the data. Implementation of the processes outlined in the following subsections will determine whether the project data is scientifically sound data of known and documented quality that will satisfy the project objectives.

10.1 Data Quality Control Review

Data review, as a function of the contract laboratory, is an essential tool to ensure the validity of the reported data prior to the submittal of the data. The data review is an evaluation of the data generation, reduction and a minimum of three levels of documented review.

Each level of the review process requires the evaluation of data quality based on the results of the QC data and the professional judgment of the reviewer. Each review will be documented using an appropriate checklist signed and dated by the reviewer.

Level I – Consists of a review of the quality of the analytical work performed by the analyst who performed the test and has documented training in the method and the laboratory standard analytical requirements. At a minimum, this review ensures that:

- Sample preparation, analytical results and information is correct;
- Appropriate SOPs were followed;
- QC samples are within established limits;
- Data transfers were verified; and
- Documentation is correct and complete.

Level II – Consists of a technical review performed by personnel who have documented training for the method and laboratory standard analytical requirements. The purpose of this review is to provide an independent, complete peer review of the analytical batch data package. Level II review requires the review of all calibration data, QC sample results, and analytical results back to the raw data.

Level III – Consists of total overview of the data package performed by a QA/QC officer, supervisor, or other laboratory designee with documented training on third level review. Level III review includes the following:

- Spot check of raw data;
- Review of manual integration and manual calculations;
- Review of the sample receipt information; and
- Final report verification to ensure compliance with project-specific requirements.

Any errors will be corrected and documented. The Level III reviewer is responsible for final approval of the applicable LIMS. Each level of review will be performed by a different individual.

In addition to the three level review, the QA department will review the reporting process for at least 10% of the data produced by the laboratory.

10.2 Data Verification/Validation

Data verification is the process of evaluating the data presented in the data package for completeness and the laboratory QC samples to determine whether the data are of acceptable quality. Data validation is an analyte and sample-specific process of data assessment performed in accordance with EPA national functional guidelines or project-specific guidelines to determine the analytical quality of a data set. Data validation includes assessment of the raw data package from the laboratory. Data validation criteria are based on the project MQOs.

Any problems with the data, discovered during the evaluation process, will be discussed with the laboratory QC manager and the Navy project chemist and corrected as determined by the Navy.

The data assessment for the Remedial Action at the Site 8 NCBC will be performed in accordance with the EPA National Functional Guidelines for Chlorinated Dioxin/Furan Data review (EPA, 2002). One hundred percent of the project data will undergo data verification.

10.3 Data Quality Objective Reconciliation

Data assessment will provide documentation of the quality and usability of the data. Data qualifiers will be applied as appropriate to alert the data user of deficiencies in the data. The data validator will assign data qualifiers during the validation process. The qualifiers may be different depending on the type of data evaluation performed. Data validation by EPA functional guidelines may require different qualification criteria than a project-specific guidance. Qualifiers will be used to qualify the usability of the data, usually due to laboratory QC failures.

10.4 Project Completeness Assessment

Project completeness will be calculated according to the analytical completeness and field completeness goals as required by the project.

Utilization of the QAPP requires implementation of procedures for obtaining and evaluating data in a manner that will result in a quantitative and qualitative representation of the DQI parameters. The parameters of precision, bias, and completeness provide a quantitative measure of the statistical significance of the data collected in the field program. The parameters of representativeness and comparability utilize documentation of the field and laboratory procedures to qualitatively evaluate the data. Section 8.0 of this QAPP defines the DQIs and describes how the DQIs will be calculated. Following the collection and analysis of the samples, a determination will be made whether the DQOs established for this project were satisfied.

11.0 REFERENCES

U. S. Environmental Protection Agency (EPA), 2000a. Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/G-4 HW. January, 2000.

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EPA, 2000c. Guidance for Data Quality Assessment - Practical Methods for Data Analysis, EPA QA/G-9. July, 2000.

EPA, 2001a. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5. March, 2001.

EPA, 2001b. Data Quality Objectives for Quality Assurance Project Plans, EPA QA/R-5. March, 2001.

EPA, 2002. EPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan DataReview, EPA-540-R-02-003. August, 2002.

TABLES

Table 4-1
Measurement Quality Objectives and Data Quality Indicators
Site 8 – Herbicide Orange Storage Area and Off-Base Area of Contamination
Naval Construction Battalion Center
Gulfport, Mississippi

Analyte	Acceptance Criteria		Data Quality Indicators				
	LCS	MS/MSD	Precision (RPD)	Bias	Representativeness	Completeness	Comparability
2,3,7,8-Tetrachlorodibenzo-p-dioxin	Within laboratory established limits	Within laboratory established limits	Calculated by evaluating duplicate pairs: The RPD will be calculated by dividing the difference by the average of the duplicate pairs For 2,3,7,8-tetrachlorodibenzo-p-dioxin the goal is 30%	Calculate using spiked samples: The percent recovery is calculated by dividing the difference of and original concentration by the spike concentration added. For 2,3,7,8-tetrachlorodibenzo-p-dioxin the goal is 70-130%	Quantitatively assessed using duplicates. Qualitatively assessed by determining if the QAPP sample design and analytical procedures were followed.	Amount of valid data divided by the amount of expected valid data times 100. Sampling goal is 95% Analytical data goal is 90% Overall project completeness goal is 90%	Qualitative assessment of analytical techniques, data quality, and sampling design: Sampling data set is compared to each additional data set.

Notes:

QAPP: Quality Assurance Project Plan
% Rec: Percent recovery
MS/MSD: Matrix spike/matrix spike duplicate
LCS: Laboratory control sample
RPD: Relative percent difference

APPENDIX A

QUALITY ASSURANCE MANUAL

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July 22, 2003

APPROVED BY

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Appendices

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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is a professional consulting laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sediment, sludge, tissue, industrial and hazardous waste, and other material.

It is a policy at CAS that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

The information in this document has been organized according to the format described in *National Environmental Laboratory Accreditation Conference (NELAC) Quality Systems Standards*, July 1998, and *Interim Guidance for the Preparation of Quality Assurance Project Plans,* QAM-005, USEPA, 1980; and *Guidance on Preparation of Laboratory Quality Assurance Plans*, USEPA, February 14, 1991.

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at CAS is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of CAS:

"The mission of Columbia Analytical Services, Inc., is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset – our people – in a way that encourages professional growth, personal development and company commitment."

In support of this mission, our QA program addresses all aspects of laboratory operations, including laboratory organization and personnel, standard operating procedures, sample management, sample and quality control data, calibration practices, standards traceability data, equipment maintenance records, method proficiency data (such as method detection limit studies and control charts), document control/storage and staff training records.

4.1 Facilities and Equipment

CAS/Houston features 6,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each workspace. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types.

These specialized areas include:

- Sample Management and Shipping & Receiving areas
- Wet Laboratory for sample preparation
- HRGC-HRMS/HRGC-LRMS Laboratory
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation is appropriate for the procedures in use. Appendix A lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.2 Technical Elements of the Quality Assurance Program

4.2.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks

CAS maintains SOPs for use in both technical and administrative functions. Included in the list of available SOPs are procedures for the preparation of an SOP document, and for enforcing the control of documents through the laboratory (ADM-SOP & ADM-DOCCTRL, respectively.) The Laboratory Director and the QA Manager have reviewed each SOP generated in the laboratory. The document control process associated with a SOP ensures that only the most currently prepared version of a SOP is being used for guidance and instruction. In addition to SOPs, the Laboratory Director maintains a current file of all the promulgated methodology used to perform analyses. This file is accessible to all laboratory staff regardless of discipline. Laboratory notebook entries have been standardized following the guidelines in the Making Entries into Logbooks and onto Bench sheets SOP (SOP No. ADM-DATANTRY.) The entries made into laboratory notebooks are peer reviewed and approved by a second analyst.

4.2.2 Standard Reference Materials

All analytical measurements generated at CAS are performed using materials and/or processes that are traceable to a Standard Reference Material (SRM). Metrology equipment (analytical balances, thermometers, etc.) is calibrated using SRMs traceable to the National Institute of Standards and Technology (NIST). Consumable SRMs routinely purchased by the laboratories (e.g. primary stock standards) are purchased from nationally recognized, reputable vendors. Most vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. Traceability throughout the laboratory is accomplished by following the guidelines set in the SOP, ADM-SPSR (Documentation of Standard Preparation and Receipt). All sampling containers provided to the client by the laboratory are purchased as pre-cleaned containers, with certificates of analysis available for each bottle type. Certifications of Analysis provided by the vendors of reference materials and bottles are kept on file by the laboratory.

4.2.3 Operational Assessments

There are a number of methods used to assess the laboratory and its daily operations. In addition to the routine quality control (QC) measurements used by a laboratory to measure quality, the senior laboratory management staff at CAS examine a number of other performance indicators to more accurately assess the overall ability of the laboratory to successfully perform analyses for its clients. On-time performance, Analytical Report defect rate and Customer Invoice defect rate are a few of the measurements performed at CAS that are used to assess performance from an external perspective (i.e. client satisfaction). A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload. CAS utilizes a

number of different methods to insure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results.

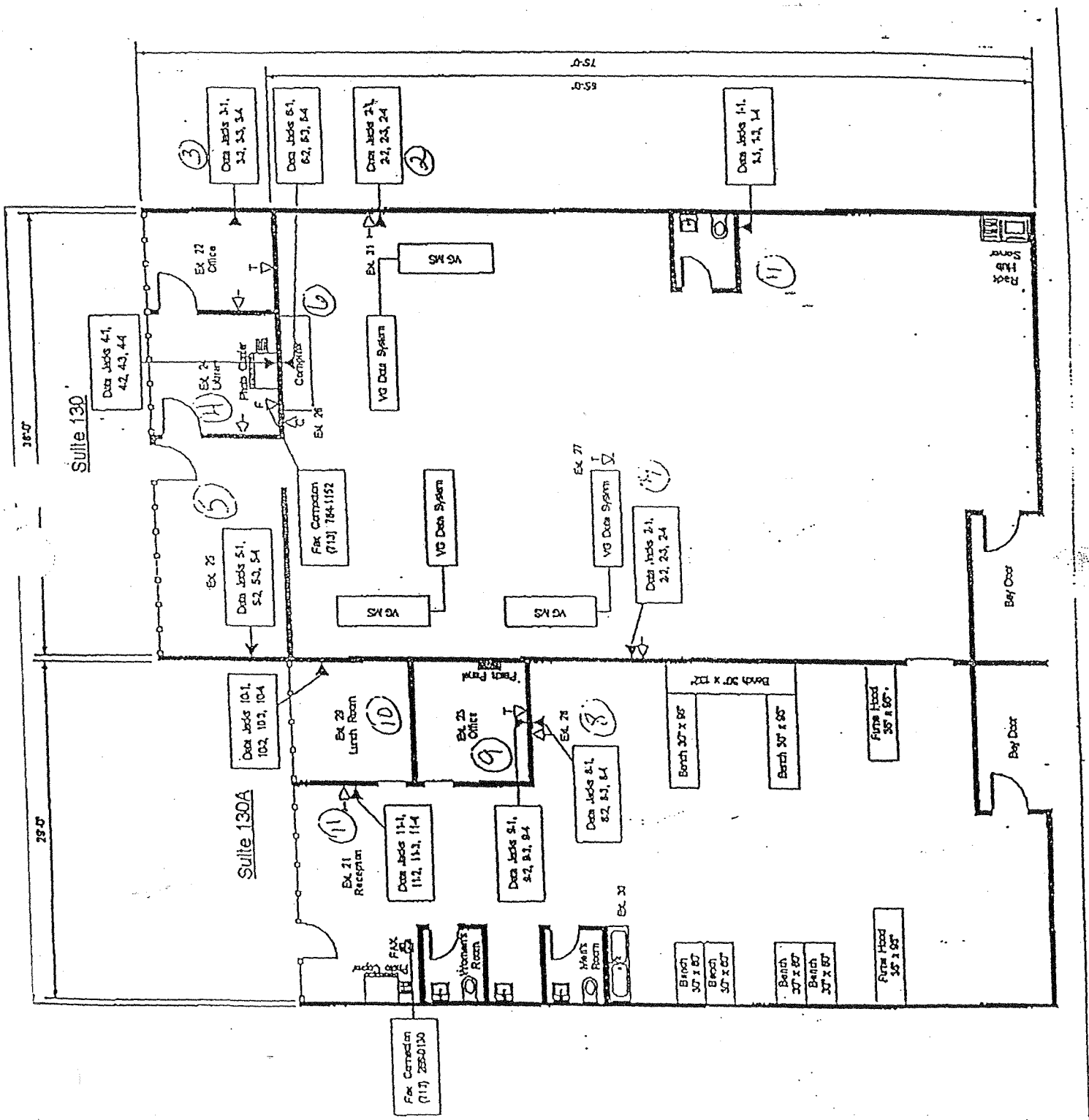
4.2.4 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP, the project chemist handling that project must discuss the proposed deviation with the laboratory director to obtain approval for the deviation. A detailed description of the deviation should be attached to the quotation in the project file and the service request upon logging in the samples.

4.3 Subcontracting

Analytical services are subcontracted when CAS/Houston needs to balance workload and/or when CAS/Houston does not perform the requested analyses. However, subcontracting is only done with the knowledge and approval of the client. Subcontracting to another CAS laboratory is preferred over external-laboratory subcontracting. Further, subcontracting is done to capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the SOP for Qualification of Subcontract Laboratories Outside of CAS Network (ADM-SUBLAB).

Figure 4-1
CAS/Houston Laboratory Floor Plan



5.0 STATEMENT OF PROFESSIONAL CONDUCT AND LABORATORY PRACTICE

One of the most important aspects of the success of CAS as a company is the emphasis placed on the integrity of the data that are provided and the services that are performed. To promote product quality, CAS requires certain standards of conduct and ethical performance among our employees. The following examples of documented CAS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.

Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.

Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.

Unauthorized release of confidential information about the company or its clients is taken very seriously and is subject to formal disciplinary action.

Each new employee receives eight hours of training in Ethical Conduct. Legal responsibilities, including potential penalties and punishments, are included in this training course. All employees receive one hour of additional training in Ethical Conduct once each quarter.

CAS considers the hard-copy report to be the official Certificate of Analysis. We maintain the same high level of confidentiality with our electronic data deliverables that we maintain with the hard-copy. Our data confidentiality procedures are the same whether the data is presented on paper or electronically.

6.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The CAS/Houston staff, consisting of less than ten employees, includes chemists and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that a modern, state-of-the-art analytical laboratory requires.

CAS is committed to providing an environment that encourages excellence. Everyone within CAS shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B. Specific job descriptions are available and kept on file by Human Resources.

- The role of the ***Laboratory Director*** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Houston facility. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

  The Laboratory Director is also responsible for identifying and responding to QA needs, problems and requests from the staff. This person is a technical advisor and is responsible for summarizing and reporting overall unit performance, including round-robin programs, certification and accreditation activities, and blind and reference sample analysis.

- The responsibility of the ***Quality Assurance Manager*** (QAM) is to oversee implementation of the quality program and to coordinate overall QA activities within the laboratory. The QAM works with individual laboratory production units to establish effective quality control and assessment plans. The QAM is also responsible for maintaining the QA Manual and performing an annual review of it, updating it if necessary; reviewing, approving and controlling SOPs, coordinating PE sample analyses and preparing corrective action reports for any missed PE sample results (Section 15.0); reviewing data (Section 12.0); maintaining the laboratory's certifications and approvals (Section 13.0); performing internal QA audits (Section 13.0); preparing QA reports (Section 16.0); etc.

- The ***Quality Assurance Director*** is responsible for the overall QA program at all the CAS laboratories. The QA Director is responsible for performing an annual on-site audit at each CAS laboratory and preparing a written report; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of CAS-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing an annual QA activity report; etc.

The QA Director is also responsible for identifying and responding to organizational QA needs, problems and requests from the technical staff. This person is a technical advisor and is responsible for summarizing and reporting overall CAS performance, including round-robin programs, certification and accreditation activities and blind and reference sample analysis.

- The ***Health and Safety Officer*** is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer
- The ***Sample Management Office*** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.
- The ***Client Services Manager*** is responsible for the customer services, project managers and business development functions. Client Services provides the direct communication with clients from initial project specifications to final deliverables products.
- The ***Project Manager*** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project manager is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the CAS laboratory and administrative staff to ensure that client-specific needs are understood, and that the services CAS provides are properly executed and satisfy the requirements of the client.

  Analytical work will be conducted by the laboraotry under the approval of the client. If any aspect of a project requires subcontracting, CAS project managers will notify the client and obtain approval for any subcontractors prior to beginning the analytical work.
- The ***Analytical Laboratory*** is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within that department's responsibilities. Each analyst in the laboratory has the responsibility to carry out testing according to prescribed methods, standard operating procedures and quality control guidelines particular to the laboratory in which he/she is working.
- ***Information Technology*** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, education of analytical staff in the use of scientific software, software development and implementation, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

7.0 SAMPLING, SAMPLE PRESERVATION, AND HANDLING PROCEDURES

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. CAS recommends that clients follow sampling guidelines described in ***40 CFR 136, USEPA SW-846***, and state-specific sampling guidelines, if applicable. Sample handling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

CAS uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010 and Supplement I, 600/4-82-057, 600/R-93/100, 600/4-88-039 and Supplements I and II, and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. The complete citation for each of these references can be found in Section 18.0 of this document. The container, preservation and holding time information is summarized in Table 7-1.

CAS routinely provides sample containers with appropriate preservatives for our clients. The containers are purchased as "precleaned" to a level 1 status, and conform to the requirements for analytical sample established by the USEPA. Certificates of analysis for the sampling containers are available to clients if requested. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (decontaminated inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. An example of a sample container label and a custody seal is shown in Figure 7-1. Figure 7-2 is a copy of the chain-of-custody form routinely used at CAS. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to CAS. The proper preservative will be always be added to the sample containers prior to shipment, unless otherwise instructed by the client. If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. CAS keeps client-specific shipping requirements

on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. CAS also provides its own courier service that makes trips to the Greater Houston area upon client request.

When environmental samples are shipped by CAS to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures for maintaining the chain of custody of the sample(s) (*Standard Operating Procedure for Chain of Custody for Sample Transfer between Laboratories* [SOP No. ADM - COC]), proper packaging and shipment, specification of proper methodology, etc. Blue or gel ice is the only temperature preservative used by CAS, unless otherwise specified by the client or receiving laboratory.

Table 7-1
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Fecal and Total	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity	W	P,G	Cool, 4°C	14 days
Alkalinity	W	P,G	Cool, 4°C	14 days
Ammonia	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (BOD)	W	P,G	Cool, 4°C	48 hours
Bromide	W	P,G	None Required	28 days
Chemical Oxygen Demand (COD)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride	W	P,G	None Required	28 days
Chlorine, Total Residual	W	P,G	None Required	24 hours
Color	W	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	W	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Fluoride	W	P,G	None Required	28 days
Hardness	W	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH)	W	P,G	None Required	24 hours
Kjeldahl and Organic Nitrogen	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrate	W	P,G	Cool, 4°C	48 hours
Nitrate-Nitrite	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite	W	P,G	Cool, 4°C	48 hours
Orthophosphate	W	P,G	Filter Immediately, Cool, 4°C	48 hours
Oxygen, Dissolved (Probe)	W	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Phenolics, Total	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Elemental	W	G Only	Cool, 4°C	48 hours
Phosphorus, Total	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	W	P,G	Cool, 4°C	7 days
Residue, Settleable	W	P,G	Cool, 4°C	48 hours
Residue, Volatile	W	P,G	Cool, 4°C	7 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Silica	W	P Only	Cool, 4°C	28 days
Specific Conductance	W	P,G	Cool, 4°C	28 days
Sulfate	W	P,G	Cool, 4°C	28 days
Sulfide	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite	W	P,G	None Required	24 hours
Surfactants (MBAS)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin	W	P,G	Cool, 4°C	28 days
Temperature	W	P,G	None Required	Analyze immediately
Turbidity	W	P,G	Cool, 4°C	48 hours
Metals				
Chromium VI	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
	S	P,G	Cool, 4°C	28 days
Metals, except Chromium VI and Mercury	W	P,G	HNO_3 to pH<2	6 months
	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Organic Tests				
Oil and Grease	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (TOC)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (TOX)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (AOX)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total Recoverable	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	28 days
Petroleum Hydrocarbons, Total	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Purgeable Halocarbons	W	G, Teflon-Lined Septum Cap	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C, No Headspace	14 days
	S	G, Teflon-Lined Cap, or 5035[j]	Cool, 4°C, Minimize Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE)	W	G, Teflon-Lined Septum Cap	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C, No Headspace	14 days
	S	G, Teflon-Lined Cap, or 5035[j]	Cool, 4°C, Minimize Headspace	14 days
Acrolein, Acrylonitrile, Acetonitrile	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
EDB and DBCP	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days
Alcohols and Glycols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phenols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phthalate Esters	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrosamines	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Nitroaromatics and Cyclic Ketones	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Polynuclear Aromatic Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Haloethers	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
High Resolution Dioxins/Furans Low-Resolution Dioxins/Furans	**W,S**	**G, Teflon-lined Cap**	**Cool, 4°C[g]**	**30 days until extraction; 45 days after extraction**
High Resolution Dioxins/Furans Low-Resolution Dioxins/Furans	**tissue**	**G, Teflon-lined Cap**	**Cool, -20°C[g,k]**	**30 days until extraction; 45 days after extraction**
Resin and Fatty Acids	W	G, Teflon-Lined Cap	NaOH to pH ≥10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction
Semivolatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Contract Laboratory Program (CLP)				
Cyanide, Total and Amenable to Chlorination	W	P,G	Cool, 4°C, NaOH to pH 12, plus 0.6 g Ascorbic Acid	12 days[h]
	S	P,G	Cool, 4°C	12 days[h]
Mercury	W	P,G	HNO_3 to pH<2	26 days[h]
	S	P,G	Cool, 4°C	26 days[h]
Metals, except Mercury	W	P,G	HNO3 to pH<2	6 months[h]
	S	P,G	Cool, 4°C	6 months[h]
Volatile Organics	W	G, Teflon-Lined Cap	HCl to pH <2, Cool, 4°C, Minimize Headspace	10 days[h]
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	10 days[h]
Semivolatile Organics	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	5 days until extraction;[h,i] 40 days after extraction
Organochlorine Pesticides and PCBs	W,S	G, Teflon-Lined Cap	Cool, 4°C	5 days until extraction;[h,i] 40 days after extraction

a See Section 18.0 for sources of holding time information.
b W = Water; S = Soil or Sediment; HW = Hazardous Waste; T = Tissue
c P = Polyethylene; G = Glass
d For chlorinated water samples
e The recommended maximum holding time is variable, and is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

f Fourteen days until extraction for soil, sediment, and sludge samples
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate
h Number of days following sample receipt at the laboratory
i Ten days until extraction for soil, sediment, and sludge samples
j For EPA Method 5035, refer to the method for details on sampling and preservation
k Fish and adipose tissue samples must be stored at -20^0C in the dark

Figure 7-1
Sample Container Label and Custody Seal

COLUMBIA ANALYTICAL SERVICES, INC.

Project Name: _____

Sample Name: _____

Date_____ Time_____ Sampler _____

Analysis _____

_____

_____

Preservative: _____

Comments:

Sample Container Label

Custody Seal No Nº 005545

Date_____ Project _____

Signature_____ Container#_____ of _____

8540 Baycenter Rd. • Jacksonville, FL 32256 • (904) 739-2277 • 800-695-7222 • FAX (904) 739-2011

Custody Seal

Columbia Analytical Services INC.
An Employee-Owned Company

CHAIN OF CUSTODY/LABORATORY ANALYSIS REPORT FORM

10655 Richmond Ave., Ste 130A • Houston, TX 77042 • (713) 266-1599 • FAX (713) 266-0130

DATE_____ PAGE _____ OF_____

PROJECT NAME_____

PROJECT # _____

COMPANY/ADDRESS _____

_____

_____PHONE_____

REPORT TO: _____

ANALYSIS REQUEST

SAMPLE I.D.	DATE	TIME	SAMPLE MATRIX	NUMBER OF CONTAINERS																			REMARKS

RELINQUISHED BY:

Signature

Printed Name

Firm

Date/Time

RECEIVED BY:

Signature

Printed Name

Firm

Date/Time

TURNAROUND REQUIREMENTS
(in working days)

RUSH .....5 Days

RUSH .....10 Days

STD.....15 DAYS

DELIVERABLES

____ I. Analytical Report

____ II. Analytical Report Plus MS/MSD

____ III. Data Validation Report (includes All Raw Data)

INVOICE INFORMATION:

P.O.# _____

Bill To_____

_____

_____

_____

SAMPLE RECEIPT:

Shipping VIA:_____

Shipping to: _____

Condition: _____

_____

Lab No: _____

RELINQUISHED BY:

Signature

Printed Name

Firm

Date/T

RECEIVED BY:

Signature

Printed Name

Firm

Date/Time

SPECIAL INSTRUCTIONS/COMMENTS:

SAMPLER'S SIGNATURE

Figure 7-2
Chain of Custody Form

8.0 SAMPLE CUSTODY

Standard Operating Procedures have been established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain-of-custody forms, is complete and consistent with the samples received. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Samples delivered to the CAS sample management office (SMO) and are received by a sample custodian. The chain-of-custody (COC) is reviewed for completeness and accuracy and cooler receipt and preservation form (CRPF) (Figure 8-1) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity by the sample custodian includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.):

  Entries should be made in black ink and at a minimum, shall include sample identification, description, date, time and location of sample collection, the name and signature(s) of the sample collector and intermediate sample custodian(s), date and time of each sample transfer, and signature of the CAS sample custodian upon receipt. For an example COC, see Figure 7-2.
- Sample containers checked for integrity (broken, leaking, etc.);
- Sample is clearly marked with the sample ID, date and time of collection;
- Appropriate containers (size, type) are received for the requested analyses;
- Sample container labels and/or tags agree with chain-of-custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (No assessment of proper preservation is performed for VOC containers by SMO personnel).

Any anomalies or discrepancies observed during the initial assessment are recorded on the CRPF and chain-of-custody documents. All potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When a satisfactory resolution has been reached by the project manager and client, the log-in process may commence and analysis may begin. During the log-in process, each sample is given a unique laboratory code and a service request form is generated. The laboratory code consists of an order number and a sample submission number. Each sample is given a consecutive order number the LIMS system based

upon order of log-in. A submission number is assigned to a particular job in the same manner. The submission number is coded with the month and year of log-in as follows:

No. 00070132 = 00 (year/2000)
07 (month/July)
0132 (job number/132nd job logged in July, 2000)

The service request contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. This service request is reviewed by the appropriate project manager for accuracy, completeness, consistency of requested analyses, for client project objectives and COC.

Laboratory internal chain-of-custody documents are generated upon sample receipt so that the internal COCs accompany the samples/extracts to the instrumentation department for analysis. The sample custodian relinquishes custody to an analyst of a particular department for which he/she is responsible to maintain the integrity of the sample/extract. The samples/extracts are then relinquished back to SMO for sample archival prior to disposal.

All samples, with the exclusion of sample extracts, are kept in a refrigerated condition until they undergo anlaysis and disposal. CAS stores samples in one of three walk-in refrigerators. These refrigerators are segregated by method of analysis. The temperature of each storage facility used at CAS is monitored daily and the data recorded in a bound logbook.

Upon completion of all analyses, most aqueous and soil samples are retained at 4C $\pm$ 2C refrigerators for 30 days (unless other arrangements have been made in advance). Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved idsposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and segregated accordingly. All hazardous waste samples are disposed according to formal procedures outlined in CAS Hazardous waste SOP.

It should be noted that all waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is trated in accordance with all applicable local and federal laws. Complete internal chain-of-custody documentation is maintained for each sample from initial receipt through final disposal. This ensures an accurate history of each sample is documented.

Cooler Receipt and Preservation Check Form

Client: _____ Work order: _____

Project: _____

Cooler received on: _____ and opened on _____ by _____

		Yes	No	N/A
1	Were custody seals on outside of cooler?	☐	☐	☐
	If yes, how many and where? _____			
	Were signature and date correct?	☐	☐	☐
2	Were custody papers properly filled out (ink, signed, etc....)?	☐	☐	☐
3	Did all bottles arrive in good condition (unbroken, etc....)?	☐	☐	☐
4	Were all bottle labels correct (analysis, preservation, etc....)?	☐	☐	☐
5	Did all bottle labels and tags agree with custody papers?	☐	☐	☐
6	Were correct bottles used for test indicated?	☐	☐	☐
7	Were VOA vials checked for absence of air bubbles, and noted?	☐	☐	☐
8	Temperature of cooler upon receipt	_____ Degrees C		

Explain any discrepancies: _____

		Yes	No
pH	Reagent	x	
12	NaOH	x	
2	HNO_3	x	
2	H_2SO_4	x	

Yes = all samples OK
No = Samples were preserved at lab as listed

Comments: _____

Sample I.D.	Reage	Vol.

9.0 QUALITY CONTROL OBJECTIVES

A primary focus of Columbia Analytical Services Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. CAS has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated in its laboratories. These QC limits are either specified in the methodology or are statistically derived based on the laboratory's actual historical data obtained from control-charting the various QC measurements for each analytical method. The Quality Control objectives are defined below and the numeric values are shown in the table in Appendix C.

9.1 Accuracy

Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of standard reference materials, standard solutions and laboratory-fortified blanks. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value.

The acceptance limits for accuracy (Appendix C) originate from two different sources. Where acceptance limits are defined and stated in the individual methods, CAS has adopted the limits without modification. Where no acceptance limits are given in a method, CAS adopts the limits derived from control charts that are generated for each method. These control charts are updated once a year for the associated surrogates, laboratory control samples and matrix spike compounds.

$$\text{Accuracy (\%REC)} = \frac{A - B}{C} \times 100$$

where A = Analyte total concentration from spiked sample
B = Analyte concentration from unspiked sample
C = Concentration of spike added

9.2 Precision

Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis.

Precision is measured through the use of replicate sample analyses within the same batch and is expressed as the relative percent difference (RPD) between replicate measurements.

$$RPD = \frac{D1 - D2}{(D1+D2)/2} \times 100$$

where D1 = Original result
D2 = Duplicate result

9.3 Method Calibration Limits

The MCLs used at CAS/Houston are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MCLs are the levels to which CAS routinely reports results in order to minimize false positive or false negative results. The MCL is determined by a procedure outlined in ***40 CFR 136, Appendix B.*** MCLs are determined annually, by sample matrix and sample size.

9.4 Representativeness

Representativeness is the degree to which the field sample represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed gives results identical to analysis of the entire field sample. CAS has sample handling procedures and protocols to ensure that the sample used for analysis is representative of the entire sample. These include the SOP for Sample Preparation, Compositing, and Subsampling, the SOP for Solid Sample Preparation, and the SOP for Tissue Sample Preparation. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in the entire sample.

9.5 Completeness

Completeness is a measure of the amount of valid data that is obtained, compared to the amount that is expected. It is expected that all analyses conducted in accordance with the approved analytical methods and standard laboratory operating procedures will meet QC acceptance criteria for 95% of the samples tested.

$$\text{Completeness (\%)} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100$$

9.5 Completeness

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$$\text{Completeness (\%)} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100$$

9.6 Comparability

Comparability expresses the confidence with which one data set can be compared to another. To ensure comparability, standard operating procedures are used for the preservation, handling, and analysis of all samples. Data is reported in units specified by the customer.

10.0 QUALITY CONTROL PROCEDURES

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below. In addition, a number of other quality control processes which may impact analytical results are also described below.

10.1 Modified Procedures

CAS strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Standard operating procedures are available to analysts and are also available to our clients for review. If the modification is such that the method becomes "Performance Based," client approval is obtained for the use of the method prior to the performance of the analysis.

10.2 Procedures for Accepting New Work

Due to the increase in analytes used in the industry and found in the environment, analytes are requested to be analyzed using existing methodologies and/or new methodologies. These requests must be reviewed prior to accepting new work and creating new methodologies. These requests typically include:

- The addition of analytes to an existing scan.
- Complete start-up of an established method.
- Analyte(s) requested with no established method.

10.2.1 The addition of analytes to an existing scan

The analytical method is reviewed to determine if its use is appropriate for the new analyte. The standards are purchased from a commercial vendor and prepared. If the analyte is available from more than one source, a second source may be purchased to verify the calibration standard. A reference is spiked with a mid-level concentration of the appropriate standard and analyzed to determine retention time, resolution, etc. Temperature programs and instrument conditions may be modified to optimize resolution for the analyte. If the analyte may be resolved and detected by the method, an MDL study is performed to determine a detection limit suitable for the analyte. An in-house SOP may be written or modified to include the analyte.

10.2.2 Complete start-up of an established method

The method is obtained and reviewed by the analyst, technical manager, and/or supervisor to determine if the instrumentation and reagents needed by the method are available. If the required instrumentation is available, then reagents, standards, equipment, and supplies are gathered and purchased. If the analyte(s) are available from more than one source, a second source may be purchased to verify the calibration source. A qualified analyst performs the method, elution times are determined, temperature programs are optimized, and batch QC is performed to monitor accuracy and precision. An MDL study is performed to determine detection limit(s) and each analyst performing the method must complete an Initial Demonstration of Capability (IDC) study. An internal SOP is written and used by the analysts.

10.2.3 Analyte(s) requested with no established method

The analyte to be analyzed is researched and reviewed by the technical manager for chemical nature, formula, and other related information. The Merck Index and CRC Handbook are reviewed for boiling point, vapor pressure to determine the type of compound. After determining the type of compound, it is assumed that it can be analyzed by an existing method. If not, perhaps a modification of a method or the creation of a method could be tried. The different approaches to testing the analyte may be tried, comparing the efficiency of the various approaches. The method, which allows for the acceptable precision and accuracy, shall be used.

10.3 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples, are to be handled and processed in exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner.

The <u>minimum</u> requirements of an analytical batch are:

1. The number of (field) samples in a batch is not to exceed 20.

2. All (field) samples in a batch are of the same matrix.

3. The QC samples to be processed with the (field) samples include:
   - Method Blank - to determine possible laboratory contamination.
   - Laboratory Control Sample - to assess method performance.
   - Matrix Spike (field sample) - to assess possible matrix problems.
   - Duplicate Matrix Spike - to assess batch precision.

4. A single lot of reagents is used to process the batch of samples.

5. Each operation within the analysis is performed by a single analyst or team of analysts.

6. The time frame is not to exceed 24-hours. "Open batches" extending over more than one 24-hour period are not allowed.

7. (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8. The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch, the QC samples in the batch do not require analysis each time a field sample is analyzed.

9. Batch QC refers to the QC samples that are analyzed in a batch of (field)samples.

10. Specific project, program, or method SOP requirements may be exceptions. The more stringent QC requirements shall be followed in most all cases.

10.4 Method Blank

The method blank is either analyte-free water or analyte-free soil (when available), subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute may be used instead. The method blank is analyzed to demonstrate that the analytical system itself is not contaminated with the analyte(s) being measured. The method blank results should be below the Method Calibration Limit (MCL) for the analyte(s) being tested. A method blank is included with the analysis of every analytical batch, every 20 samples, or as stated in the method, whichever is more frequent.

10.5 Calibration Blank

Calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest, and provide the zero point of the calibration curve. The term, 'calibration blank' is used interchangeably with the term, 'instrument blank.'

10.6 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard solutions which are, in turn, prepared from stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

10.7 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method, whichever is greater. [see HRMS-8290A-rev 3.0-11/27/00; Section 11.0]

10.8 Labeled standards

Labeled standards are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each labeled standard, and the recovery is a measurement of the overall method performance. The acceptance criteria for these various analytes are listed in Appendix C, along with other data quality capabilities.
[see HRMS-8290A-rev 3.0-11/27/00; Section 11.0]

10.9 Matrix Spikes

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) has been added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. The stock solutions used for spiking the sample(s) are purchased and prepared independently of calibration standards. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$\text{Recovery (\%)} = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and

T = The theoretical concentration of analyte added to the spiked sample.

For the appropriate methods, matrix spiked samples are prepared and analyzed at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples.

Note: A sample identified as a field blank, equipment blank, or trip blank is not to be matrix spiked.

10.10 Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. A matrix spiked sample and duplicate matrix spiked sample (MS/MSD) are analyzed. The relative percent difference between an MS and MSD is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

Relative Percent Difference (RPD) = $(S1 - S2) \times 100 \div S_{ave}$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, MS/MSD analyses are performed at a minimum frequency of one set per 20 samples.

Note: A sample identified as a field blank, equipment blank, or trip blank is not to be duplicated.

10.11 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free soil (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is(are) added. A standard reference material (SRM) of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. The LCS sample is prepared and analyzed in the same analytical batch, and in exactly the same manner, as the other routine samples. Stock solutions used for LCSs are purchased or prepared independently of calibration standards. The percent recovery (% REC.) of the target analytes in the LCS assists in determining whether the methodology is in control and

whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and
accuracy. Acceptance criteria for LCS analyses are based on EPA methods. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent.

If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

10.14 Source and Preparation of Standard Reference Materials

CAS relies on a primary vendor for the majority of its analytical supplies. In addition, consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method, as discussed in section 4.2.2 of this document. Supelco and Cambridge Isotope Laboratories (CIL) are examples of the vendors used by CAS. All reference materials that are received at CAS are recorded by the technical staff in the appropriate notebook(s) and are stored under conditions that provide maximum protection against deterioration and contamination. The notebook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. In addition, the date that the standard is received in the laboratory is marked on the container. When the SRM container is used for the first time, the date of usage and the initials of the applicable technician are also recorded on the container. Stock solutions and/or calibration standard solutions are prepared fresh as often as necessary according to their stability. After preparation, all standard solutions are properly labeled as to name, concentration, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook. To ensure traceability, all standards are labeled with an in-house code that can be traced back to the original stock standard received by the vendor and thus, the certificate of analysis. Prior to introduction into the analytical system/process, all reference materials are verified for accuracy with a second, independent source of the material. Once the reference material has been verified to be accurate, it may then be used for instrument calibration and subsequent quantitative purposes. In addition, the independent source of reference material is also used to check the calibration standards for signs of deterioration.

10.15 Proficiency Testing Participation

Each decipline and test method for most analytes are monitored using NIST approved vendors for Proficiency Testing on a semi-annual basis. Results of the proficiency samples are reviewed by the Laboratory Director, the QA Coordinator, the Corporate QA Director and the laboratory staff. Any problems surfacing during the review are investigated, and corrective action is taken regarding any and all deficiencies.

Interlaboratory Proficiencies are performed annually to determine continued lab performance throughout the network of CAS laboratories. Such studies are organized by Corporate QA.

10.16 Glassware Washing

Glassware washing and maintenance play an crucial role in the daily operation of a laboratory. The glassware used at CAS undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at CAS; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

11.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at CAS are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Personnel who have been properly trained in these procedures perform operation and calibration. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below.

11.1 Temperature Control Devices

Temperatures are monitored and recorded for all of our temperature-regulating devices including ovens, incubators and refrigerators. Bound record books are kept which contain daily recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the appropriate SOP (SOP No. SMO-DALYCK). All thermometers have been identified according to serial number, and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is re-certified by a professional metrology organization on an annual basis.

11.2 Analytical Balances

Analytical balances are serviced on an annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on an annual basis. The calibration of each analytical balance is checked daily with three class-S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high ranges. As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Bound record books are kept which contain the recorded measurements, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the appropriate SOP (SOP No. SMO-DALYCK).

11.3 Water Purification System

CAS purchases HPLC water for the preparation of standards and reagents. This purchased water meets specifications for ASTM Type I water, with a resistivity of 18 megaohms.

11.4 Low-Res and High-Res GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All SRMs used for this function are "EPA-Certified" standards. Compounds selected as system performance check compounds (SPCCs) must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds (CCCs) must also meet method specifications for percent difference from the multipoint calibration. Method-specific instrument tuning is regularly checked using perfluorokerosene (PFK). Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed.

11.5 Pipets

The calibration of pipets and auto pipettors used to make critical-volume measurements is verified following the *SOP for Checking Pipet Calibration*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. Autopipet calibration is verified each day of use. The results of all calibration verifications are recorded in bound logbooks.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

CAS reports the analytical data produced in its laboratories to the client via the certified analytical report. This report typically includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Sample Login System

CAS/Houston currently uses CASELS login system. This data login system is an Excel based system that runs on a Windows 98 server. CASELS is used for sample tracking and invoicing.

12.2 Data Reduction and Data Custody

All data is initially reviewed and processed by analysts using appropriate methods (e.g. chromatographic software, instrument printouts, hand calculation, etc.) A file of all raw data is printed, reviewed for completeness and quality criteria against an in-house checklist and signed off by the analyst. The operations manager reviews all reported data against the raw data; validating completeness and quality. The final report data package is then reviewed by the project manager for compliance with previously established project requirements. Typically, all data is reported in the units and ~~MCLs~~ Compound Reporting Limits listed in Appendix C. OK 5/19/3

Assessment of the analytical data includes a check on data consistency by looking for comparability of duplicate analyses, comparability of previous data from the same sampling location (if available), adherence to accuracy and precision control limits, and anomalous low or high parameter values. The results of this review will be discussed with either the departmental supervisor or lab director for resolution prior to final release of the package.

Once the data has been checked for accuracy and acceptability, the final report and raw data is forwarded to the lab director or quality assurance coordinator, who further reviews the data package for errors. When the entire data set has been found to be acceptable the lab director signs the report, the report is distributed and the raw data is filed for approximately one year, then archived. All hard copy and electronic backups are archived in a secured file room for a period of at least 5 years from the date of the final report. It is not unusual to have various clients require a 10-year retention of records, therefore, the archivist, project chemist, and possibly the client are consulted prior to distruction of the records.

12.3 Confirmation Analysis of 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF)

12.3.1 All positive results of the 2,3,7,8-TCDF that are quantitated on the DB-5 column, are confirmed by a second (DB-225) column.

When sample results are confirmed by two dissimilar columns, the agreement between quantitative results must be evaluated.

12.3.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for high-resolution GC/MS methods is summarized below:

- High-resolution GC/MS methods – criteria used to verify identification:
    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
    2. Signal/noise ratio (S/N) $\geq$ 2.5
    3. Satisfy ion abundance ratio criteria

12.4 Data Validation

The integrity of the data generated in the laboratory is primarily assessed by the analyst, supervisor and project chemist through the use of a variety of measures that may include reagent blanks, laboratory fortified blanks, duplicates, matrix spikes and QC samples. The numerical criteria for evaluation of these QC samples are listed in Appendix C; these various QC sample analyses are evaluated using the flow diagrams found in Figures 12-1 through 12-9. Other validation measures of the data include a check of the linearity of the calibration curve, an accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed. Specific calculations used for determining the concentration or value of the measured parameters from the raw data are given in each of the analytical methods or CAS SOPs.

12.5 Data Reporting

When an analyst determines that the data has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data is validated by the supervisor. Prior to release of the report to the client, the project manager must also review the entire body of data for completeness and to ensure that any and all client-specified objectives were successfully achieved. If required, samples exceeding any established state/federal maximum contaminant level or reportable concentration level, must be reported to the client. A case narrative may be written by the project manager to explain any unusual problems with a specific analysis or sample, client-specific objectives, exceedences, etc... The original raw data, along with a copy of the final report, is archived. CAS maintains control of analytical results by adhering to standard operating procedures and by observing sample custody

requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report. Typical qualifiers used to flag analytical results are listed in Appendix D.
[Reference; SOP: ADM-REP, Reporting Analytical Results, rev. 0.0, 12/11/00]

12.6 Documentation

A document control system ensures that all documents are accounted for when the project is complete. A service request number is assigned to each project for reporting and filing purposes. This number is associated with each order number (sample).

12.6.1 Documentation and Archiving of Routine Analysis Data

The archiving system includes all of the following items for each set of analyses performed:

- Chain-of-custody documentation
- Benchsheets describing sample preparation
- Sample analysis sequence
- Analysis benchsheets and instrument printouts
- Chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns
- Log book ID number for the appropriate standards
- Copies of report submitted to the client
- Copies of Nonconformity and Corrective Action Report (NCAR) forms, if needed

Individual sets of analyses are indexed by analysis date and/or service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

The archive room is an off-site file room in which files shall be maintained for a period of at least five years (from date of report issue). It is not unusual to have various clients require a 10-year retention of records, therefore, the archivist, project manager, and possibly the client are consulted prior to distruction of the records. The archive eabinet and/or off site storage area is kept locked and access keys are controlled. All documents must be signed out if needed outside of the archive room and returned in a timely manner. A designated archivist monitors filing, incoming, and outgoing data from the archive.

12.6.2 Reporting Deliverables

In order to meet individual project needs, CAS provides several levels of analytical reports. Basic specifications for each level of deliverable are described in Table 12-1. Turnaround time and package level are negotiable on a project to project basis.

12.6.3 Electronic Data Deliverables (EDD)

CAS/Houston offers standard Excel format as well as a variety of custom developed EDDs such as ASCII, dBase, and GISKEY. EDDs are available upon request on a project to project basis.

Figure 12-1
Evaluation of Initial Calibration

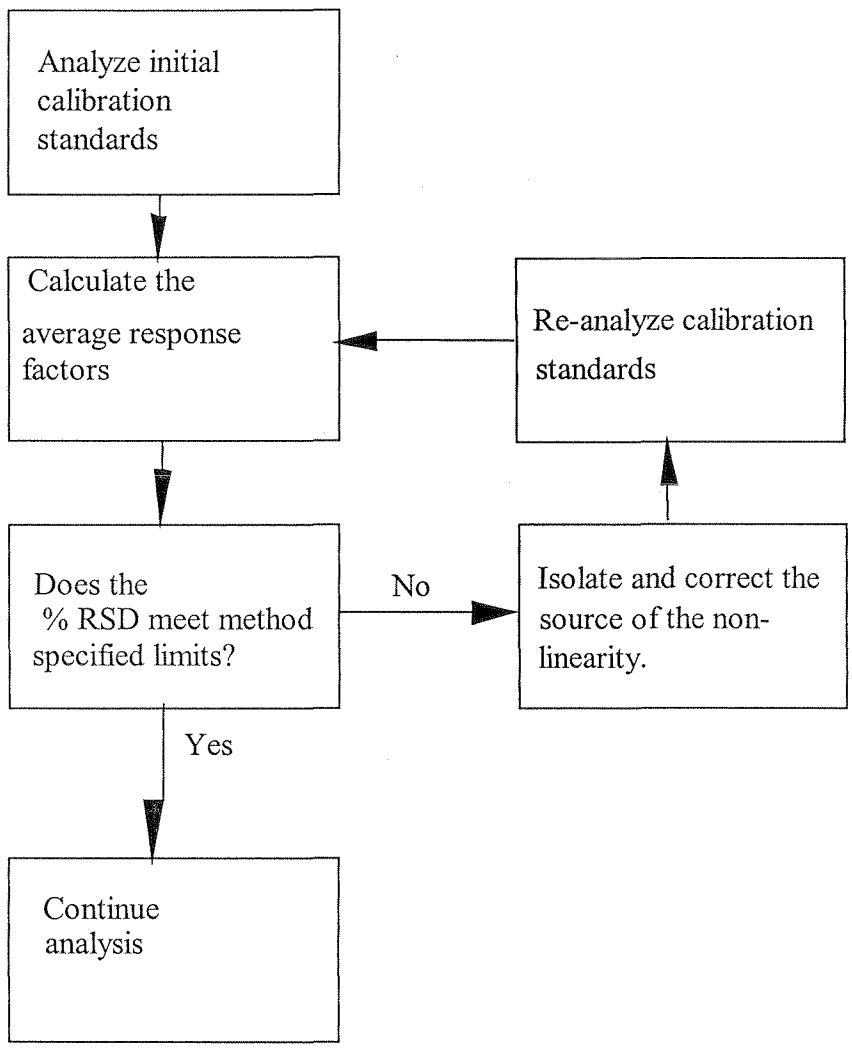


Figure 12-2
Evaluation of Continuing Calibration

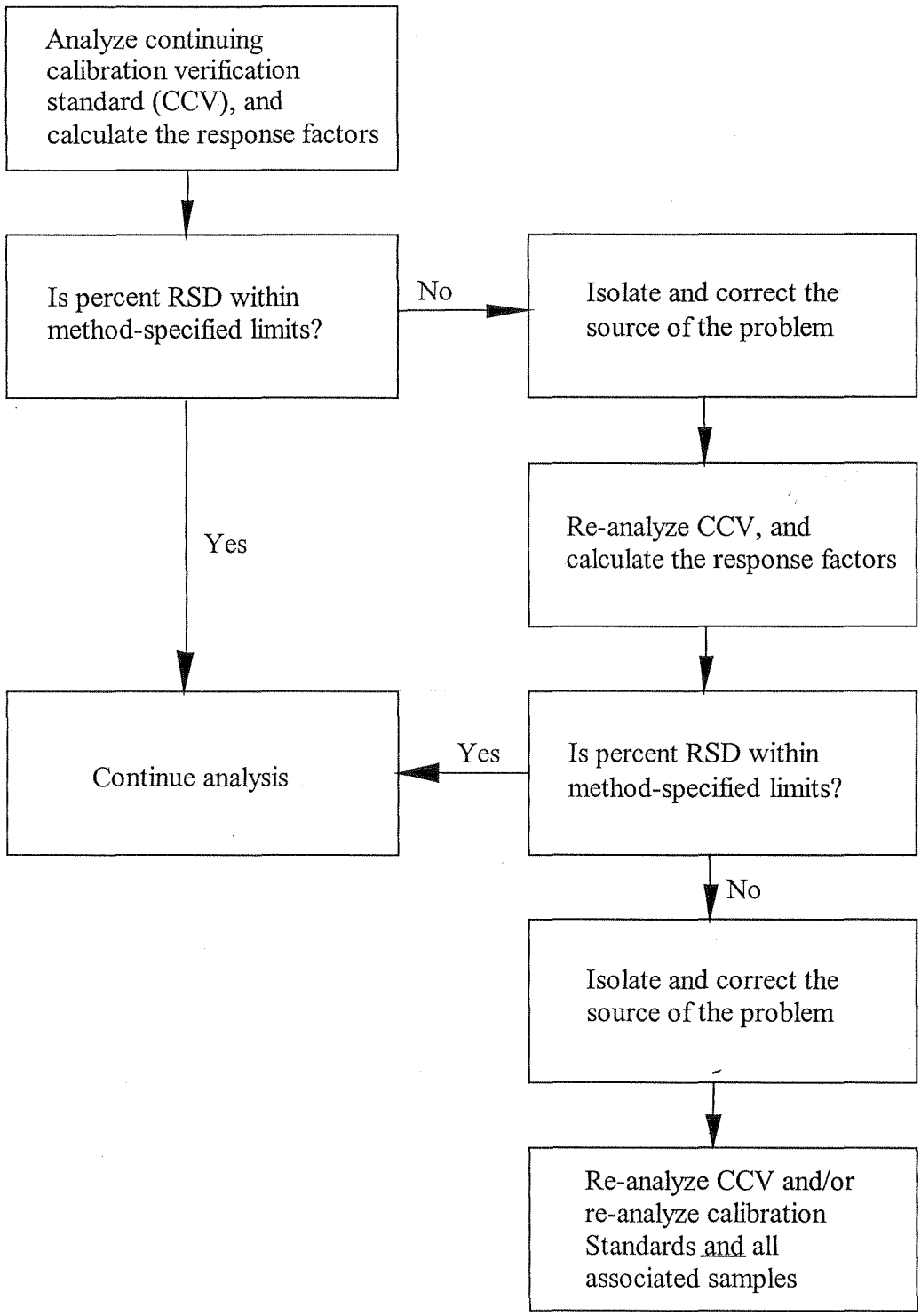
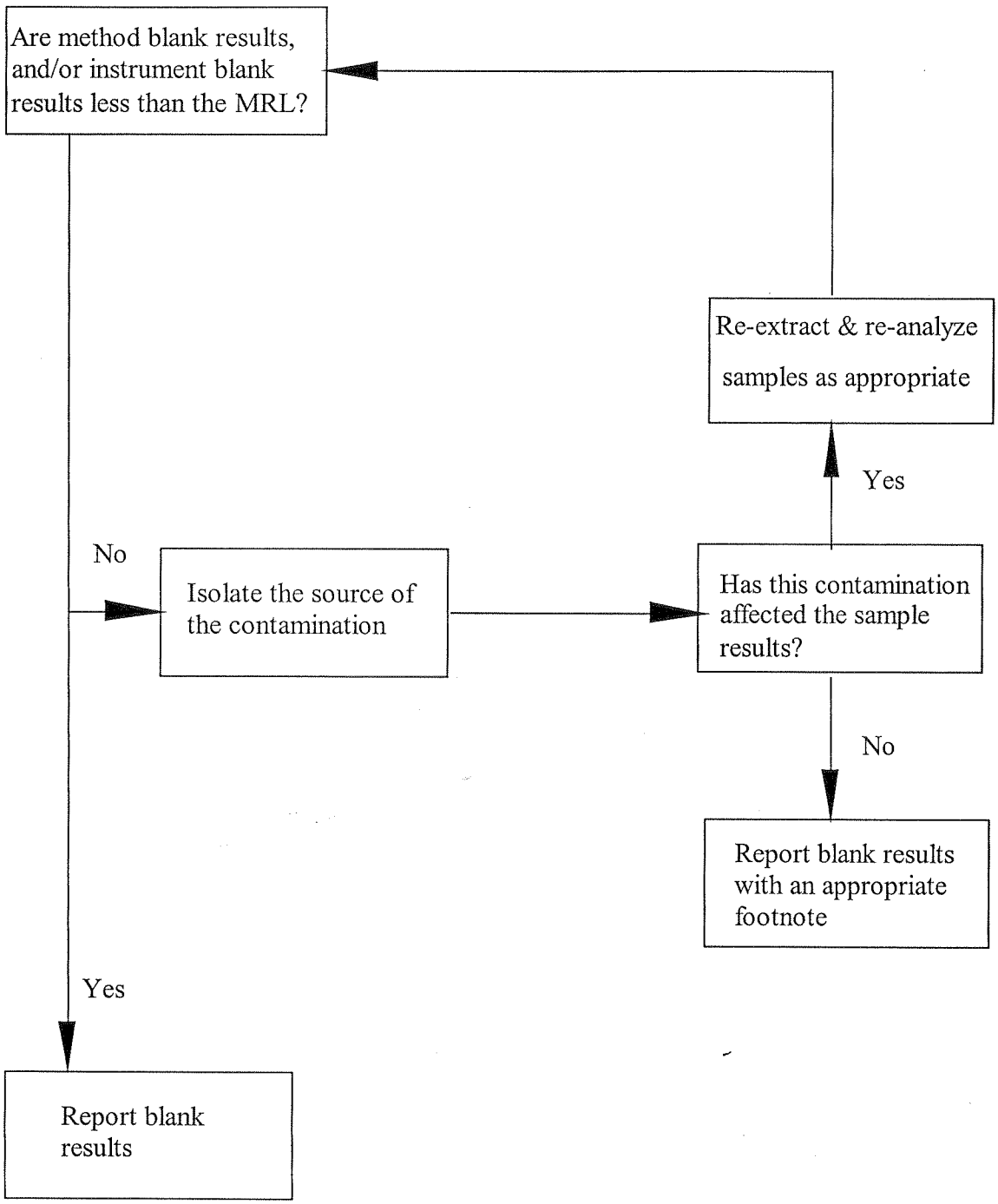


Figure 12-3
Evaluation of Method Blank and Instrument Blank Results



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Evaluation of Sample Results for Organic Analyses

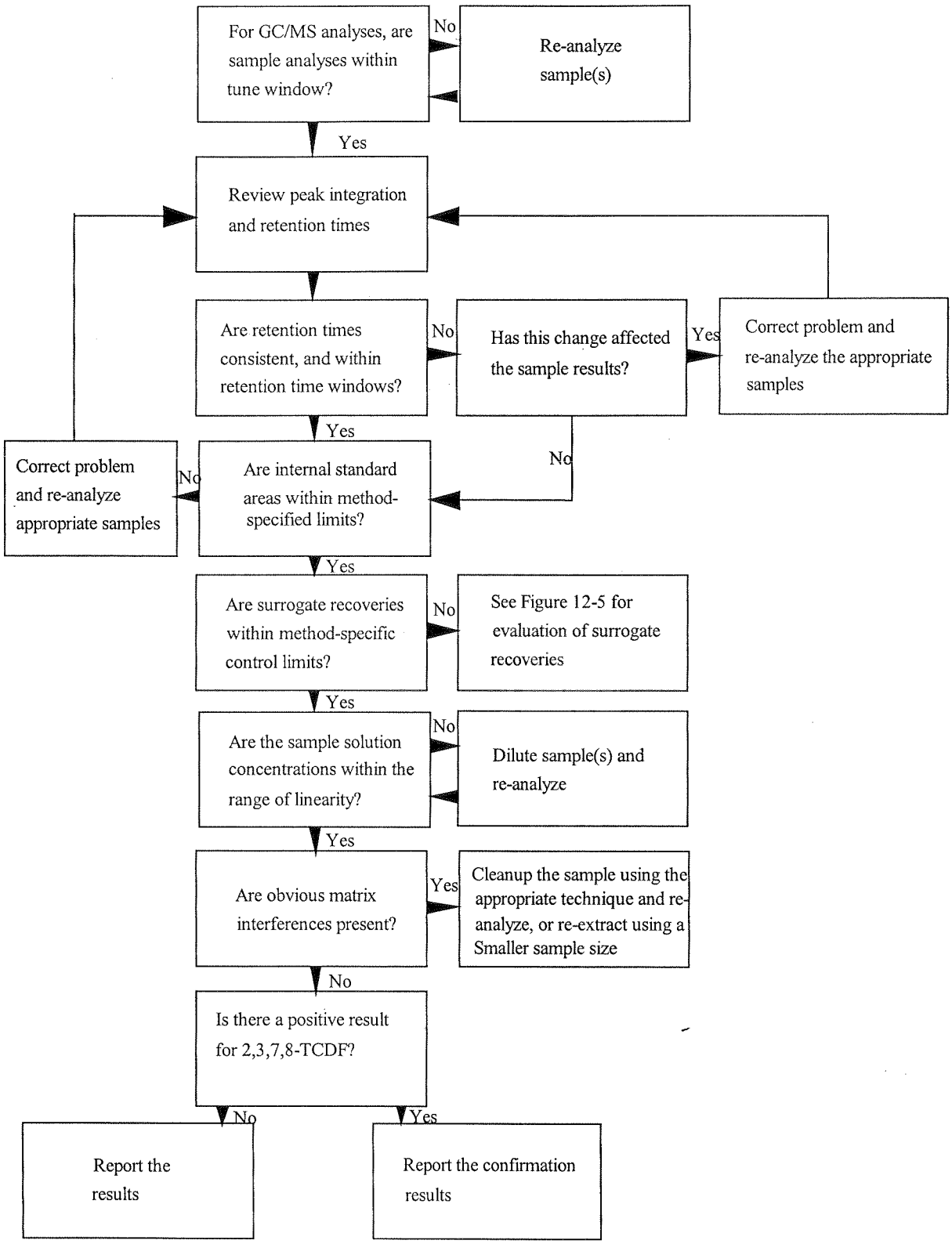


Figure 12-5
Evaluation of Labeled Standard (Surrogate) Compounds

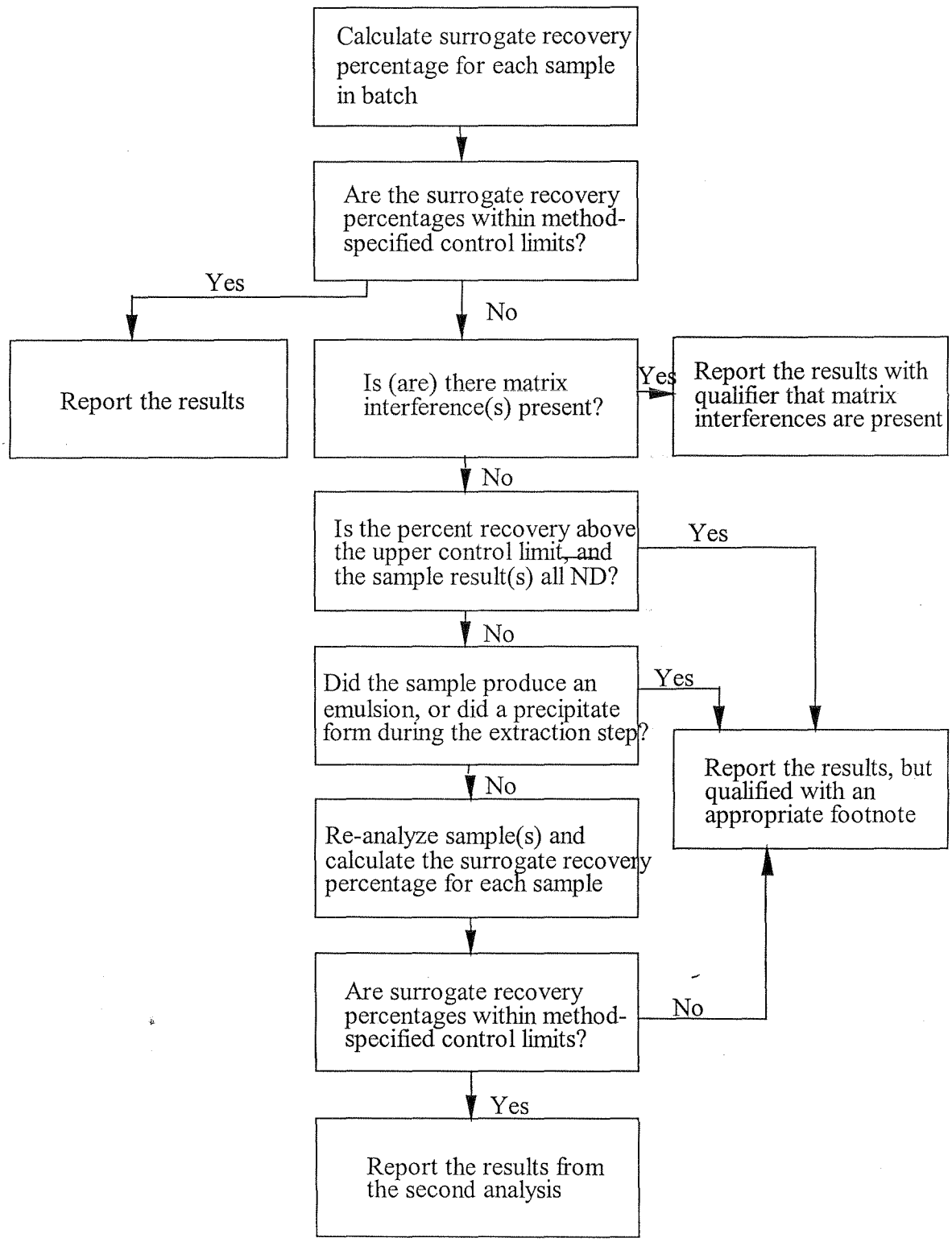


Figure 12-6
Evaluation of Duplicate Sample and/or Duplicate Matrix Spike Results

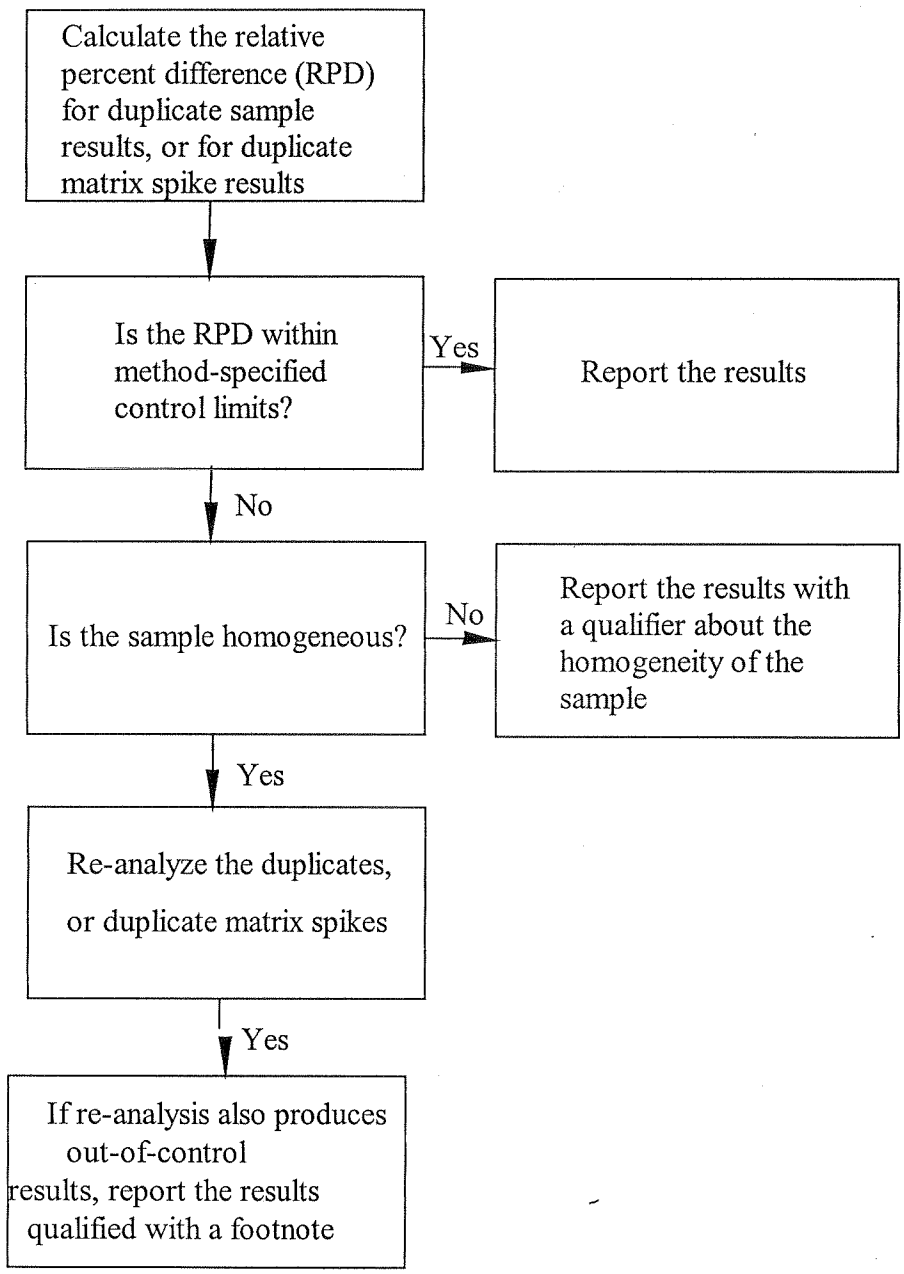


Figure 12-7
Evaluation of Matrix Spike Recoveries

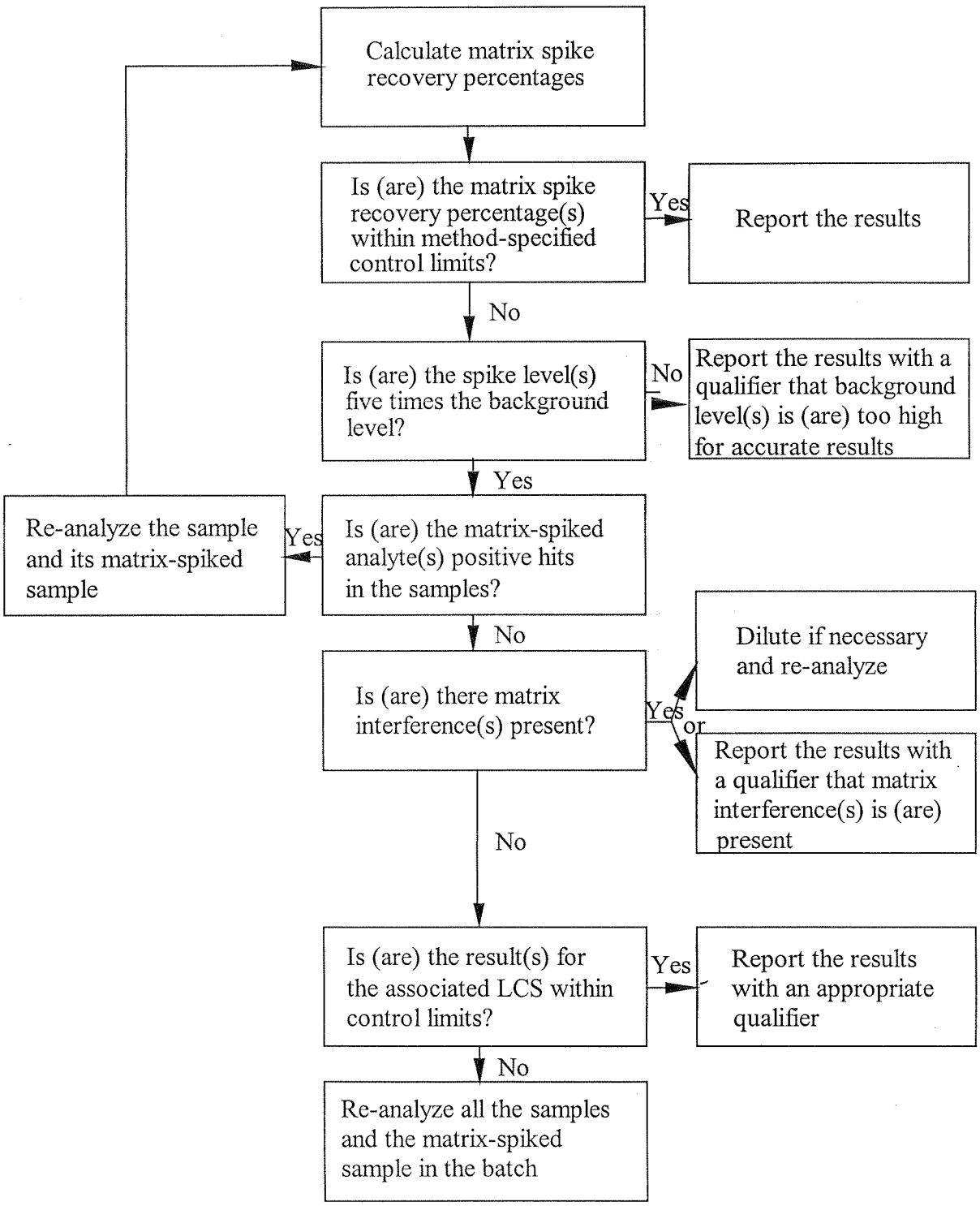


Figure 12-8
Evaluation of Laboratory Control Sample (LCS) Results

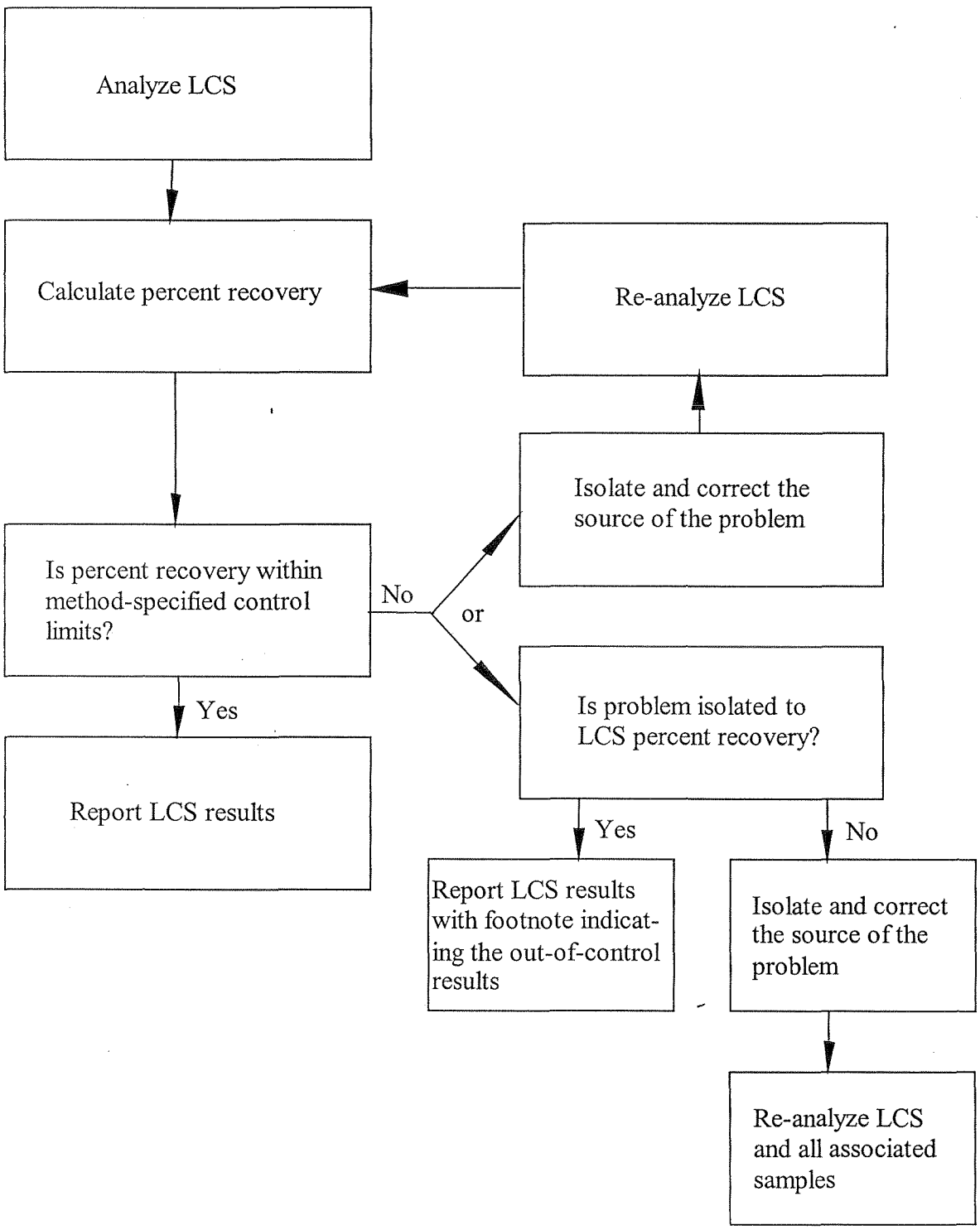


Table 12-1
Laboratory Data Deliverables

Package 1. A Routine Certified Analytical Report Includes the Following

1. Transmittal Letter
2. Sample Analytical Results
3. Method Blank Results
4. Surrogate Recovery Results for appropriate organic methods, including associated EPA or CAS acceptance criteria
5. Chain of Custody Documents

Package 2. In Addition to the Package 1 Deliverables, this Report Includes the Following:

1. Case Narrative

Package 3. In Addition to the Package 2 Deliverables, this Report Includes the Following:

1. Calibration Summaries and Results of initial and continuing calibration verification standards, with calculated recoveries
2. Method Blank Summaries

Package 4. In Addition to the Package 3 Deliverables, this Report Includes the Following:

1. Sample Quantitation Report
2. Standards Prepartation Information

Columbia Analytical Services, Inc.

Houston, Texas

Last updated: 03/07/02

Employee Signature Record

EMPLOYEE	SIGNATURE	INITIALS
CRAWFORD, KAREN	Karen Crawford	KC
DIAZ, CARLOS	[illegible]	CID
DIAZ, ROLANDO	[illegible]	RD
FREEMYER, JANE	Jane Freemyer	JF
LIANG, XIANGQIU	Xiangqiu Liang	XL
VILLALOBOS, ALEX	Alex Villalobos	AV

Columbia Analytical Services, Inc.

Houston, Texas

Last updated: 05/23/02 (AF)

Employee Signature Record

EMPLOYEE	SIGNATURE	INITIALS
ELHARDT, CHRIS	Chris Elhardt	cee
VERSCHOOR, KAREN	Karen Verschoor	KVV
Edelman, Dave	Dave Edelman	de
~~Wieget, Joe~~	~~Joe Wieget~~	~~JW~~

(AF 6/4/2)

(~~AF~~ 6/13/2)

AF 5/19/3

Columbia Analytical Services INC.

An Employee-Owned Company

ACCREDITED IN ACCORDANCE WITH nelac

Columbia Analytical Services, Inc.

Houston, Texas

~~June 30, 2003~~ Jan. 5, 2004

Employee Signature Record

EMPLOYEE	SIGNATURE	INITIALS
ELHARDT, MARY	Mary Elhardt	M.E.
LIANG, DAVID	David Liang	D.L.
GNANAPOOMPAVAI SHANMUGAM	G. Gnanapoompavai	G.S.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality Control (QC) audits are an essential part of CAS's QA program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the field and laboratory QA program, while Performance Audits are conducted by analyzing performance evaluation samples in order to quantitatively evaluate the outputs of the various measurement systems.

The system audit examines the presence and appropriateness of laboratory systems. External system audits of CAS are conducted regularly by various regulatory agencies and clients. Appendix F summarizes some of the major programs in which CAS/Houston participates. Additionally, internal system audits of CAS/Houston are conducted regularly by the Quality Assurance Coordinator and by the CAS Quality Assurance Director. The internal system audits are scheduled as five auditing events as follows:

- Comprehensive lab-wide system audit - annually
- Comprehensive "vertical" project audits examining compliance with all QA program requirements as applied to selected projects and implementation of QA program requirements - 1 per year
- Focused audits examining the lab-wide implementation of a selected QA program requirement – 1 per year

The results of each audit are reported to the Laboratory Director for review and comment. Any deficiencies noted by the auditor are summarized in an audit report and corrective action is taken within a specified length of time to correct each deficiency. If problems impacting data quality are found during an internal audit, any client whose data is adversely impacted will be given written notification if not already provided.

Additionally, CAS/Houston participates in the analysis of performance evaluation (PE) samples. Results of the performance evaluation samples and audits are reviewed by the Laboratory Director, the QA Coordinator, the Corporate QA Director and the laboratory staff. Any problems surfacing during the audit are investigated, and corrective action is taken regarding any and all deficiencies.

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of Columbia Analytical Services Quality Assurance program. Instruments at CAS (e.g., high-resolution and low-resolution GC/MS systems, analytical balances, etc.) are maintained under commercial service contracts. All instruments are operated and maintained according to the instrument operating manuals and technical SOPs. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at CAS contain relevant information about the instruments used at the laboratory.

A system calibration check (HRCC3 or LRCC3) is performed to demonstrate a return to analytical control after an analytical instrument has undergone maintenance, before sample analysis is resumed. System calibration checks bracket sample analysis, as described in the analytical methods. Instrument failure or anomalies determined to have an impact on previous calibrations or tests are investigated and documented using Nonconformity and Corrective Action Reports. These reports are filed in the analytical project files by SR#.

An initial demonstration of analytical control is required on each instrument used at CAS before sample analyses may proceed. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can continue. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number.
- Date the equipment was received.
- Date the equipment was placed into service.
- Condition of equipment when received (new, used, reconditioned, etc...)
- Prior history of damage, malfunction, modification or repair (if known).

Preventative maintenance procedures, frequencies, etc. are available for each instrument used at CAS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase. Responsibility for ensuring that routine maintenance is performed lies with the laboratory director. Each laboratory maintains a critical parts inventory. The parts inventories include the items needed to perform the preventative maintenance procedures listed in Appendix E. This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs.

When performing maintenance on an instrument (whether preventative or otherwise), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem
- Repairs and/or maintenance performed
- Description and/or part number of replaced parts
- Source(s) of the replaced parts
- Analyst signature and date
- Demonstration of return to analytical control

For most major equipment, back-up equipment is available to avoid downtime. All major analytical equipment is summarized in Appendix A. The laboratory director coordinates repair with the manufacturer. The project chemist shall assess the effect of the downtime on the samples in-house and notify the appropriate clients of any delays and/or the possibilities of subcontracting.

15.0 CORRECTIVE ACTION

Failure to meet established analytical controls, such as the quality control objectives outlined in Section 9.0, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the laboratory director, and/or the quality assurance manager may examine and pursue alternative solutions. In addition, the appropriate project chemist may be notified in order to ascertain if contact with the client is necessary.

The quality assurance manager initiates corrective action due to a performance audit or a check sample problem; the affected laboratory personnel are promptly informed.

A Nonconformity and Corrective Action is generated to document and notify the appropriate personnel of the nonconformity. Nonconformity include, but are not limited to, method blank contamination, re-extractions, dilutions, etc.

In special cases, the laboratory director may give permission to the analyst or Project Chemist to deviate from CAS Policy. A Nonconformity form must be signed by the quality assurance manager.

In cases were there are complaints from the clients, follow policy procedures outlined in the SOP, ADM-CMPLT (Dealing with Complaints).

Figure 15-1

Nonconformity and Corrective Action Report

SAMPLES/SYSTEM/JOB/CLIENT AFFECTED

NONCONFORMITY

Analysis/Event: _____

Instrument/System: _____ Date: _____

Detailed Description:

Originator: _____ Date: _____

Supervisor Verification: _____ Date: _____

CORRECTIVE ACTION AND OUTCOME

Detailed Description: (Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the problem. Describe the outcome.)

Person Responsible: _____ Date: _____

Supervisor Verification: _____ Date: _____

NOTIFICATION - CUSTOMER/CLIENT - INTERNAL/EXTERNAL

Project Chemist Notified? No Yes: Date: _____

Customer Notification Necessary? (Attach telephone record) No Yes: Date: _____

Notifier: _____ Date: _____

ACCEPTANCE OF CORRECTIVE ACTION

QA Manager: _____ Date: _____

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by CAS personnel at all levels of the organization. Information flow and feedback mechanisms are designed so that analysts, supervisors and managers are aware of quality assurance issues in the laboratory.

Analysts performing routine tests in the laboratory are aware of the various method acceptance criteria and in-house control limits that must be met in order to generate acceptable results. Any non-conformities and corrective actions may also be attached to the data prior to review. Supervisors review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and corrected if possible.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must also accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness. The project chemist must also review the entire body of data to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc...

The Quality Assurance Manager (QAM) provides overview support to the project chemists if required to do so (e.g. contractually specified, etc.) The QAM is also responsible for the oversight of all internal and external audits, for all performance evaluation sample and analysis programs, and for all laboratory certification/accreditation responsibilities.

The QAM also prepares quarterly reports for the Laboratory Director which summarize the various QA/QC activities that have occurred during the previous quarter. These reports may include a summary of the findings of the various audits performed during the last quarter, copies of audit-deficiency correspondence between the laboratory and external auditors, new accreditations/certifications received by the laboratory, scores of the most current performance evaluation studies, updates/revisions to controlled documents, etc... Any problems noted by the lab director are then discussed during the regularly-scheduled staff operations meetings with all appropriate staff.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the QA Manager and Human Resources. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at CAS are evaluated, in part, against the appropriate technical description.

Information of previously acquired skills and abilities for a new employee is entered into a centralized database maintained by Human Resources. The database is also used to record the various technical skills and abilities acquired and maintained by an employee while employed by CAS. Information in the database includes the employee's name, a description of the skill including the appropriate method reference, the name of the supervisor who certified completion of the training, and the date the training was completed. Technical training is documented following CAS SOP requirements. CAS-Houston maintains a training summary file for all Houston employee's.' The training summary lists all Standard Operating Procedures for the facility and is a tool for tracking individual employee training status for those procedures. The training summary is used to track and schedule training as relevant to the employee's position.

Training begins the first day of employment at CAS (ADM-TRANDOC) when the company policies are presented and discussed. Training in analytical procedures typically begins with the reading of the SOP for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the analysis must include an Initial Demonstration of Capability Study of four replicate quality control samples. Continued demonstration of capability is monitored with batch QC to maintain continuing qualification. Initial Demonstration of Capability is required anytime a new method is used, a new analyst is performing the method, or new instrumentation is installed.

Safety training begins with the reading of the *Safety Manual*. All employees are recommended to attend monthly safety meetings during which the safety programs discussed and safety training is presented by the Environmental, Health and Safety Officer.

CAS encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the company. Ongoing training occurs for all employees through a variety of mechanisms. The "CAS University" education system, external and internal technical seminars and training courses, laboratory-specific training exercises and performance of external PE samples analysis are all used to provide employees with professional growth opportunities.

Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all processes at CAS.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES

The analytical methods used at CAS generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. Typical methods used at CAS are taken from the following references:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, 1986 and Updates I (7/92), II (9/94), IIA (8/93), IIB (1/95), and III (12/96). See Chapters 1, 2, 3, and 4.

- *Methods for Chemical Analysis of Water and Wastes*, EPA 600/4-79-020, Revised March 1983.

- *Methods for the Determination of Metals in Environmental Samples*, EPA 600/4-91-010, June 1991 and Supplement I, EPA/600/R-94/111, May, 1994.

- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057, July 1982 and 40 CFR Part 136, Appendix A.

- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA 600/R-93/100, August 1993.

- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA 600/4-88-039, December 1988 and Supplement I (7/90) and Supplement II (8/92).

- *Standard Methods for the Examination of Water and Wastewater*, 16th Edition, 1985; 17th Edition, 1989; 18th Edition, 1992, and 19th Edition, 1995.

- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.

- 40 CFR Part 141, National Primary Driniking Water Regulations.

- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products.

- Annual Book of ASTM Standards.
- EPA Contract Laboratory Program, Statement of Work for Organics Analysis, OLM04.2. May 1999.
- EPA Contract Laboratory Program, Statement of Work for Inorganics Analysis, ILM04.0.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012, February 1993.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013, February 1994.
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition, August 1987.
- *Good Automated Laboratory Practices, Priciples and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185, August 1995.
- *National Environmental Laboratory Accreditation Conference, Quality Standards, Chapters 1-5*, July 1999.

Test Methods Performed
(10/15/03)

Method Name	Method Number	Sample Matrices
Dibenzo-p-dioxins and Dibenzofurans	8290A	Water, soil, sediment, tissue, industrial products, food, wipes
Dibenzo-p-dioxins and Dibenzofurans	8280A	Water, soil, sediment, tissue, industrial products, food, wipes
Dibenzo-p-dioxins and Dibenzofurans	1613B	Water, soil, sediment, tissue, industrial products, food, wipes
Dibenzo-p-dioxins and Dibenzofurans	Method 23	Air
Polychlorinated Coplanar PCBs	1668A	Water, soil, sediment, tissue, industrial products, food, wipes

SOP INDEX

FY03: APRIL 2004 – MARCH 2005

PREPARED BY: JANE FREEMYER

SUBJECT: INDEX OF SOPS IN PRACTICE AT CAS-HOUSTON LABORATORY

LAST UPDATED: 5/6/04

STANDARD OPERATING PROCEDURES (SOPS)

FILING #	TITLE	CODE	REV#	REV. DATE	CORP. SOP NOTED
1	**SOPs:** Preparation of SOPs	ADM-SOP	4	07/31/03	CORP
2	**SOPs:** Document Control	ADM-DOC_CTRL	4	07/31/03	CORP
3	**SOPs:** Training Documentation	ADM-TRANDOC	7	08/01/03	CORP
4	**SMO:** Purchasing	ADM-PUR	1	12/17/03	CORP
5	**SMO:** Documentation Requirements for Standard & Reagent Login, Preparation & Tracking SOP	SMO-SPSR	1	02/20/03	
6	**SMO:** Checking New Lots of Chemicals for Contamination	ADM-CTMN	2	08/01/03	CORP
7	**SMO:** Daily Check of Balances, Refrigerators, Ovens & Incubators	SMO-DALYCK	0	02/27/03	
8	**SMO:** Sample Receiving	SMO-SR	1	12/06/02	
9	**SMO:** Chain of Custody for Sample Transfer between Laboratories	ADM-COC	1	02/22/01	CORP
10	**SMO:** Subcontracting to Outside Laboratories	ADM-SUB	1	01/30/02	CORP

FILING #	TITLE	CODE	REV#	REV DATE	CORP SOP NOTED
11	**SMO:** Measuring Total Solids	GEN-160.3	0	ARCHIVED	
12	**SMO:** Solid Sample Preparation	GEN-SPLPREP	0	ARCHIVED	
13	**SMO:** Waste Disposal	SMO-WASTDISP	1	11/19/02	
14	**SMPL:** Fixed Volume Pipets	PREP-PCAL	1	02/20/03	
15	**SMPL:** Checking Pipet Calibration	ADM-CPIP	0	02/20/03	
16	**SMPL:** Sample Batches	ADM-BATCH	3	03/14/02	CORP
17	**SMPL:** Making Entries into Logbooks and Bench Sheets	ADM-DATANTRY	4	09/08/03	CORP
18	**SMPL:** Determination of Method Detection Limits	ADM-MDL	5	08/01/03	CORP
19	**SMPL:** Significant Figures	ADM-SIGFIG	5	03/10/03	CORP
20	**SMPL:** Determination of Control Limits	ADM-CTRL_LIM	3	10/03/03	CORP
21	**DATA:** Manual Integration of Chromatographic Peaks	ADM-INT	2	02/20/04	CORP
22	**DATA:** Non-conformity and Corrective Action Documentation	ADM_NCAR	3	07/17/02	CORP
23	**DATA:** Laboratory Data Review and Reporting	ADM-REP	0	12/10/02	
24	**DATA:** Dealing with Complaints	ADM-CMPLT	3	03/14/03	CORP
25	**DATA:** Data Archiving	ADM-ARCH	0	11/08/02	

FILING #	TITLE	CODE	REV#	REV DATE	CORP SOP NOTED
26	**DATA:** Tape Backup and Archive	DATA_TAPE	2	07/16/02	CORP
27A	**HRMS:** Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS	HRMS-1613B	4	07/23/03	
27B	**HRMS:** Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS – wastewater matrix only	HRMS-1613B_WW	5	08/04/03	
28	**HRMS:** Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS	HRMS-8290	5	02/26/03	
29	**HRMS:** Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS	HRMS-M23	1	09/03/03	
30	**LRMS:** Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/LRMS	LRMS-8280A	2.1	05/30/03	
31	**QA:** Quality Assurance Manual/CAS-Houston	QAM	4.0A	01/16/03	
32	**QA:** Software Quality Assurance Plan	ADM_SQAP	1	09/26/01	CORP
33	**QA:** Preparation of Electronic-Data for Organic Analyses for Electronic-Data Audits	ADM-E_DATA	1	03/14/03	CORP
34	**WET:** Cleaning and Presoxhing 500mL Flat-bottom Flasks	500MLFB	1	03/06/03	
35	**ADM:** Estimation of Uncertainty Measurements	ADM-UNCERT	1	10/01/03	CORP
36	**ADM:** Confirmation of Organic Analyte Identification and Quantitation	ADM-CONFIRM	1	07/16/02	CORP
37	**SAFETY:** Chemical Hygiene Plan	CHP	2	09/03/03	
38	**SMO:** Bottle Order Preparation and Shipping	SMO-BOT	1	07/14/03	
39	**SMO:** Washing Glassware	SMO-WASH	0	07/25/03	
40	**ARCH_HRMS:** Archiving Data for HRMS	ARCH_HRMS	0	07/28/03	

FILING #	TITLE	CODE	REV#	REV DATE	CORP SOP NOTED
41	**MANAGERIAL REVIEW:** of Laboratory's Quality Systems	ADM-MGMTRVW	0	10/20/03	Corp
42	**METHOD 1668, Revision A:** Chlorinated Biphenyl Congeners in	Published Method			

As directed by NELAP, CAS/Houston is referencing the published method for 1668A, while we are writing our internal SOP. JF. 04/05/04.

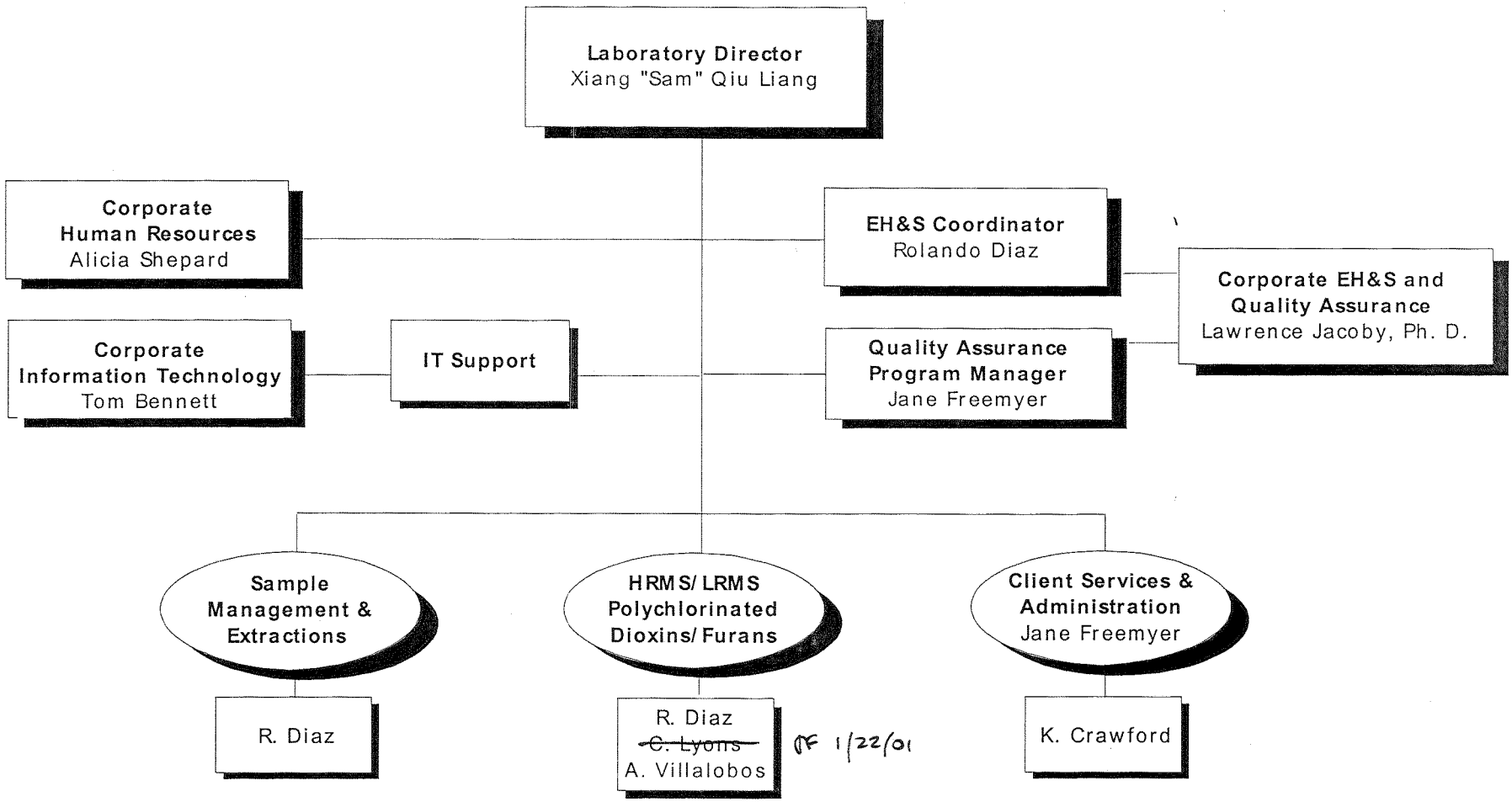
Appendix A: Major Analytical Equipment

Major Analytical Equipment

Equipment	Year Purchased	Manufacturer (MM) or Laboratory (LM) maintained	Number of trained operators	Instrument ID – HRMS/SN#
GC-MS Systems (3):				
▪ VG Analytical HRGC/HRMS 70S w/Opus VMS data system	1986	MM	3	**A** - 7043
▪ VG Analytical HRGC/HRMS 70S w/Opus VMS data system	1987	MM	3	**B** - 7059
▪ VG Analytical HRGC/HRMS 70S w/Opus VMS data system	1988	MM	3	**C** - 7070
Wet Lab & Organics' Prep:				
Innova Platform Shaker	1985	LM	3	
Buchi Rotavapor R110	1985	LM	3	
Evaporator NVAPIIII	1985	LM	3	
GS Drying Oven	1985	LM	3	
Atrason Ultrasonic Cleaner	1985	LM	3	
Analytical Balances (2):				
▪ Mettler H51AR	1985	MM	3	
Clay Adams Centrifuge	1985	LM	3	
Fisher Ultrasonic Bath	1985	LM	3	
Automated Data Processing Equipment:				
(1) NT application server	2002	LM	6	
(3) HP Laserjet printers (various types from IIIs to SI-IVs) linked via LAN	2000	LM	6	
HP/Gateway PC on LAN w/10BT/100BT	1999	LM	6	
MS Office 2000 software	2002	LM	6	
MS Outlook 2000 software				
(1) Facsimile machine; 9600-14400 baud, 11 pages/minute	1999	LM	6	

Appendix B: Organizational Chart and Resumes of Key Personnel

Columbia Analytical Services, Inc.
Houston, Texas Laboratory Organization



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ANGQIU LIANG 1988- PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Houston Branch Manager**

Experience

August 2000-present – Responsibilities include management of technical, business and operations set-up, development and validation of LRMS/HRMS and HRMS/HRGC analysis methods, supervision of QA/QC programs, and analytical staff training.

1993-2000 Senior Chemist – Southwest Lab of Oklahoma, Broken Arrow, OK. Responsibilities included research and development of new methods for HRMS/HRGC and LRMS/HRGC for dioxins and furans, PAH, PCB and pesticides, analytical staff training, coordination of activities with other laboratory sections and maintenance of analytical instruments, DECNET and LIMS network system.

1990-1993 Analytical Lab Supervisor – Triangle Labs of Houston, Sugarland, TX. Responsibilities included supervision of operation and maintenance of GCMS/GC, maintenance of DEC and network system, QA/QC for sample analysis (VOA, Semi-VOA, PCB, PAH, Pesticides, Dioxins and Furans) and data packages.

1988-1990 VAX Operator – Canata Research Computer Center, University of Houston, TX. Responsibilities included network maintenance and data backup.

1987-1990 Research Assistant – University of Houston, TX Chemistry Department. Worked on Masters using VG-70SEQ.

Instrument rience/ Computer Skills

Tandem High Resolution Mass Spectrometer Auto Spec-Ultima and VG-70SEQ with CTC-A200 Auto Sampler, VG-70EQ, LC/MS (a Quadropole Mass Spectrometer (HP5988A) fitted with Vestec Thermospray Ion Sources and a Gradient LC system).

GC/MS (HP5972 Mass Spectrometer and HP 5890 GC fitted with O/I analytical model MPM-16 purge and trap and O/I analytical sample concentrator 4560).

HP 5890 GC series II with HP 7673 Automatic sampler.

C and C++, MS Windows 98, Dbase, Q-basic, Opus, OpusQuan, VMS, RXS-11M-PLUS software for VAX, DEC system.

Education M.S. Chemistry, University of Houston, TX 1990

Publications/ Presentations

Unimolecular Dissociation of [Mn2(CO)10]+; November 30 – December 2, 1988, 44th Southwest Regional Meeting, Corpus Christi, TX.

Unimolecular Dissociation of Metastable [Mn2(CO) 10]+ ion ; 37th ASMS May 21 – 26, 1989, Miami Beach, FL.

Collision-Induced Dissociation Spectra of Polyaza Ruthenium Complexes; 37th ASMS May 21-26, 1989, Miami Beach, FL.

Collision-Induced Reaction of Coordinately Unsaturated Dimanese Carbonyl Cluster Fragment Ions and Alkyl Halides in the Gas Phase; 38th ASMS, June 3 – 8, 1990, Tuscon, AZ.

Fast-Atom Bombardment Tandem Mass Spectrometry of (Polypyridyl) Ruthenium (II) Complexes.

Inorg. Chem. Vol. 30, No. 4, 1991

Affiliations American Society of Mass Spectometry

JANE FREEMYER *1998-PRESENT*

Columbia Analytical Services, Inc. 10655 Richmond Avenue, Suite 130A Houston, TX 77042 (713) 266-1599

Current Position

Manager of Quality Assurance Program and Client Services

Responsibilities

As Quality Assurance Program Manager, lead CAS's Quality Assurance Program in the Houston Laboratory. Responsible for reviewing, approving and controlling QA Manual, SOPs, MDLs and IDCs. Coordinate PE samples and Nonconformance Reports. Review analytical data, perform internal audits and assure compliance with external audit findings. Maintain state and federal certifications. Train staff. Prepare quarterly quality assurance reports to senior management.

As Client Services Manager, also responsible for implementing client requirements within the laboratory. Assure project details are understood by technical and administrative staff and assure analytical reports comply with established project requirements.

Experience

Process Control Manager - Southern Petroleum Laboratory, Houston, Texas. Responsible for implementing data validation systems and troubleshooting complex deliverables in a laboratory with a large industrial client base. 1998

Laboratory Director - CH2M Hill Analytical Services, Redding, California. Worked closely with a core management team to re-integrate CH2M Hill Analytical Services into an efficient, productive analytical asset. Took the lead role for installation of a new LIMS system. 1996-1997

Program/Laboratory Director - BC Analytical, Glendale, California. Promoted to progressively responsible leadership positions within the organization. Ultimately responsible for the operation of three laboratory facilities in California. Managed the large lab accounts for optimum performance to contract requirements. 1978-1996.

Chemist - City of Turlock, Water Quality Control, Turlock, California. Process control chemist for the City of Turlock, Water Quality Control, during an upgrade from secondary to tertiary treatment. 1975-1978

Education

University of Phoenix/Online, San Francisco, California - MA in Organizational Management. 1992.
California State University at Stanislaus, Turlock, California - BA in Chemistry 1974.

KAREN L. VERSCHOOR

2002 TO PRESENT

Columbia Analytical Services INC.
An Employee - Owned Company

Columbia Analytical Services, Inc., 10655 Richmond Avenue, Suite 130A, Houston, TX 77042 (713) 266-1599

Current Position

Business Development Manager – 2002 to Present

Responsibilities

Leads the sales, marketing and new business development program for the Houston Laboratory. Responsible for developing new clients, nationally and globally, for dioxin/furan and related analyses. Also responsible for understanding and communicating client expectations to the Houston team.

Experience

Business Development Manager, *XENCO Laboratories, Houston, Texas,* 1999-2002. Responsibilities included providing sales and marketing services. Also responsible for developing sales and marketing program for newly developed dioxin group.

Marketing Manager, *Pace Analytical Services, Houston, Texas,* 1995-1998. Responsibilities included sales of environmental analytical services for petrochemical, refining, industrial and private consulting firms in Houston and surrounding areas.

Marketing Representative, *Huntington Engineering & Environmental, Houston, Texas,* 1993-1994. Responsibilities included sales and marketing for environmental laboratory services for the Houston area.

Education

BS, Chemistry, *Colorado State University, Fort Collins, Colorado,* 1977.

ROLANDO A. DIAZ *MARCH 1999-PRESENT*

Columbia Analytical Services, Inc. 10655 Richmond Avenue, Suite 150, Houston, TX 77042 (713) 266-1599

Current Position

Scientist III, Wet Laboratory. 1999-Present.

Responsibilities

Primary responsibilities include sample login, extraction, clean-up and final concentration. Additionally, act as Safety Officer for the Houston, Texas Laboratory.

Experience

Senior Chemist - Ionics International, Inc., Houston, Texas. Duties predominantly as listed above, without the Safety Officer responsibilities. 1998-1999

Senior Laboratory Technician - Diagnostic Systems Laboratories, Webster, Texas. Iodination of antibodies and antigens with I-125 and testing of final product. Additional duties included operation and maintenance of robotics system, compliance with ISO 9002 standards, and Column Chromatography. 1994-1998

Laboratory Supervisor, Research and Development Chemist - Triangle Laboratories of Houston, Houston, Texas. Supervision of the Wet Chemistry Laboratory, including development of new clean-up methods and sample analysis for dioxin and for both volatile and semi-volatile organics.

Production/Research Technician - Diagnostic System Laboratories, Webster, Texas. Manufacture and test RIA kit components. Also assist in development and improvement of RIA test kits. 1988-1990

Education

University of Texas, Austin, Texas - BA in Microbiology. 1987.
Victoria College, Victoria, Texas - AA in General Studies. 1983.

ublications/ Presentations

"Radio Immuno Assay Kit for the Quantitative Measurement of C-Peptide in Serum," Rama Nrang, Rolando Diaz, and Laura Panzerella, *American Association of Clinical Chemistry/Abstracts*. 1990

ALEX M. VILLALOBOS MARCH 1999-PRESENT

Columbia Analytical Services, Inc. 10655 Richmond Avenue, Suite 150, Houston, TX 77042 (713) 266-1599

Current Position

Scientist IV, Instrumentation Laboratory. 1999-Present.

Responsibilities

GC/MS analysis and interpretation of samples, including review of chromatograms, for EPA methods 8280, 8290, 1613, and 613 and for high and low resolution samples using VG-70*S*. Perform login, data review and report generation, and daily operation of the Extraction Laboratory. Additional responsibilities include routine maintenance, daily tuning, and calibration of GC/MS per method criteria and coordination with other laboratory personnel for sample analysis and project scheduling.

Experience

Instrumentation Analyst/Chemist - Ionics International, Inc., Houston, Texas. Duties predominantly as listed above. 1995-1999

Senior Chemist/Extractionist - Triangle Laboratories of Research Triangle Park, Research Triangle Park, North Carolina. Extraction and fractionation of environmental samples by EPA 8290, 8220, 8270, M23, and 8240 methods. Also responsible for coordination/preparation of daily M8280 testing. 1994-1995

Data Review Chemist, Extraction Laboratory - Triangle Laboratories of Houston, Sugar Land, Texas. Data processing and review for PCDD's/PCDF's following EPA protocols. Also responsible for generation of case narratives and final client reports. 1990-1994

Laboratory Technician - Diagnostic System Laboratories, Webster, Texas. Preparation of steroids and proteins aliquots using 14 PLC analysis. Also responsible for testing radioactive tracer and preparation of buffer solution for laboratory use. 1989-1990

Education

University of Panama, Panama City, Panama, BS in Pharmacy. 1979.

CHRISTOPHER ELHARDT 2002-PRESENT

Columbia Analytical Services, Inc., 10655 Richmond Avenue, Suite 130A, Houston, TX 77042 (713) 266-1599

Current Position **Scientist II** - 2002 to present.

Responsibilities Primary responsibilities included the analysis, reporting, and archiving of data within the dioxin department. Primary responsibility is to perform dioxin analysis within the department. May assist in training analysts and junior scientists. May assist in writing standard operating procedures.

Experience

Environmental Inspector – City of Waco,TX. Responsibilities included : GC and GC/MS analysis of disinfection byproducts; metals analysis on AAflame and graphite furnace; customer service calls as required; prepared SOPs; and wrote and maintained LIMS. 1997-2002. Texas "C" treatment license;.

Chemist III – Texas Department of Health, Austin, TX. Responsibilities included GC/MS analysis of volatiles from Texas indoor air quality program; volatiles from Texas seafood safety program; and EDB in drinking water program.. 1995-1997.

Analyst – Scott & White, Temple. TX. Responsibilities included GC/MS analysis of 8270 semivolatiles, 608 pesticides/PCB, 8260 volatiles, extractions, metals digestions and analysis by ICP, and reviewed reports from other sections of the lab. 1990-1995.

Research Assistant – Baylor University, Waco, TX. Responsibilities included assisted chemistry department chairman in development of moved solid phas affinity matrices and lectured in biochemistry. 1984-1990.

Education

M.S., Lab Technology, San Francisco State University, San Francisco, CA 1978
B.S., Zoology, University of Washington, Seattle, WA 1971

Publications / Presentations

Affiliations American Chemical Society, Texas Surface Water Treatment

CARLOS DIAZ 2001-PRESENT

Columbia Analytical Services, Inc., 10655 Richmond Avenue, Suite 130A, Houston, TX 77042 (713) 266-1599

Current Position **Scientist I** - 2001 to present.

Responsibilities Include sample log-in, preparation, extraction and cleanup.

Experience

Environmental Manager – Global Octanes, Deer Park, TX. Responsibilities included managing all environmental activities of the company. 1991-2000.

Corporate Environmental Manager – Westlake Plymers, Houston, TX. Responsibilities included managing all corporate environmental activities. 1991.

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Education

B.S., Chemistry, University of Texas, Austin, TX 1978

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Publications/ Presentations

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Affiliations

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Appendix C: Data Quality Capabilities

Method 23 & TO9/Dioxins & Furans
Compound Reporting Limits
(last updated: 07/12/02jf)

Congener	CAS RN	Air (pg/trap)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	10
1,2,3,7,8-Pentachlorodibenzo-p-dioxins (PeCDD)	40321-76-4	25
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7	25
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6	25
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3	25
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-39-4	25
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9	50
2,3,7,8-Tetrachlorodibenzofuran (2378-TCDF)	51207-31-9	10
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6	25
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4	25
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9	25
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9	25
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9	25
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5	25
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4	25
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7	25
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	39001-02-0	50

NOTE:
The contract-required limits for Internal Standard percent recovery are 40-130%. The limits for Surrogate Standards are 70-130%.

Method 1613B/Dioxins & Furans
Compound Reporting Limits
(last updated: 12/05/02)

Congener	Congener Abbreviation	CAS RN	REPORTING LIMITS aqueous	REPORTING LIMITS non-aqueous
			PG/L	NG/KG
2,3,7,8-Tetrachlorodibenzo-p-dioxin	2378-TCDD	1746-01-6	10	1
1,2,3,7,8-Pentachlorodibenzo-p-dioxins	12378-PeCDD	40321-76-4	50	5
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	123478-HxCDD	39227-28-6	50	5
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	123678-HxCDD	57653-85-7	50	5
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	123789-HxCDD	19408-74-3	50	5
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	1234678-HpCDD	35822-39-4	50	5
Octachlorodibenzo-p-dioxin	OCDD	3268-87-9	100	10
2,3,7,8-Tetrachlorodibenzofuran	2378-TCDF	51207-31-9	10	1
1,2,3,7,8-Pentachlorodibenzofuran	12378-PeCDF	57117-41-6	50	5
2,3,4,7,8-Pentachlorodibenzofuran	23478-PeCDF	57117-31-4	50	5
1,2,3,6,7,8-Hexachlorodibenzofuran	123478-HxCDF	70648-26-9	50	5
1,2,3,7,8,9-Hexachlorodibenzofuran	123678-HxCDF	57117-44-9	50	5
1,2,3,4,7,8-Hexachlorodibenzofuran	123789-HxCDF	72918-21-9	50	5
2,3,4,6,7,8-Hexachlorodibenzofuran	234678-HxCDF	60851-34-5	50	5
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1234678-HpCDF	67562-39-4	50	5
1,2,3,4,7,8,9-Heptachlorodibenzofuran	1234789-HpCDF	55673-89-7	50	5
Octachlorodibenzofuran	OCDF	39001-02-0	100	10

NOTE:
Soil samples are reported on a dry-weight basis and tissue samples are reported on a wet-weight basis.

SW-846 Method 8290/Dioxins & Furans
Compound Reporting Limits
(09/29/00)

Congener	CAS RN	Water (pg/L)	Soil (ng/Kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	10	1.0
1,2,3,7,8-Pentachlorodibenzo-p-dioxins (PeCDD)	40321-76-4	25	2.5
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7	25	2.5
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6	25	2.5
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3	25	2.5
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-39-4	25	2.5
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9	50	5.0
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9	10	1.0
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6	25	2.5
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4	25	2.5
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9	25	2.5
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9	25	2.5
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9	25	2.5
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5	25	2.5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4	25	2.5
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7	25	2.5
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	39001-02-0	50	5.0
PCDD/PCDF Screening Method 4425 or ASTM E1853M-98			
Dioxin/Furan Mixture		500	25

SW-846 Method 8280A/Dioxins & Furans
Compound Reporting Limits
(09/15/00)

Congener	CAS RN	Water (ng/L)	Soil (ug/Kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	10	1.0
1,2,3,7,8-Pentachlorodibenzo-p-dioxins (PeCDD)	40321-76-4	25	2.5
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7	25	2.5
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6	25	2.5
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3	25	2.5
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-39-4	25	2.5
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9	50	5.0
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9	10	1.0
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6	25	2.5
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4	25	2.5
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9	25	2.5
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9	25	2.5
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9	25	2.5
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5	25	2.5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4	25	2.5
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7	25	2.5
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	39001-02-0	50	5.0
PCDD/PCDF Screening Method 4425 or ASTM E1853M-98			
Dioxin/Furan Mixture		500	25

Method 1668A/WHO-12
Coplanar PCB Congeners
Method Reporting Limits

Congener	IUPAC #	TEF **	Water (pg/L, ppq)	Solid (ng/Kg, ppt)
3,3',4,4'-Tetrachlorobiphenyl	PCB 77	0.0001	500	50
3,4,4',5-Tetrachlorobiphenyl	PCB 81	0.0001	500	50
2,3,3',4,4'-Pentachlorobiphenyl	PCB 105	0.0001	200	20
2,3,4,4',5-Pentachlorobiphenyl	PCB 114	0.0005	500	50
2,3',4,4',5-Pentachlorobiphenyl	PCB 118	0.0001	500	50
2',3,4,4',5-Pentachlorobiphenyl	PCB 123	0.0001	500	50
3,3',4,4',5-Pentachlorobiphenyl	PCB 126	0.1	500	50
2,3,3',4,4',5-Hexachlorobiphenyl	PCB 156	0.0005	500	50
2,3,3',4,4',5'-Hexachlorobiphenyl	PCB 157	0.0005	500	50
2,3',4,4',5,5'-Hexachlorobiphenyl	PCB 167	0.00001	500	50
3,3',4,4',5,5'-Hexachlorobiphenyl	PCB-169	0.01	500	50
2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB 189	0.0001	500	50

NOTE: MRL values are taken directly from Method 1668A. Soil/sediment samples are reported on a dry-weight basis and tissue samples are reported on a wet-weight basis.

**TEF (Toxicity Equivalence Factors) are from the World Health Organization.

Method 1668A/Coplanar PCB Congeners
Total Homologues
Method Reporting Limits

Total Homolgue	Water (pg/L, ppq)	Soil/Tissue (ng/Kg, ppt)
Monochlorobiphenyl	500	50
Dichlorobiphenyl	500	50
Trichlorobiphenyl	500	50
Tetrachlorobiphenyl	500	50
Pentachlorobiphenyl	500	50
Hexachlorobiphenyl	500	50
Heptachlorobiphenyl	500	50
Octachlorobiphenyl	500	50
Nonachlorobiphenyl	500	50
Decachlorobiphenyl	500	50

NOTE: MRL values are taken from Method 1668A. Soil/sediment samples are reported on a dry-weight basis and tissue samples are reported on a wet-weight basis.

— Example —

[Form 1]

CLIENT ID.

Method Blank

PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

Lab Name: Columbia Analytical Services Episode No.:

Lab Code: CAS SDG No.: Method: 8290 Lab Sample ID: EB70092-MB

Client Name: Sample Wt/Vol: 10.00 g or mL: g

Matrix (aqueous/solid/leachate): solid Initial Calibration Date: 10/02/00

Sample Receipt Date: Instrument ID: 70S

Ext. Date: 11/07/00 GC Column:DB-5

Ext. Vol(ul):20.0 Inj. Vol(ul):2.0 Sample Data Filename: A17040#1

Analysis Date: 15-NOV-00 Time: 14:17:25 Blank Data Filename: A17040#1

Dilution Factor: 1 Cal. Ver. Data Filename: A17039#1

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture:

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	Qual. (1)	ION ABUND. RATIO (2)	RRT (2)	MEAN RRF
2,3,7,8-TCDD	*	0.094	U	*	*	0.93
1,2,3,7,8-PeCDD	*	0.094	U	*	*	0.99
1,2,3,4,7,8-HxCDD	*	0.085	U	*	*	0.95
1,2,3,6,7,8-HxCDD	*	0.079	U	*	*	1.02
1,2,3,7,8,9-HxCDD	*	0.079	U	*	*	1.03
1,2,3,4,6,7,8-HpCDD	*	0.114	U	*	*	0.86
OCDD	1.489	0.137	J	0.87	1.169	0.90
2,3,7,8-TCDF	*	0.135	U	*	*	0.88
1,2,3,7,8-PeCDF	*	0.066	U	*	*	0.97
2,3,4,7,8-PeCDF	*	0.067	U	*	*	0.95
1,2,3,4,7,8-HxCDF	*	0.058	U	*	*	1.08
1,2,3,6,7,8-HxCDF	*	0.058	U	*	*	1.08
1,2,3,7,8,9-HxCDF	*	0.076	U	*	*	0.82
2,3,4,6,7,8-HxCDF	*	0.064	U	*	*	0.96
1,2,3,4,6,7,8-HpCDF	*	0.095	U	*	*	1.30
1,2,3,4,7,8,9-HpCDF	*	0.140	U	*	*	0.88
OCDF	*	0.164	U	*	*	1.15
Total Tetra-Dioxins	*	0.094	U			
Total Penta-Dioxins	*	0.094	U			
Total Hexa-Dioxins	*	0.079	U			
Total Hepta-Dioxins	*	0.114	U			
Total Tetra-Furans	*	0.135	U			
Total Penta-Furans	*	0.067	U			
Total Hexa-Furans	*	0.058	U			
Total Hepta-Furans	*	0.095	U			

(1) Qualifiers: See flag definitions.

(2) RRTs and ion ratios are specified in Tables 11 and 8, Method 8290.

8290F1

- Example -

[FORM 2]

PCDD/PCDF LABELED COMPOUND AND
CLEANUP STANDARD RECOVERIES

CLIENT ID.

Method Blank

Lab Name: Columbia Analytical Services Episode No.:

Lab Code: CAS SDG No.: Method: 8290 Lab Sample ID: EB70092-MB

Client Name: Sample Wt/Vol: 10.00 g or mL: g

Matrix (aqueous/solid/leachate): solid Initial Calibration Date: 10/02/00

Sample Receipt Date: Instrument ID: 70S

Ext. Date: 11/07/00 GC Column: DB-5

Ext. Vol(ul): 20.0 Inj. Vol(ul): 2.0 Sample Data Filename: A17040#1

Analysis Date: 15-NOV-00 Time: 14:17:25 Blank Data Filename: A17040#1

Dilution Factor: 1 Cal. Ver. Data Filename: A17039#1

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture:

	SPIKE CONC. (pg)	CONCENT. FOUND (pg)	RECOV. %	Q	ION ABUND. RATIO(2)	RRT (2)	MEAN RRF
LABELED COMPOUNDS							
13C-2,3,7,8-TCDD	1000	645.04	64.50		0.80	1.008	1.07
13C-1,2,3,7,8-PeCDD	1000	781.72	78.17		1.56	1.178	0.78
13C-1,2,3,6,7,8-HxCDD	2500	1900.72	76.03		1.23	0.993	0.91
13C-1,2,3,4,6,7,8-HpCDD	2500	1963.00	78.52		1.02	1.076	0.78
13C-OCDD	5000	4085.91	81.72		0.87	1.169	0.51
13C-2,3,7,8-TCDF	1000	551.17	55.12		0.78	0.976	1.67
13C-1,2,3,7,8-PeCDF	1000	746.79	74.68		1.55	1.138	1.21
13C-1,2,3,4,7,8-HxCDF	2500	1728.09	69.12		0.50	0.971	1.46
13C-1,2,3,4,6,7,8-HpCDF	2500	1658.54	66.34		0.43	1.049	0.96
CLEANUP STANDARD							
37Cl-2,3,7,8-TCDD	800					1.009	

1) Contract-required limits for percent recovery are 40%-135% (section 8.4, Method 8290).

2) Contract-required Reference Attributions for RRTs and ion abundance ratios are specified in Tables 11 and 8, respectively, Method 8290.

NOTE: There is no ion abundance ratio for 37Cl4-2378-TCDD (cleanup standard).

PCDD/PCDF SPIKED SAMPLE SUMMARY

EPA SAMPLE NO.

LCS

Lab Name: COLUMBIA ANALYTICAL SERVICES Contract:

Client: Case No: LAB. ID.: EB14034-LCS SDG No

Matrix: Solid (Aqueous/Solid/Leachate/Ash/Air)

CONCENTRATION UNITS: (pg/L or ng/Kg) ng/Kg

ANALYTE	SPIKE ADDED (PG)	LCS CONCEN.	SAMPLE CONCEN.	% REC #	QC LIMITS
2378-TCDD	200	21.934	0.000	109.7	70-130
12378-PeCDD	500	49.458	0.000	98.9	70-130
123478-HxCDD	500	53.154	0.000	106.3	70-130
123678-HxCDD	500	48.594	0.000	97.2	70-130
123789-HxCDD	500	44.719	0.000	89.4	70-130
1234678-HpCDD	500	56.655	0.000	113.3	70-130
OCDD	1000	95.267	0.000	95.3	70-130
2378-TCDF	200	21.647	0.000	108.2	70-130
12378-PeCDF	500	50.716	0.000	101.4	70-130
23478-PeCDF	500	49.949	0.000	99.9	70-130
123478-HxCDF	500	50.431	0.000	100.9	70-130
123678-HxCDF	500	52.758	0.000	105.5	70-130
123789-HxCDF	500	49.423	0.000	98.8	70-130
234678-HxCDF	500	49.102	0.000	98.2	70-130
1234678-HpCDF	500	51.419	0.000	102.8	70-130
1234789-HpCDF	500	56.698	0.000	113.4	70-130
OCDF	1000	103.216	0.000	103.2	70-130

Appendix D: Data Qualifiers

ABBREVIATIONS, ACRONYMS AND FLAGS

Abbreviations, acronyms and definitions

Cal	Calibration
Conc	Concentration
Dioxins	Polychlorinated dibenzo-p-dioxin(s)
EDL	Estimated detection limit
EMPC	Estimated maximum possible concentration
Flags	Data qualifiers
Furans	Polychlorinated dibenzofuran(s)
g	Grams
ICAL	Initial calibration
ID	Identifier
Ions	Masses monitored for the analyte during data acquisition
L	Liter (s)
LCS	Laboratory control sample
LCSD	Laboratory control spike duplicate
MB	Method blank
MDL	Method detection limit
ML	Mililiters
MS	Matrix spike sample
MSD	Matrix spiked sample duplicate
NO	Number of peaks meeting all identification criteria
PCDD	Polychlorinated dibenzo-p-dioxin(s)
PCDF	Polychlorinated dibenzofuran(s)
ppm	Parts per million
ppb	Parts per billion
ppt	Parts per trillion
ppq	Parts per quadrillion
QC	Quality control
Ratio	Ratio of areas from monitored ions for an analyte
% Rec.	Percent recovery
RPD	Relative percent difference
RRF	Relative response factor
RT	Retention time
SDG	Sample delivery group
S/N	Signal-to-noise ratio
TEF	Toxicity equivalence factor
TEQ	Toxicity equivalence

(Flags – see next page)

Data Qualifier Flags

[*CAS/Houston* data qualifiers - adapted from EPA's Contract Laboratory Program data qualifiers]

- **B** Used when the analyte is found in the associated blank, as well as in the sample
- **C** Indicates the value for the TCDF analyte was obtained by analysis using a DB-225 confirmation column
- **E** Indicates an estimated value
  Used when the analyte concentration exceeds the upper end of the linear calibration range
- **J** Indicates an estimated value
  Used when the analyte concentration is below the method reporting limit (MRL) and above non-detect.
- **K** EMPC; estimated maxiumum possible concentration
- **U** Indicates the compound was analyzed and not detected.
- **X** User defined; see case narrative for detailed explanation
- **Y** Indicates the recovery of the labeled standard is outside the established control limits

Appendix E: Preventive Maintenance Procedures

Preventive Maintenance Procedures

Instrument	Activity	Frequency
Refrigerators and coolers	▪ Record temperatures ▪ Clean coils ▪ Check coolant	▪ Daily ▪ Annually ▪ Annually
Vacuum Pumps	Clean and change pump oil	Monthly
Fume Hoods	▪ Face velocity measured ▪ Sash operation ▪ Change filters ▪ Inspect fan belts	▪ Quarterly ▪ As needed ▪ Annually ▪ Annually
Ovens	Clean	Annually
Analytical Balances	▪ Check alignment ▪ Check calibration ▪ Clean pans	▪ Before each use ▪ Before each use ▪ After each use
GCMS/High Resolution	▪ Check gas supplies	▪ Daily, replace when pressure reaches 50psi
	▪ Change in-line filters	▪ Quarterly
	▪ Change septum	▪ Daily
	▪ Change injection port liner	▪ As needed
	▪ Clip first foot of capillary column	▪ As needed
	▪ Change guard column	▪ As needed
	▪ Replace analytical column	▪ As needed
	▪ Clean source	▪ As needed
	▪ Change pump oil	▪ Every six months

Appendix F: Certifications/Accreditation/Contracts

Columbia Analytical Services, Inc.

Houston, Texas

Last updated: 04/26/04

Laboratory Certifications
2004 - 2006

STATE/PROGRAM	CERT#	SDWA CERT#	DATE EXPIRES	CERTIFIED?
ARKANSAS	88-0635	N/A	04/18/05	Yes
CALIFORNIA	2452	2452	02/28/05	Yes
CONNECTICUT	PH-0300	PH-0300	12/31/05	Yes
FLORIDA/NELAP	E87611	E87611	06/30/04	Yes
HAWAII	--	--	06/30/04	Yes
KANSAS/NELAP	E10333	E10333	07/31/04	Yes
LOUISIANA/NELAP	03048	N/A	06/30/04	Yes
MAINE	TX901	N/A	06/05/05	Yes
NEW YORK/NELAP	11707	11707	04/01/05	Yes
NFESC/NAVY	N/A	N/A	05/21/04	Yes
OREGON/NELAP	TX200002	TX200002	02/19/05	Yes
SOUTH CAROLINA	82014	N/A	06/30/04	Yes
UTAH/NELAP	COLU2	COLU2	08/31/04	Yes
WASHINGTON	C291	N/A	11/14/04	Yes

APPENDIX B

Cooler Receipt and Preservation Check Form

Client: _____ Work order: _____

Project: _____

Cooler received on: _____ and opened on _____ by _____

		Yes	No	N/A
1	Were custody seals on outside of cooler?	☐	☐	☐
	If yes, how many and where? _____			
	Were signature and date correct?	☐	☐	☐
2	Were custody papers properly filled out (ink, signed, etc....)?	☐	☐	☐
3	Did all bottles arrive in good condition (unbroken, etc....)?	☐	☐	☐
4	Were all bottle labels correct (analysis, preservation, etc....)?	☐	☐	☐
5	Did all bottle labels and tags agree with custody papers?	☐	☐	☐
6	Were correct bottles used for test indicated?	☐	☐	☐
7	Were VOA vials checked for absence of air bubbles, and noted?	☐	☐	☐
8	Temperature of cooler upon receipt	_____ Degrees C		

Explain any discrepancies: _____

		Yes	No
pH	Reagent	x	
12	NaOH	x	
2	HNO_3	x	
2	H_2SO_4	x	

Yes = all samples OK
No = Samples were preserved at lab as listed

Comments: _____

Sample I.D.	Reage	Vol.