User's Guide to the Contract Laboratory Arogram

FOREWORD

This document has been prepared by the CLP Sample Management Office specifically for the guidance and direction of program clients.

The organic and inorganic analytical program descriptions herein outline the requirements and analytical procedures of the new CLP protocols developed from technical caucus recommendations. These protocols were implemented into CLP analysis contracts in 1985. Other analytical programs, procedures and documentation described herein reflect the status of the program as of July 1986. Critical information for CLP samplers and user groups is contained in Chapter III and Appendix C. This information should be distributed to all contractors collecting samples for the CLP and to each user group of the EPA and of the States.

Updated User's Guide sections containing changes to CLP analytical programs, procedures and documentation will be provided to clients periodically, in the form of User's Guide amendments. For further information on the CLP or to obtain additional copies of the User's Guide, contact the Sample Management Office at 703/557-2490 or FTS 557-2490.

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CLP USER'S GUIDE

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CHAPTER I

BACKGROUND AND INTRODUCTION

CHAPTER I BACKGROUND AND INTRODUCTION

The purpose of this chapter is to present the basic Contract Laboratory Program (CLP) objective and orientation, and to familiarize the reader with program structure. This background information is provided to facilitate better understanding and more efficient utilization of program services.

A. CLP Objective and Orientation

The CLP supports the Agency's Superfund effort, originally under the 1980 Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and presently under the 1986 Superfund Amendment and Reauthorization Act (SARA) by providing a range of state-of-the-art chemical analysis services of known quality on a high-volume, cost-effective basis. The central and overriding assumption governing the structure and function of the CLP is the basic requirement to provide legally-defensible analytical results for use in supporting Agency enforcement actions. Therefore, a high level of quality assurance and documentation has been incorporated in all aspects of program activities.

The ongoing CLP objective is to develop, manage and improve its analytical programs in support of all Superfund requirements. This objective is accomplished by continuously increasing analytical capacity and adjusting analytical program requirements and related support services to better meet Agency needs.

The CLP supplies analytical services in direct response to requests from the EPA Regions, the primary users of the program, as well as states and other EPA programs, such as RCRA, which have become part of the CLP user community. The CLP is a service program designed to provide a wide range of enforcement-quality analytical services in response to the changing needs and requirements of the user community. This client orientation is a key factor in the design and application of all CLP services and responses.

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B. CLP Structure

The CLP effort involves numerous Agency, contractor and other groups throughout the country. These organizations are identified and their role in the program described in the following sections. The following table, Interrelationships of Program Principals, graphically illustrates the interaction of these groups in the CLP operation. In addition, Appendix A is a program directory containing addresses and telephone numbers of key program personnel.

1. Program Management

a. National Program Office (NPO)

The CLP is directed by the National Program Office, in EPA Headquarter's Analytical Support Branch (ASB), Hazardous Response Support Division (HRSD), Office of Emergency and Remedial Response (OERR), in Washington, DC. The NPO is comprised of the National Program Manager; Organic, Inorganic and Dioxin Technical Project Officers (PO); the Sample Management Office Project Officer; and a Quality Assurance (QA) Officer.

NPO responsibilities include: overall management of the CLP in terms of program objectives, expansion and interface with clients and other groups; policy and budget formation and implementation; administration of analytical and support contracts; development and technical review of analytical protocols; review of special analytical services subcontracts and CLP-generated laboratory data; development of CLP analytical and support services contracts; monitoring and formal evaluation of analytical and support contractors; and, direction of CLP quality assurance (QA) in coordination with overall OERR QA activities.

The National Program Manager (NPM), in addition to directing program staff, is responsible for the formulation of program policies

INTERRELATIONSHIP OF PROGRAM PRINCIPALS



and direction; communicates with the Regional and Agency communities on a continuing basis, keeping all parties apprised of program activities and receiving input on program effectiveness.

The Technical Project Officers (POs) are responsible for technical program decisions, contract administration and contractor performance evaluation. The POs work closely with the Regional Deputy Project Officers (DPOs) and laboratories on a daily basis in resolving technical issues. The POs direct the ongoing effort to improve contract language and analytical methodologies, and conduct technical caucuses for purposes of CLP data and protocol review.

The Sample Management Office (SMO) Project Officer is responsible for the administration of the SMO contract as well as overseeing the overall supply/demand balance between CLP contracts and client needs. This PO is also responsible for the administration of the Sample Bottle Repository contract.

The Quality Assurance (QA) Officer coordinates all aspects of program application of QC procedures. The QA Officer works closely with EPA Headquarters Office of Research and Development (ORD) and the ORD's Environmental Monitoring Systems Laboratory in Las Vegas (EMSL/LV) which provides QA support to the CLP. The QA Officer also coordinates with the POs and EMSL/LV in refining and updating analytical method QC procedures.

b. Sample Management Office (SMO)

The contractor-operated Sample Management Office functions in direct support of the NPO, providing management, operations and administrative support to the CLP. The primary objective of the SMO operation is to facilitate optimal use of program analytical resources. SMO activities fall into the following areas: (1) sample scheduling and tracking; (2) Contract Compliance Screening; (3) Special Analytical Services subcontracting; (4) laboratory invoice processing; (5) maintenance of CLP records and management reporting; and (6) NPO management, technical and administrative support.

SMO routinely receives analytical requests from the Regions, coordinates and schedules sample analyses, tracks sample shipment and analyses, receives and checks data for completeness and compliance, processes laboratory invoices, and maintains a repository of sampling records and program data. In response to client requests for nonroutine types of analyses, SMO subcontracts for Special Analytical Services (SAS), performing scheduling and tracking for SAS efforts as outlined above. SMO maintains a comprehensive data base of CLP services, performance and utilization, and generates a variety of management and user reports.

c. USEPA Office of Research and Development (ORD), Environmental Monitoring Systems Laboratory/Las Vegas (EMSL/LV)

Program quality assurance support is provided by EPA ORD through EMSL/LV. EMSL/LV functions as the quality assurance arm of the CLP, providing advice and support to the NPO. Specifically, EMSL/LV assists in performing pre-award and post-award on-site laboratory evaluations; prepares performance evaluation (PE) samples for pre-award and post-award evaluations of laboratory performance; evaluates pre-award and post-award PE sample data; and performs QA audits on CLP-generated data. Additionally, EMSL/LV is responsible for: providing analytical reference standards to program laboratories through the contractor-operated QA Materials Bank; operating the program's QA Database, performing program and laboratory trend analyses used in developing and updating contract QC criteria; directing operation of the Superfund Quality Assurance Support Laboratory (QASL) at the University of Nevada, Las Vegas; and assisting in evaluation of CLP analytical methods and protocols.

d. National Enforcement Investigations Center (NEIC)

The NEIC advises the NPO in defining and applying program enforcement requirements. NEIC developed sample custody procedures, chain-of-custody records, sample tags, and custody seals; which are utilized in the CLP to maintain the validity of sample analyses for supporting Agency enforcement actions. NEIC routinely performs evidence audits of CLP laboratories and generates sample profiles used in Agency enforcement litigation. A description of NEIC's evidence audit process appears in Chapter IV, Section C.

2. Regional Program Support

The Regions play an integral role in program activities, both as the primary CLP user and as a key part of analytical program management. The decentralization of program responsibilities to the Regions has evolved with the expansion of the program, as a means to more effectively direct program operations nationwide. Extended Regional participation in the program has and will continue to increase the program's responsiveness to Superfund requirements.

a. Contract Deputy Project Officers

In January, 1984, Regional Administrators appointed a CLP technical Deputy Project Officer (DPO) for each Regional office. Under direction of the NPO, the Regional DPO assumes a portion of the responsibility for monitoring the laboratory contractors physically located in the Region. The DPO works closely with the NPO Project Officer in responding to identified problems in laboratory operations and participates in laboratory on-site evaluations.

b. Regional Sample Control Centers

In January, 1984, each Region established a Regional Sample Control Center (RSCC) to centralize ordering of CLP sample analyses within the Region. The RSCC is comprised of three or more individuals designated as CLP Authorized Requestors, with one individual named as the Primary Authorized Requestor (AR) directing the RSCC. The RSCC is responsible for coordinating the level of Regional sampling activities to correspond with monthly allocations of CLP analyses, where applicable. The Primary AR makes final determinations regarding Regional analysis priorities when conflicts occur. RSCC ARs routinely place all Regional requests for CLP analyses, coordinate with SMO during sampling and sample shipment, and resolve any problems which arise concerning the samples. The RSCC serves as the central point of contact for questions concerning Regional sampling efforts.

c. Technical Caucuses

In September 1982, the NPO implemented the concept of Technical Caucus sessions as a means to consistently utilize the scope of available technical resources in updating analytical program methodologies and data reporting requirements. Technical caucuses are held on a periodic basis and involve participation of the following groups: EPA Regions, EMSL/LV, EMSL/Cincinnati, NEIC, contract laboratories, program support contractors, SMO, NPO and others, as appropriate. These caucuses have been instrumental in improving CLP protocols and orienting deliverables directly to user needs.

d. Regional/Laboratory Communication System

In January 1983, the NPO established a system of direct communication between the Regions and contract laboratories, as a routine method for Regional data review staff to obtain answers to technical questions concerning program data in the most timely and direct manner possible. In this system, designated Regional communi-cation contacts call designated laboratory communication contacts as needed to resolve technical data questions. This communication link also benefits the laboratory by providing direct feedback on its data product.

3. <u>Clients/Users</u>

a. EPA Regions

The ten EPA Regions are the primary clients of the CLP. As described in the previous section, each Region has established an RSCC, which schedules all CLP analyses requests for the Region, coordinating Regional sampling to balance with allocated numbers of CLP sample analyses available each month, and prioritizing the Region's analytical workload when conflicts occur. RSCC personnel coordinate closely with SMO throughout Regional sampling events, assisting in tracking sample shipments to the laboratory and resolving any problems that arise. In this role, the RSCC also processes analytical requests from state or other program users that are located in the Region's geographical area.

b. States

Under RCRA - CERCLA Cooperative Agreements, any state undertaking initial site investigations and entering into cooperative agreements with the government for cleanup of local waste sites, can utilize CLP services. States must access CLP analytical services th ough the RSCC and data packages are distributed to states through the RSCC.

c. Non-Superfund Clients

Program services are available to support non-Superfund clients on a "noninterfering" basis. Non-Superfund analyses and other CLP support are provided by the CLP through transfer of funds from the non-Superfund program to the CLP. Non-Superfund clients currently include other government agencies and other EPA programs, such as Office of Acid Disposition, Office of Solid Waste, and the National Dioxin Study.

4. Analytical and Support Contractors

a. Contract Analytical Laboratories

The CLP's analysis contractors come from the nationwide community of chemical analytical laboratory facilities. To become part of the CLP, laboratories must meet stringent requirements and standards for equipment, personnel, laboratory practices, analytical operations, and quality control operations. Firm, fixed-price contracts are awarded competitively to the lowest responsive, responsible bidders through the government's Invitation for Bid (IFB) process. Lowpriced bidders must successfully analyze performance samples and pass a pre-award laboratory audit before a contract is awarded. After contract award, laboratories are closely monitored to assure compliance with the terms and conditions of the contract. Details of pre-award and post-award evaluations are addressed in Chapter V.

b. Sample Bottle Repository

The Superfund Sample Bottle Repository program was established by the NPO in May 1982 to provide a common source of clean, QCtested sampling containers for samples processed through the CLP. The objective of the program is to eliminate the potential of bottle contamination that would affect the validity of sample data. The contractor-operated repositories serve as a central source for several types of pre-cleaned sample bottles which are routinely utilized by Regional and contract personnel performing Superfund sampling activities. Repository services are detailed in Chapter IV.

CHAPTER II

DESCRIPTION OF ANALYTICAL SERVICES

CHAPTER II DESCRIPTION OF ANALYTICAL SERVICES

The Contract Laboratory Program provides standardized and specialized analytical services to support a variety of Superfund sampling activities, from those associated with the smallest preliminary site investigation to those of large-scale, complex remedial, monitoring and enforcement actions. In response to the increasing analytical demands of its client base, the CLP has continually expanded its analytical capacity for standardized analyses through frequent IFB solicitations. On the average, the CLP is able to provide over 6,000 sample analyses per month through its routine and specialized analytical services programs. The CLP will continue to adjust analytical capabilities and capacity in response to Regional client needs.

The CLP operates four separate analytical programs:

- o Organic Routine Analytical Services (RAS),
- o Inorganic RAS,
- o Dioxin RAS, and
- o Special Analytical Services (SAS).

Organic, inorganic and dioxin RAS program analyses are performed by a network of laboratories operating under firm, fixed-price contracts with the EPA, which provide analytical services to Superfund clients. The SAS program provides unique, nonstandardized analytical services to Superfund and non-Superfund clients for organic, inorganic, dioxin and other compounds in a variety of matrices, to meet specific analytical requirements which do not fall under RAS programs. SAS services are provided through individual fixed-price subcontracts awarded to qualified laboratories.

The two tables which follow outline the menu of services available under the CLP's RAS and SAS programs. The remainder of Chapter II describes each analytical program in terms of:

- o Sample matrices, concentration levels and volumes required.
- o Compounds identified and quantified.
- o Contract deliverable requirements.

Table 2

Category	RAS Organic Analysis	RAS Inorganic Analysis	RAS Dioxin Analysis
Sample Matrices	Low & Medium Concentration Water & Soil/Sediment Samples	Low & Medium Concentration Water & Soil/Sediment Samples	Low & Medium Concentration Soil/Sediment Samples
Compounds Identified & Quantified	HSL Compounds & Library Matches of 30 Highest Compounds (In the ppb Range)	Metals & Cyanide (In the ppb Range)	2,3,7,8-TCDD (In the ppb Range)
Deliverables	Extraction in 5 Days for H ₂ O & 10 Days for Soil Samples VOA Analysis in 10 Days for H ₂ O & 10 Days for Soil Samples Data Delivery in 30, 40 or 45** Days	Data Delivery in 30 or 35** Days	Data Delivery in 15 or 30 Days Automatic Rerun Data 10 Days Following Initial Data Due Date
Analytical Procedures	GC/MS Analysis Following Sample Preparation/Extraction	Flame/Flameless & Cold Vapor AA, ICP & Colorimetric Analysis	GC/MS Analysis by FSCC Following Solvent Extraction/Clean-Up
QA/QC	Surrogate Spike in Each Sample MS & MS Duplicate Per 20 Samples, Per Case, For Each Matrix & Concentration On Per-Fraction Basis	Matrix Spike (MS) & Duplicate Per Case,* Per 20 Samples For Each Matrix & Concentration	MS & Duplicate Per Batch of 24 Samples or Less

MENU OF ROUTINE ANALYTICAL SERVICES

*A Case designates a group of samples collected at one site or geographical location during a specific finite period of time.

**As specified by contract schedule.

Table 3 MENU OF SPECIAL ANALYTICAL SERVICES (SAS)

RAS Plus SAS Category		All SAS Category		
E	Examples of Services Available:		Examples of Services Available:	
o	Fast Turnaround Analysis by RAS Organic, Inorganic or Dioxin IFB Protocol	0	Organic Analysis Per Non-RAS Protocols, Matrices, Compounds	
o	RAS/Organic Analysis with Additions/Adjustments to IFB Protocols.	0	Inorganic Analysis Per Non-RAS Protocols, Matrices, Compounds	
o	RAS Inorganic Analysis with Additions/Adjustments to IFB Protocol	o	Dioxin Analysis Per Non-RAS Protocols, Matrices, Compounds	
ο	RAS Dioxin Analysis with Additions/Adjustments to IFB Protocol	0	Organic and Inorganic High Concentration Sample Preparation and Analysis	
		о	Special Topics Analysis (As Requested)	

NOTE: The client requestor is responsible for designating IFB method adjustments in RAS Plus SAS work and for supplying suitable analytical protocols for All SAS work. Additionally, the client must provide QA/QC procedures and criteria, and must specify analysis and data reporting delivery schedules. This information must accompany the client's request for SAS services.

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- o Description of analytical protocols and detection limits.
- o Contract quality control requirements.

The organics and inorganics RAS sections present the caucus-revised protocols implemented in 1985.

The client should carefuly consider the provisions of each CLP analytical program during the planning stages of a sampling event to determine the applicability of the analysis to user needs.

In addition to this Guide, Regional DPOs maintain a Master Copy notebook of each Statement of Work (SOW) under which CLP RAS laboratory contractors operate. Users are instructed to consult the Region's Master Copy SOWs for detailed analytical information.

A. Organic Routine Analytical Services (RAS)

1. Sample Matrices, Concentration Levels and Volumes Required

The organic RAS contract methods apply to analysis of water (aqueous) and soil/sediment samples. Samples for analysis should be single-phase, homogeneous samples of a similar matrix. Sample matrices other than water, sediment or soil are processed through the SAS program.

Organic RAS contract methods determine concentrations of organic compounds ranging from low or environmental levels of concentration to medium levels, where a compound may comprise as much as 15 percent of the total sample, at the lowest appropriate detection limits. Low level samples are considered to be those collected off-site, around the perimeter of a waste site, or in areas where hazards are thought to be significantly reduced by normal environmental processes. Medium level samples are most often those collected on-site, in areas of moderate dilution by normal environmental processes. Low and medium level designations are made in the field by the sampler to determine packaging and shipment procedures only. Low and medium level analysis designations are performed within the laboratory to determine the appropriate analytical protocol to be used. For soil/sediment samples only, there are separate procedures for semivolatiles organics analysis depending upon whether the samples are determined to be low (>20 ppm) or medium (\geq 20 ppm) concentration.

The sample volume and container types required for RAS organic analysis vary according to the matrix and estimated concentration level of the sample. For RAS organic analysis of a water sample estimated as low level, one gallon sample volume is required for extractables - base, neutral, acid (B/N/A) and pesticides/PCB, and 80 ml for volatiles (VOA). The extractables sample is collected in two 80-ounce amber glass bottles, four 1-liter amber glass bottles, or one 4-liter amber glass bottle. The volatiles sample is collected in two 40-ml glass vials. For RAS organics analysis of a water sample estimated as medium level, a four liter volume is required for extractables and 80 ml for volatiles. The extractables sample in two 40-ml glass vials. For RAS organics analysis of a soil/sediment sample estimated as low or medium level, a six ounce volume is required for extractables. The sample should be collected in one 8-ounce glass jar for extractables and two 120-ml glass vials for volatiles.

For a laboratory to perform matrix spike, matrix spike duplicates, and contractual reanalyses, triple the sample volume is required in at least one sample in twenty for each sample with the same concentration/matrix. Additionally, for water samples, one field blank should be supplied per Case, and one volatile trip blank should be supplied per shipment. No additional volume is required for soil sample duplicate analyses. Soil blanks are supplied to Regions by EMSL/LV. Use of aqueous blanks for soil samples is not appropriate.

For shipping purposes, each sample estimated as medium level (water or soil) must be enclosed and sealed in a metal paint can for shipment. Because it is not certain whether a sample is actually low or medium level, volume should be collected as specified for low level samples, however shipping procedures must be followed as designated for medium level samples.

Sample portions for volatile analysis (water and soil) should be collected so that the containers are completely filled, leaving no headspace.

If sufficient sample volume is not provided, completion of all required parameters and/or complete QA/QC determinations may not be possible. If this occurs, SMO will contact the RSCC to determine appropriate adjustments in analysis.

Required sample volumes and container types for RAS organic analysis of water and soil samples are illustrated in Appendix C. Pre-cleaned sample bottles are available through the Sample Bottle Repository, as detailed in Chapter IV.

2. Compounds Identified and Quantified

The organic RAS program provides identification and quantification of EPA Target Compound List (TCL) (previously termed the Hazardous Substances List -- HSL) organic compounds (VOA, B/N/A, and pesticide/PCB fractions) in water and soil/sediment samples. These compounds, which include priority pollutant compounds and other organics of interest, are identified on the organic data reporting sheets in Appendix B.

In addition, the laboratory is required to execute a maximum of 30 NBS Mass Spectral Library searches for compounds not identified on the TCL (HSL). The 10 peaks of greatest apparent concentration in the volatile fraction and the 20 peaks in the base/neutral/acid fraction are tentatively identified and the concentration estimated, following a visual comparison of sample spectra with the nearest library matches. The tentative identification of non-TCL (HSL) organic compounds provides information on potential organic contaminants outside of the analytical parameters of the RAS program.

3. Contract Deliverable Requirements

The organic RAS program specifies contractually required deliverables by the laboratory for sample extraction, volatile analysis and data reporting. These requirements include:

- o Completion of sample extraction for water samples within 5 days of sample receipt and for soil samples within 10 days of sample receipt;
- o Completion of volatile analysis for water samples within 7 days of sample receipt and for soil samples within 10 days of sample receipt;
- Completion of extractable analysis and reporting of data within 30 or
 40 days (as specified by the contract delivery schedule) following sample receipt.

Laboratories are subject to financial penalties for late delivery in meeting these deadlines and incentives for early delivery of the final data package. Illegible data reports are considered unacceptable, and the laboratory is required to resubmit readable versions of any illegible pages.

The organic RAS data package provides a complete set of data for independent review by the client user. Through review of data package components, the client can determine the quality of the analytical data.

Each organic RAS data package includes the following components:

- o Narrative report, describing analytical problems encountered and internal QC processes applied.
- o Copies of sample Traffic Reports.
- o Quality control summary, containing surrogate, method blank, matrix spike and matrix spike duplicate analyses recoveries, and instrument tuning and performance information.
- o Sample data, including tabulated results of the organic TCL (HSL) compounds identified and quantified, and the tentative identification and estimated concentration of up to 30 non-TCL (HSL) organic

compounds in greatest apparent concentration, reported in ug/l or mg/kg.

- o Raw sample analytical data, including sample chromatograms, spectra, quantitation reports, and calculations.
- o Standards data package, including chromatograms, spectra and data system printouts amd initial and continuing calibration reports.
- o QC data package, documenting instrument tuning and analytical QC criteria.

The organic RAS deliverables index and copies of organic data reporting sheets are contained in Appendix B.

4. Analytical Protocols

The standardized organic analytical methods are based on Federal Register (FR) Methods 625 (B/N/A), 608 (pesticide), and 624 (VOA) modified for CLP use in the analysis of both water and soil samples. Analysis for the organic HSL compounds includes an optional GC screen (to determine appropriate dilution fraction or aliquote sizes for GC/MS analysis) and GC/MS analysis.

a. Water Method

Water samples for full organic analysis (base/neutral/acid, volatile and pesticide/PCB compounds) are first prepared and/or solvent extracted, resulting in three individual sample fractions: semivolatile (B/N/A); volatile (VOA); and pesticide/PCB. Extracts are cleaned up when necessary, using optional column chromatography techniques.

The identification and quantification of the organic TCL (HSL) compounds in water samples is performed by GC/MS for B/N/A and VOA fractions, and by GC/EC for the pesticide/PCB fraction.

In addition, the 20 highest non-TCL (HSL) base/neutral/acid compound peaks and the 10 highest non-TCL (HSL) volatile peaks are tentatively

identified and their concentrations estimated, using a forward search of the NBS Mass Spectral Library.

b. Soil Method

Soil samples for full organic analysis (base/neutral/acid and pesticide/PCB compounds) are prepared by sonification prior to solvent extraction. Extracts are cleaned up using optional column chromatography techniques when necessary.

The identification and quantification of the organic TCL (HSL) compounds in soil samples is performed by GC/MS for B/N/A and VOA fractions, and by GC/EC for the pesticide/PCB fraction.

In addition, the 20 highest non-TCL (HSL) base/neutral/acid peaks and the 10 highest non-TCL (HSL) volatile peaks are tentatively identified and their concentrations estimated, using a forward search of the NBS Mass Spectral Library.

c. Contract Required Quantitation Limits (CRQLs)

Low level analysis contract required quantitation limits for water samples are based on CRQLs for each organic compound using FR Methods 624, 625, and 608 and are at the part-per-billion (ppb) level. Approximate achievable sample quantitation levels for low and medium level water and soil samples can be calculated based on the sample size and on concentration/dilution factors.

CRQLs are provided for analytical guidance, as the limits are highly matrix dependent. Matrix interferences vary considerably depending on the nature and homogeneity of the sample, on the interferent contaminants which coextract from the sample, and on the sample volume taken for analysis.

5. Contract Quality Control Requirements

The CLP quality control (QC) program for organic RAS laboratory analysis is structured to provide consistent results of known and documented quality. The program, therefore, places stringent quality control requirements on all laboratories performing sample analyses. Sample data packages contain documentation of a series of QC operations that allow an experienced chemist to determine the quality of the data and its applicability to each sampling effort. In addition, laboratory contracts contain provisions for sample re-analysis if and when specified QC criteria are not met by the contract laboratory. Each CLP laboratory is also encouraged to develop additional internal QA/QC procedures.

The minimum QC requirements of the organic RAS program consist of both an initial and ongoing demonstration of laboratory capability to generate acceptable performance with the contract methods in the analysis of water and soil samples. CLP contracts define extensive QC procedures that must be performed and documented, and criteria that must be met. These include, but are not limited to, the following.

Instrument QC procedure:

- GC/MS instrument tunes for both volatile and semi-volatile compound analyses.
- o Initial multi-level calibration for each TCL (HSL) compound.
- o Continuing calibration for each TCL (HSL) compound.

Sample QC procedure:

- o Addition of surrogate compounds to each sample and blank for determining percent recovery information.
- o Duplicate matrix spike analyses.
- o Method blank analyses.
- Certain QC procedures listed above demonstrate that the instrument is operating within contract specifications. These include:

- o A demonstration that the two tuning compounds (DFTPP for extractables and BFB for volatiles) meet the defined ion abundance criteria.
- o Determination of an average response factor (\overline{RF}) based on a calibration using several concentrations of each HSL compound that must meet a defined relative standard deviation (RSD) and minimum \overline{RF} .
- A continuing calibration at a single concentration for each HSL compound for which specified compounds are flagged as controls which must meet defined percent difference (%D) from the initial RF, or a new initial calibration must be performed.

Other QC procedures are required to demonstrate the quality of the analytical data generated. These include:

- Addition of surrogate spikes to all samples and blanks to monitor sample preparation and analysis and to provide percent recovery information for each sample, so that the suitability of the method for each sample (regardless of matrix) may be established.
- Analysis of duplicate matrix spiked samples to display the precision of the method for the particular matrix and also to provide percent recovery information for defined TCL (HSL) compounds (specified matrix spikes) as for surrogates.
- Analysis of reagent blanks for each Case or each set of 20 samples (whichever is less) and for each matrix within a Case, to assure that laboratory contaminants are not reflected in data results.

It is the responsibility of the contractor laboratory to document, in each data package submitted, that both initial and ongoing instrument and analytical QC criteria have been met. The laboratory must demonstrate that instrument tuning and calibration criteria have been met, that interferences from the analytical system are under control, and that surrogate spike, matrix spike and matrix spike duplicate recoveries falling outside contract acceptance windows are attributable to sample matrix interferences and not to laboratory analytical errors. Any samples analyzed when contract QC criteria have not been met must be reanalyzed by the laboratory if sufficient sample volume is available.

B. Inorganic Routine Analytical Services (RAS)

1. <u>Sample Matrices, Concentration Levels, Volumes Required and</u> Preservation Techniques

The inorganic RAS contract methods apply to analysis of water and soil/sediment samples. Samples for analysis should be single-phase, homogeneous samples of an appropriate matrix. Sample matrices other than water, sediment or soil are processed through the SAS program.

Inorganic RAS contract methods determine concentrations of inorganic priority pollutant constituents ranging from low or background levels of concentration to medium levels, where a compound may comprise up to 15 percent of the total sample. Low level samples are generally those collected off-site, around the perimeters of a waste site, or in areas where hazards are thought to be significantly reduced by normal environmental processes. Low level samples are estimated to contain less than 10 ppm of the inorganic priority pollutant (PP) contaminants. Medium level samples are most often those collected on-site, in areas of moderate dilution by normal environmental processes. Medium level samples are estimated to contain concentrations of inorganic PP contaminants up to 15 percent. Low and medium level designations are made for sample collection volume and shipment purposes, and for determination of appropriate analytical methods and QA/QC requirements. Samples estimated to contain concentrations of any PP contaminant higher than 15 percent of the sample must be sent through High Concentration SAS for sample preparation and analysis.

The sample volume, container types, and preservations required for inorganic analysis vary according to the matrix and estimated concentration level of the sample. For RAS inorganic analysis of a water sample estimated as low level, 1 liter volume is required for metals analysis and 1 liter volume is required for cyanide analysis. These samples should each be collected in a 1-liter polyethylene bottle. For RAS inorganic analysis of a water sample estimated as medium level, 16 ounce volume is required for metals and 16 ounce volume for cyanide. These samples should each be collected in a 16-ounce glass jar. For RAS inorganic analysis of a soil sample estimated as low or medium level, 6 ounce sample volume is required for both metals and cyanide analyses. These samples should each be collected in an 8-ounce glass jar.

For the inorganics RAS program only, it is recommended that a Case of samples be collected over no more than a three-day period and samples shipped collectively when the Case is completed.

The standard procedure applied by the analytical laboratory for homogenization is to shake the sample in its original sample container and transfer 100 mL aliquots to a 250 mL beaker. For aqueous samples with high solids content, the user has the option to specify that the sample not be mixed and the analysis be performed on the supernatant. When collecting low level water samples, different preservation techniques apply to the metals and cyanide portions, as follows. For "total" metals analyses, the sample is acidified to $pH \leq 2$ with HNO3. (Total meaning inclusion of particulate and dissolved fractions.) For dissolved metals analyses, the sample is filtered, then acidified to $pH \leq 2$ with HNO3. Note of caution: if the sample contains a significant particulate fraction, acidification without filtration could result in deceptively high metal values for the water sample. Varying amounts of particulate matter can also give large differences in metal values for duplicate acidified water samples.

For the cyanide aliquot, the following guidelines should be followed:

- Test a drop of sample with potassium iodide-starch test paper (KI-starch paper); a blue color indicates the presence of oxidizing agents and the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6g of ascorbic acid for each liter of sample volume.
- o Test a drop of sample on lead acetate paper previously moistened with acetic acid buffer solution. Darkening of the paper indicates the presence of S^{2-} . If S^{2-} is present, add powdered cadmium carbonate until a drop of the treated solution does not darken the

lead acetate test paper and the filter the solution before raising the pH for stabilization.

- o Preserve samples with 2 ml of 10 N sodium hydroxide per liter of sample (pH > 12).
- o Store the samples such that their temperature is maintained at 4^oC until the time of analysis.

No chemical preservation is required for medium level water samples or low or medium soil samples.

For soil samples the standard procedure applied by the analytical laboratory for homogenization is to thoroughly mix the contents of the sample container. For solid samples with significant amounts of water, the user has the option to specify that the supernatant be decanted and the remaining sample be mixed thoroughly and analyzed.

Each sample estimated as medium level (water or soil) must be enclosed and sealed in a metal paint can for shipment. If it is not certain whether a sample should be categorized as low or medium concentration, volume should be collected and the sample preserved as specified for low level samples, however shipping procedures must be followed as designated for medium level samples. For water samples, one field blank should be supplied for each Case. Soil blanks are currently not available. It is recommended that the user not submit soil field blanks for analysis. If the user submits a rinsate blank with a case of soil samples, it will be treated as a separate aqueous matrix sample with full QC and accordingly, a sufficient volume for analysis should be provided to the laboratory. When a suitable soil blank material becomes available through EMSL, one soil blank will be supplied for each Case. No additional volume is required for duplicate analyses of either water or soil samples.

The user may specify that the duplicate and matrix spike be performed on a particular sample. If sufficient sample volume is not provided, analysis of all required parameters and/or complete QA/QC determination may not
be possible. If this occurs, SMO will contact the RSCC to determine appropriate adjustments in analysis.

Required sample volume and container types for inorganic RAS analysis of water and soil samples are illustrated in Appendix C. Pre-cleaned sample bottles are available through the Sample Bottle Repositories, as detailed in Chapter IV.

2. Constituents Identified and Quantified

The inorganic RAS program provides identification and quantification of metals and cyanide in water and soil/sediment samples. These compounds are listed on the inorganic data reporting form included in Appendix B.

3. Contract Deliverable Requirements

The inorganic RAS program specifies contractually-required deliverables for completion of metals and cyanide analysis and submission of the final data package within 30 or 35 days (as specified by the contract delivery schedule) following sample receipt at the laboratory. Laboratories are subject to financial penalties for late delivery and incentives for early delivery of the final data package. Illegible data reports are considered unacceptable and the laboratory is required to resubmit readable versions of any illegible pages.

The inorganic RAS data package provides a complete set of data for independent review by the client user. Through review of data package components, the client can determine the quality of the analytical data.

Each inorganic RAS data package includes the following components:

- o Cover page, listing the samples included in the report and narrative comments describing problems encountered in analysis.
- o Tabulated results of inorganic compounds identified and quantified, reported in ug/l or mg/kg; including a brief description of the sample.

- o Individual analytical results are flagged by the laboratory when QC indicates potential bias due to matrix effects, homogeneity, etc.
- o QC results for: preparation blanks, calibration blanks, calibration verification standards, matrix spikes, duplicates, laboratory control samples, interference check samples, analytical spikes and serial dilution analyses.
- o Tabulation of instrument detection limits determined in pure water solutions.
- o Digestion/distillation logs, sample traffic reports, and raw data system printouts identifying calibration standards, calibration blanks, preparation blanks, samples and any atypical dilution, duplicates, spikes, interference checks and any instrument adjustments or apparent anomalies on the measurement record.

A summary of RAS inorganic contract deliverables and copies of data reporting forms are contained in Appendix B.

4. Analytical Protocols

The standardized inorganic analytical methods are based on Federal Register (FR) methods, EPA <u>Methods for Chemical Analysis of Water and Wastes</u> (MCAWW), and <u>Test Methods for Evaluating Solid Waste</u> (SW-846), for the analysis of water and soil samples. Analysis for specified metals and cyanide is performed by flame, furnace and cold vapor atomic absorption (AA), colorimetric, distillation, and inductively coupled argon plasma (ICP) methods.

a. Water Method

Water samples for metals analysis are prepared, acid digested and the digestate filtered to remove insoluble materials prior to analysis. Samples are analyzed by AA or ICP methods, and dilutions are performed where any analyte concentration exceeds the calibrated range.

For water samples, a quantitative determination for cyanide is made by midi-distillation and colorimetric analysis or by titration. Mercury is quantitated in water samples by the cold vapor technique.

b. Soil/Sediment Method

Soil samples for metals analysis are prepared and acid digested and the digestate filtered to remove insoluble materials prior to analysis. Samples are analyzed by AA or ICP methods, and dilutions are performed where any analyte concentration exceeds the calibrated range.

For soil samples, a quantitative determination for cyanide is made by midi-distillation and automated colorimetric analysis. Mercury is quantitated in soil/sediment samples by the cold vapor technique.

c. Contract Required Detection Limits (CRDLs)

Exhibit C of the Statement of Work of inorganics IFB contracts contains minimum contract-required detection levels (CRDLs) that must be met by all laboratories for each of the metals and cyanide in pure water. On a quarterly basis, the contract laboratories are required to verify that their instrument detection limits (IDL) meet the CRDL's.

The instrument detection limits reported by the laboratory on the inorganic data sheets (see Appendix B Form I) are based on analysis of analytes in pure water. Detection limits for the sample analyses may be higher, depending on the sample matrix.

Detection limits for low level water samples can be achieved in the part-per-billion (ppb) to low part-per-million (ppm) range; detection limits for medium water and soil samples can be achieved in the lowppm to mid-ppm range. Detection limits are significantly affected by matrix interferences and other sample parameters that vary considerably depending on the nature and homogeneity of the sample, interferent contaminants that coextract from the sample, and by the analytical method. Lowest detection limits are achieved on low level water samples in the ppb range, where sample matrix interferences are minimal.

Extrapolations from the "pure water" IDLs must be made to estimate the detection limits for low and medium water and soil samples, since the detection levels achievable for these samples will be highly dependent on the inorganic species and matrix of each sample. Although data is reported down to the "pure water" IDL, results below the CRDL should be used with caution. Results below the CRDL are bracketed by the laboratory to indicate a value near the instrument detection limit.

5. Contract Quality Control Requirements

The CLP quality control (QC) program for inorganic RAS laboratory analysis is structured to provide consistent results of known and documented quality. The program, therefore, places stringent quality control requirements on all laboratories performing sample analysis. Sample data packages contain documentation of a series of QC operations that allow an experienced chemist to determine the quality of the data and its applicability to each investigation. In addition, laboratory contracts contain provisions for sample re-analysis if and when specified QC criteria are not met by the contract laboratory. Each CLP laboratory is also encouraged to develop additional internal QA/QC procedures.

The minimum QC requirements of the inorganic RAS program consist of both an initial and ongoing demonstration of laboratory capability to generate acceptable performance with the contract methods in the analysis of water and soil samples. CLP contracts define extensive QA procedures that must be performed and documented, and criteria that must be met. These include, but are not limited to, the following:

o Initial calibration and calibration verification (ICV).

- o Continuing calibration verification (CCV).
- o ICP interference check sample (ICS) analysis.
- o ICP serial dilution analysis.
- o Preparation blank (PB) analysis.
- o Spiked sample analysis.
- o Duplicate sample analysis.
- o Furnace AA QC analysis.
- o Laboratory control sample (LCS) analysis.

The instrument QC operations include initial and continuing calibration checks, which are performed daily and/or every 10 samples. These checks determine that the analytical system is meeting contract-required criteria.

Analytical QC operations include:

- o ICP Interference Check Sample Analyses: Performed at least twice per eight-hour shift, to verify interelement and background correction factors.
- o Preparation Blank Analyses: Performed for each batch of samples or for each set of 20 samples, to ascertain whether sample concentrations reflect contamination.
- o Spiked Sample Analyses and Duplicate Sample Analyses: Performed for each concentration and matrix within a Case of samples or for each set of 20 samples of a similar matrix within a Case, to provide information concerning sample homogeneity, analytical precision and accuracy, the effect of the sample matrix on the analytical methodology, and to enable the Agency to evaluate the long-term precision of the method.
- Serial Dilution Analyses: Performed for each group of samples of a similar matrix type and concentration for each Case of samples, or for each 20 samples received (whichever is more frequent) to ascertain whether significant chemical or physical interferences exist due to sample matrix.

- o Furnace AA QC Analysis: Required for quantitation; incorporates duplicate injections and analytical spikes in order to evaluate the precision and accuracy of the individual analytical determinations on each sample.
- Laboratory Control Samples (LCS): Standards carried through sample preparation and analysis procedures to document the performance of the entire analytical process. The results for analysis of LCS are submitted with the data package. Laboratories on a quarterly basis verify their instrument detection limits, ICP linear ranges, ICP interelement correction factors and ICP integration times.

It is the responsibility of the contractor laboratory to document, in each data package submitted, that both initial and ongoing instrument and analytical QC requirements have been met. Any samples analyzed when contract QC requirements have not been met are re-analyzed by the laboratory.

C. Dioxin Routine Analytical Services (RAS)

1. Sample Matrix and Volume Required

The dioxin RAS contract method applies to analysis of soil/sediment and water samples. Soil/sediment and water amples for analysis should be single-phase, homogeneous and of a similar matrix. Sample matrices other than soil/sediment or water are processed through the SAS program.

The dioxin RAS contract method determines the presence of the 2,3,7,8tetrachlorodibenzo-p-dioxin isomer in soil/sediment and water samples. No concentration levels are designated in the dioxin program. All samples suspected to contain dioxin are considered hazardous and handled accordingly.

The sample volume required to perform RAS dioxin analysis is four ounces of soil/sediment or 2 liters of water. Each soil sample should be collected in either one 4-ounce glass jar or one 8-ounce glass jar filled one-half full. Each water sample should be collected in 2, 1-liter amber glass bottles. The collection of more than the required sample volume is strongly discouraged due to the hazardous nature and difficulty of disposing of dioxin-contaminated waste. Each dioxin sample must be enclosed and sealed in a metal paint can for shipment.

One or more field blanks should be included with each sample batch (24 or fewer samples). The sampler must designate one field blank for fortified matrix spike analysis and one field sample for duplicate analysis. A rinsate sample, consisting of trichloroethylene used in rinsing sampling equipment, may be included in a batch. (Rinsates are the only liquid samples analyzed in the dioxin RAS program.) The sample volumes indicated are sufficient for duplicate analysis; no additional volume should be collected.

Per program procedures, a QA sample should be included and identified in each sample batch. Prepared Performance Evaluation (PE) samples are available to Regions through EMSL/LV for this purpose. PE samples should be included as part of the sample batch. Required sample volume and container types for dioxin RAS analysis of soil/sediment and water samples are illustrated in Appendix C. Precleaned sample bottles are available through the Sample Bottle Repositories, as detailed in Chapter IV.

2. Isomer Identified and Quantified

The dioxin RAS program identifies and quantifies the 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) isomer of dioxin in soil/sediment and water samples.

3. Contract Deliverable Requirements

The dioxin RAS program specifies: completion of sample extraction, clean-up analysis, and data reporting within 21 days following sample receipt at the laboratory, including automatic re-extraction and re-analysis of samples where certain criteria are not met in the initial analysis. Laboratories are subject to financial penalties for late delivery and incentives for early delivery of the data package. Illegible data reports are considered unacceptable, and the laboratory is required to resubmit read-able versions of the illegible pages.

The dioxin RAS data package provides a complete set of data for independent review by the client user. Through review of data package components, the client can determine the quality of the analytical data.

Each dioxin RAS data package includes the following components:

- Completed data reporting sheets with appropriate selected ion current profiles (SICPs) and spectra attached, indicating instrumental (GC/MS) operating parameters during data acquisition and including all rejected sample runs.
- o Results of analyses of multi-level concentration calibration solutions, including SICPs, calculated response factors, and computer-generated quantitation reports.

- o SICPs generated during each performance check solution analysis and each concentration calibration solution analysis.
- o Chronological list of all analyses performed.

A summary of RAS dioxin data deliverables and copies of data reporting forms are contained in Appendix B.

4. Analytical Protocols

a. Soil/Sediment Method

The standardized dioxin analysis contracts utilize EPA-developed analytical methods for the analysis of 2,3,7,8-TCDD in soil/sediment and water samples. Analyses are performed on a "batch" basis. A sample batch consists of a shipment of 20 to 24 field samples, and normally includes an equipment rinse solvent (trichloroethylene) sample, one or more field blanks, and a QA or PE sample.

Prior to analysis, samples are prepared, homogenized and centrifuged when necessary. Samples are then solvent extracted with continuous agitation. Column chromatographic and other cleanup procedures are applied to eliminate sample components that may interfere with detection and quantification of the 2,3,7,8-TCDD isomer.

The concentrated extract is analyzed by GC/MS using fused silica capillary column (FSCC) techniques. The identification and quantification of 2,3,7,8-TCDD is performed using selected ion monitoring (SIM) GC/MS instrumentation and data systems with the capability to acquire, store and retrieve SIM data for six ions.

b. Contract Required Quantitation Limits (CRQLs)

The RAS contract method provides procedures for the detection and measurement of 2,3,7,8-TCDD in soil/sediment and water samples at concentrations as low as 1 ug/kg (equivalent to 1 ppb). Column chromatography and other clean-up procedures are used to eliminate coextracted sample components, such as PCBs, which may interfere with the detection of very low levels of TCDD. Matrix interferences can also occur, depending on the nature and homogeneity of the sample, and the lowest CRQLs may not always be achieved.

5. Contract Quality Control Requirements

The CLP quality control (QC) program for dioxin RAS analysis is structured to provide consistent, accurate and dependable results of known and documented quality. The program, therefore, places stringent quality control requirements on all laboratories performing sample analysis. Sample data packages contain documentation of a series of QC operations that allow an experienced chemist to determine the quality of the data and its applicability to each investigation. Each CLP laboratory is also encouraged to develop additional internal QA/QC procedures.

The minimum QC requirements of the dioxin RAS program consist of both initial and ongoing demonstration of laboratory capability to generate acceptable performance within the contract methods for the analysis of soil/sediment samples for 2,3,7,8-TCDD. CLP contracts define extensive QC procedures that must be performed and documented, and criteria that must be met. These include, but are not limited to, the following:

- o Initial and continuing calibration and instrument performance checks.
- o Reagent blank analysis.
- o Field blank analysis.
- o Fortified matrix spike analysis (2,3,7,8-TCDD spiked field blank).
- o Rinsate (equipment solvent) sample analysis.
- o Duplicate sample analysis.

o Re-analyses, including re-extraction (and/or additional cleanup of the sample extract), when QC criteria are not met in the initial analysis.

The instrument QC operations include initial and continual calibration and instrument performance checks. Continued calibration is performed at the beginning of each 12-hour shift. Performance checks are performed at least twice during each 12-hour shift to demonstrate continued acceptable GC/MS resolution, sensitivity, response factor reproducibility, mass range calibration, and to validate sample data.

Analytical QC operations include: reagent blank, field blank, spiked field blank, rinsate, and duplicate sample. Reagent blank analyses are performed by the laboratory prior to and during analysis of each batch, to demonstrate that identified compound concentrations do not reflect laboratory contamination. Field blank analyses are performed on one fortified (native matrix spike) and other unfortified samples of uncontaminated soil/sediment or water included in each batch of samples; to provide information on false-positive results, on the matrix effect of the sample on the analytical methodology, and on the accuracy of the method. Rinsate sample analysis is routinely performed for each batch of samples to assure that samples have not been contaminated by sampling equipment. Duplicate sample analysis is performed on one sample of each batch to determine precision of the method.

It is the responsibility of the contractor laboratory to document, in each data package submitted, that both initial and ongoing instrument and analytical QC criteria have been met. The laboratory must demonstrate that instrument calibration criteria have been met, that interferences from the analytical system are under control, and that spike and duplicate recoveries falling outside contract acceptance windows are attributable to sample matrix interferences and not to laboratory analytical errors. Samples analyzed when contract QC criteria have not been met are reanalyzed by the laboratory. (Consult the dioxin IFB Statement of Work, Exhibit C, for detailed re-analysis requirements.)

D. Special Analytical Services (SAS)

In addition to the standardized analyses provided under the Routine Analytical Services (RAS) program, the CLP's Special Analytical Services (SAS) program provides clients with limited customized or specialized analyses, different from or beyond the scope of the RAS IFB contract protocols, but consistent with program objectives. Services provided through SAS include: quick turnaround analyses, verification analyses, analyses requiring lower detection limits than RAS methods provide, identification and quantification of non-priority pollutant and non-TCL (HSL) constituents, general waste characterizations, analysis of non-standard matrices, and other specific analyses.

SAS functions as an extension of the RAS program, matching unique client needs with individual laboratory resources to accommodate varied analytical requests, often in a short or emergency timeframe. Individual SAS subcontracts are solicited, awarded and administered by Viar and Company, as part of the company's contract with the EPA for operation of the Sample Management Office (SMO). The SAS mechanism, by utilizing the subcontracting process, allows the CLP to procure specialized services in a timely manner, on an asneeded basis. The flexibility of the SAS program expands the CLP's capabilities from standardized RAS organic, inorganic and dioxin contract analyses, to include a wide variety of additional, non-routine analytical services.

The client requestor provides SMO with the analytical methods and QA/QC requirements needed for each SAS. SMO procures SAS by subcontracting with CLP RAS laboratories or, when RAS laboratories cannot meet the analytical requirement of the SAS, with other laboratories which have demonstrated the ability to meet program performance requirements. RAS contract laboratories are evaluated for current RAS performance before they are considered for SAS solicitations, and are not solicited for SAS work if deficient in this area. SAS organic, inorganic, dioxin and high concentration analysis requests are solicited to CLP laboratories with IFB contracts in the appropriate analytical program, and that are performing in accordance with contractual requirements. Other laboratories qualify to perform certain types of SAS work by successful completion of performance evaluation sample analyses or by justification of unique analytical capability.

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Once the laboratory universe is determined, SMO initiates solicitation via telephone, contacting a minimum of three laboratories (contingent upon availability of a particular analytical service) and describing the requirements. Laboratories are asked to bid firm, fixed price(s) for the performance of specific types of analyses on a defined number of samples. Laboratory bids are evaluated by SMO in terms of bid price and responsiveness to the specified task. The SAS award is made to the lowest bidding laboratory which responds to the program's analytical requirement. A written, individual SAS subcontract agreement is then made between the laboratory and Viar and Company, (the SMO contractor for laboratory performance of specified analytical work).

A laboratory's ability to bid for SAS work and the prices being bid may vary depending on: the size or scope of the analytical request; data turnaround requirements and analytical parameters of a particular task; weekly RAS sample loading; and laboratory operating conditions at the time of solicitation. Due to the fluctuation of these factors on a weekly and, often, daily basis, the CLP may not be able to accommodate all SAS requests received. Currently, SAS services are provided on a first-come basis; however, Agency requirements can necessitate that certain work be given priority. In this event, SMO notifies the involved RSCC Primary Authorized Requestors, who determine Regional sampling priorities.

An analysis request can be processed through SAS only if the types of samples to be analyzed or the analysis requirements are different from those of the RAS program. (Consult earlier sections of this chapter for RAS sample types and analysis requirements.) SAS requests are separated into two basic categories: "RAS Plus SAS" and "All SAS." These categories are utilized in defining client requests and pursuant SAS solicitation and contract award. Analytical services available through the SAS program are described in the following sections.

Pre-cleaned sample bottles are available through the Sample Bottle Repositories, as detailed in Chapter IV. These bottles are prepared specifically for RAS analytical work; however, bottles may be utilized in SAS projects as appropriate.

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1. SAS Services

- a. RAS Plus SAS
 - (1) Fast Turnaround

A fast turnaround request is a request for routine (RAS) analyses with extraction, analysis or data delivery required in a shorter timeframe than the RAS contracts provide. Fast turnaround requests require application of existing RAS analytical parameters, methodologies and detection limits, altering only the time required for performance of analysis and/or delivery of data. For information on RAS performance/delivery requirements for organics, inorganics and dioxin IFBs, reference Part 3 of Sections A - C of this chapter.

In responding to fast turnaround requests, SAS procurement is limited by and dependent upon program sample load, laboratory capacities and laboratory operating conditions at the time of the request. Because of constant fluctuations of these factors, it is not possible to obtain fast turnaround service on an unlimited basis. Therefore, fast turnaround contracts are solicited only in situations of demonstrated need, and are used primarily to support EPA emergency actions and to meet impending litigation deadlines.

The following illustrates common "RAS Plus SAS" fast turnaround requests, with the SAS portion underlined:

- o TCL (HSL) volatile organic compound analysis with VOA analysis and data delivery in 5 days.
- o IFB inorganic compound analysis <u>with data turnaround in</u> 10 days.
- o IFB dioxin compound analysis with data turnaround in 5 days.

(2) Organic – Special Requirements in Addition to RAS

A common client request is to access the standardized or RAS organic program and add to the contract requirements. Any addition to the standard RAS Target Compound List - TCL (equivalent to Hazardous Substances List - HSL) organic analysis requirements constitutes this type of SAS request. The following examples illustrate common "RAS Plus SAS" organic requests, with the SAS portion underlined:

- o TCL (HSL) volatile compound analysis <u>at lower detection</u> limits than required by the IFB.
- TCL (HSL) full organic analysis with additional non-TCL (HSL) pesticide/herbicide compounds.
- o TCL (HSL) pesticide compound analyses with minor alterations or additional procedures applied.
- o TCL (HSL) B/N/A compound extraction with analysis by a non-TCL (HSL) method.
- (3) Inorganic Special Requirements in Addition to RAS

As with organics, it is common for a client to request the standardized inorganic RAS program and add to the contract requirements. Any addition to the standard RAS inorganic analysis requirements constitutes this type of SAS request. The following examples illustrate common "RAS Plus SAS" inorganic requests, with the SAS portion shown underlined.

- o Metals and cyanide analyses <u>plus non-IFB</u> parameters <u>nitrate</u>, <u>sulfate</u>, <u>ammonia</u>, <u>sulfide</u>, <u>total</u> organic carbon and chloride.
- o Metals and cyanide analyses with rigorous sample homogenization.
- o Metals analysis at lower detection limits than required by the IFB.

- o Metals and cyanide analysis with minor alterations or additional procedures applied.
- (4) Dioxin Special Requirements in Addition to RAS

A client may need to access the standardized dioxin RAS program and add to the contract requirements. Any addition to the standard dioxin analysis requirements constitutes this type of RAS plus SAS request. The following examples illustrate "RAS Plus SAS" dioxin requests, with the SAS portion under-lined:

- o 2,3,7,8-TCDD analysis of soil/sediment samples with a detection limit lower than 1 ppb.
- 2,3,7,8-TCDD analysis <u>plus analysis of other dioxin or</u> furan isomers.
- b. All SAS

CLP clients frequently request types of analyses that are outside the scope of or not directly applicable to the RAS program. This occurs most often with samples of difficult or unusual matrices and requests to measure analytical parameters not provided through the RAS program. In these situations, requests are met through a SAS-contracting process referred to as "All SAS." Five categories of "All SAS" requests are described in the following sections.

- (1) Organic Special Requirements Not Provided by RAS
 - o Seven TCL (HSL) <u>PCB arochlors analysis</u> only (i.e., not the entire IFB pesticide fraction).
 - o <u>Non-TCL (HSL)</u> compound analysis.
 - Organic extraction of <u>non-aqueous and non-soil/sediment</u> samples (e.g., oil, tar or biological tissue samples by a non-IFB extraction procedure).
 - o Organic analysis by non-RAS methods.

- (2) Inorganic Special Requirements Not Provided by RAS
 - o <u>Specified IFB element analysis only</u> (e.g., cadmium, mercury and selenium).
 - <u>Non-IFB parameter</u> analysis (e.g., total organic carbon, Sulfate, TSS).
 - o EP Toxicity tests (metals, pesticides or herbicides).
 - Any inorganic preparation/analysis of <u>non-aqueous and</u> <u>non-soil/sediment</u> samples (e.g., oil, tar or biological tissue) by a non-IFB procedure.
 - o Metals analysis by non-RAS methods.
- (3) Dioxin Special Requirements Not Provided by RAS
 - o 2,3,7,8-TCDD in <u>fish tissue</u> (e.g., matrix other than soil/sediment).
 - o 2,3,7,8-TCDF (furan) in any matrix.
 - o Total <u>tetra- through octa-</u> dioxin and/or <u>furan</u> classes in varied matrices.
 - o Analysis by HRGC/<u>HRMS</u> or GC/<u>MS/MS</u>.
- (4) High Concentration Sample Analysis Organic and Inorganic

The SAS program provides for extraction and analysis of High Concentration (HC) samples. HC sample analysis will eventually be implemented under the RAS program. HC analysis services are described below.

- Organic extraction and analysis for TCL (HSL) compounds
 by GC/MS with tentative identification of 30 non-TCL (HSL) compounds of greatest concentration.
- Inorganic preparation/analysis for total metals and total mercury.

(5) Special Topics Analysis

The SAS program can also accommodate unusual analytical requests on an "All SAS" basis, when sufficient lead time is allowed and complete methodology and QA/QC specifications accompany the request. These types of analyses include, but are not limited to:

- o Biological samples (e.g., fish, turtle tissue) for specific organic, inorganic or dioxin analyses.
- o Air samples (e.g., tenax, charcoal and flurosil tubes) for specific organic analyses.
- o Wipe samples for specific organic or inorganic analyses.
- o Methods comparison/evaluation studies.
- o Asbestos analysis.
- o Acid deposition parameters.
- o Non-Superfund analytical services of any type.

2. Contract Deliverable Requirements

SAS contracts specify required delivery schedules for sample extraction, analysis and data reporting, <u>as defined by the client requestor</u>. Deliverable requirements for "RAS Plus SAS" and "All SAS" requests may use RAS contract deliverable requirements as a guide, but must be specified by the client at the time of request. The requestor should specify all deliverables required, to ensure that appropriate data packages are received.

3. Contract Quality Control Requirements

SAS contracts require laboratory performance of QC procedures and reporting of QC parameters, <u>as defined by the client requestor</u>. QC requirements as specified in RAS program contracts, may be used as a guide but must be specified by the client at the time of request. Clients are encouraged to maintain a high level of QC in all analysis requests, unless there is substantial reason for deleting certain QC requirements.

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CHAPTER III

UTILIZATION OF ANALYTICAL SERVICES

CHAPTER III UTILIZATION OF ANALYTICAL SERVICES

The CLP provides clients with prompt access to laboratory services through a documented system of sample scheduling and coordination in which the client plays a key role. The purpose of this chapter is to familiarize clients with the specific procedures and required documentation for each program and to provide complete, step-by-step information on how to properly access the CLP's analytical resources.

CLP procedures are based on two fundamental requirements: (1) the maintenance of ongoing communication among Regional Sample Control Center (RSCC), field sampler, SMO and laboratory personnel, and (2) the correct use of sample scheduling and tracking documents by RSCC, field sampler and laboratory personnel. The Sample Management Office (SMO) provides centralized direction and coordination in scheduling sample analyses through the CLP, and tracks the progress of samples from the point of collection through final data production. To effectively match the analytical needs of program clients with the capabilities of appropriate contractor laboratories, SMO maintains ongoing records which document, for each program: current utilization, availability of resources, and laboratory performance limitations.

SMO is authorized to accept analytical requests only through the RSCC, centered in the Region's Environmental Services Division or Waste Management Division. The RSCC, established by the EPA Regional Administrator, is responsible for defining the Region's analytical priorities and placing analytical requests for CLP services through SMO. The RSCC consists of three or more identified Authorized Requestors (AR), who routinely place analytical requests through SMO and coordinate with SMO throughout sample shipment and analysis. The RSCC is responsible for ensuring Regional compliance with the CLP's organic allocation system, as described in the following section. The Primary AR determines analytical priorities for the Region when conflicts occur.

Individuals interested in obtaining CLP analytical support are instructed to contact their Regional EPA office's Regional Sample Control Center (see Appendix A).

A. Analysis Initiation/Request Procedures

1. RAS Initiation Process

a. User Information Required

To initiate a RAS request, the RSCC Authorized Requestor contacts the appropriate SMO Coordinator by telephone and provides a complete description of the analytical requirement. (SMO personnel are identified in the CLP Directory, Appendix A.)

SMO requires the following information to initiate a RAS request:

- o Name of RSCC Authorized Requestor.
- o Name(s), association, and telephone number(s) of sampling personnel.
- o Name, city and state of the site to be sampled.
- o Superfund site/spill ID (2 digit alpha-numeric code).
- o Dioxin tier assignment, where applicable.
- o Number and matrix of samples to be collected.
- o Type of analyses required.

Organics: full (VOA, B/N/A and pesticides/PCB) or VOA and/or B/N/A and/or pesticides/PCB.

Inorganic: metals and/or cyanide.

Dioxin: 2,3,7,8-TCDD.

- o Scheduled sample collection and shipment dates.
- o Nature of sampling event (i.e., investigation, monitoring, enforcement, remedial, drilling project, Cercla Cooperative Agreements).
- o Suspected hazards associated with the sample and/or site.
- o Other pertinent information which may affect sample scheduling or shipment (i.e., anticipated delays due to site access, weather conditions, sampling equipment).
- o Name(s) of Regional or contractor contacts for immediate problem resolution.

The Authorized Requestor is responsible for applying professional judgment in accurately estimating the numbers and types of samples and the sample shipment dates of the analytical request.

Overestimation of the number of samples to be collected and/or miscalculation of shipment dates unnecessarily ties up available laboratory capacity, preventing the efficient management of CLP analytical resources and rendering the program less than maximally responsive to all clients. Underestimation of the numbers and types of samples to be collected may mean that adequate services will not be available for any additional analyses needed.

b. Lead-Time Requirement

When planning for a sampling activity has been completed and <u>at</u> <u>least one week prior to the scheduled start of sampling</u>, the AR telephones SMO and places the specific request for RAS services. In order to facilitate laboratory scheduling and resolution of questions concerning sampling and analysis procedures, and to allow the sampler adequate time to prepare the required sample documentation, the RSCC is required to provide scheduling information by noon on Wednesday of the week prior to sample shipment. Advance scheduling is available and should be utilized whenever possible.

c. Case Number Assignment and Laboratory Scheduling

At the time of request, SMO assigns a sequential Case number to each individual RAS sampling activity. The RSCC records the Case number and uses it in referencing that request throughout sampling and analysis. A Case number designates a single group of samples collected at one site or geographical location during a predetermined and finite time period and is used to identify a particular RAS sampling event throughout sample tracking and data production.

SMO then schedules the requested analyses through an appropriate RAS laboratory. This selection is determined by the types of

analyses, number of samples, program contract capacity, sample balance among the various laboratories, and laboratory loading and instrument conditions. Organic laboratory selection is also based on the Regional Distribution of Laboratories System developed by the NPO, designed to minimize the number of laboratories producing data for any one Region. When possible, the nearest available laboratory is assigned to minimize sample shipping costs.

Once RAS laboratory assignments are made, SMO contacts the AR to confirm the field investigation plans, identify the laboratories to be used for the Case, and answer any further questions the sampler may have regarding program procedures or documentation. At that point, the AR must indicate all known or anticipated sample scheduling changes. Any other changes occurring after this time should be communicated to SMO <u>immediately</u> upon identification to ensure the timely resolution of conflicts and the optimal allocation of program resources.

After the initial placement of the RAS request, the RSCC may choose to assign a logistical contact, such as the team leader in the sampling effort, to follow up with SMO in finalizing sampling requirements, initiating changes, and coordinating sample shipment.

d. User Knowledge of Analytical Protocol

It is the responsibility of each RSCC Authorized Requestor to acquire and maintain a working knowledge of current RAS protocols and analytical services. SMO provides each Regional DPO with Master Copy notebooks of each RAS program IFB Statement of Work (SOW), which are periodically updated to reflect program protocol changes. The SOW represents the standardized requirements which each individual RAS laboratory is contractually bound to follow. Regional DPOs (see Appendix A) maintain the Region's Master Copy SOW notebooks.

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In addition to the summary information contained in this User's Guide, the RAS Statement of Work should be consulted by program users to confirm that the RAS program is suited to an analytical request. The analytical SOWs contain specific information on sample types suited to RAS analysis, compounds identified and quantified, analytical methods, protocols, detection limits, deliverable requirements, and quality control requirements. Analytical requirements differing from these RAS parameters are processed through the SAS program, as described in Chapter II, Section E.

2. SAS Initiation Process

a. User Information Required

To initiate a SAS request, the RSCC Authorized Requestor contacts the appropriate SMO Coordinator by telephone and provides a complete description of the analytical requirement. (SMO personnel are identified in the CLP Directory, Appendix A.)

SMO requires the following information to initiate a SAS request:

- o Name of RSCC Authorized Requestor.
- o Name(s), association, and telephone number(s) of sampling personnel.
- o Name, city and state of the site to be sampled.
- o Superfund site/spill ID (2 digit alpha-numeric code).
- o Number and matrix of samples to be collected.
- o Specific analyses required and appropriate protocols and QA/QC.
- o Required detection limits.
- o Matrix spike and duplicate frequency.
- o Data turnaround and data format.
- o Justification for fast turnaround request, if appropriate.
- o Scheduled sample collection and shipment dates.
- o Nature of sampling event (i.e., investigation, monitoring, enforcement, remedial, drilling project, Cercla Cooperative Agreements).

- o Suspected hazards associated with the samples and/or site.
- o Other pertinent information which may affect sample scheduling or shipment (i.e., anticipated delays due to site access, weather condition, sampling equipment).
- o Name(s) of Regional or contractor contacts for immediate problem resolution.

In follow-up to the verbal request, the AR must submit a completed SAS Client Request form to SMO. This form serves as the written record to clarify and confirm the client's requirement for specialized analysis work. A copy of the SAS Client Request form is included in Appendix C.

The Authorized Requestor is responsible for applying professional judgment in accurately estimating the numbers and types of samples and the sample shipment dates of the SAS request. Overestimation of the number of samples to be collected and/or miscalculation of shipment dates unnecessarily ties up available laboratory capacity, preventing the efficient management of CLP analytical resources and rendering the program less than maximally responsive to all clients. Underestimation of the numbers and types of samples to be collected may mean that adequate services will not be available for any additional analyses needed. Depending on the size and extent of the miscalculation, it may require that the entire request be resolicited, and sampling plans postponed accordingly.

b. Lead-Time Requirements

When planning for a sampling activity has been completed, the AR telephones SMO and places the specific request for SAS services. Because SAS services are individually procured on a competitive basis, a <u>minimum lead-time of two weeks is required</u> to process a completely defined SAS request. More lead-time is strongly recommended whenever possible. SAS solicitation will not be started until the SAS requirements have been completely defined by the AR. Modifications to any SAS request will cause the entire process to

begin again. Fully-defined requests initiated with less than two weeks, lead-time may not be solicited and awarded in time to meet the original shipment date.

Certain types of SAS requests require a longer lead-time, as follows. A <u>minimum lead-time of two to three weeks</u> is required for SAS requests which involve distribution of protocols (reference item d., this section). A <u>minimum lead-time of four or more weeks</u> is recommended for large-scale, analytically complex and/or non-Superfund SAS requests. Award of non-Superfund SAS subcontracts may only be made after the appropriate funding process is complete.

The AR should consider the above-outlined criteria in determining the lead-time required to schedule a particular SAS effort. As a general rule, due to protocol diversity and laboratory procurement procedures, accessing SAS demands greater advance planning and more lead-time than that required for the standardized RAS programs. The AR should contact SMO several weeks in advance, if there is a question regarding the advance time needed to schedule a particular SAS request.

c. SAS Number Assignment and Laboratory Scheduling

At the time of request, SMO assigns a sequential SAS number for each individual SAS sampling activity. If SAS services are being provided in association with RAS services, SMO also designates the assigned Case number. The AR records the SAS number and Case number (if applicable) and uses both case and SAS numbers in referencing the request throughout sampling and analysis. Like the Case identification, the SAS number designates a single group of samples collected at one site or geographical location during a predetermined and finite time period, and is used to identify a particular SAS sampling event throughout sample tracking and data production.

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SAS laboratory selection is based on a telephone solicitation process for each individual request, which results in a written SAS award to the lowest qualified bidder. Once SAS laboratory assignments are made, SMO notifies the AR of the laboratories that will be performing the analyses.

As indicated, the nature of the SAS laboratory solicitation process requires the Authorized Requestor to be as exact as possible with all elements of a request at the time of request. It is understood that actual site conditions can vary considerably from expected conditions and necessitate changes in the sampling plan. However, the AR has the responsibility to notify SMO <u>immediately</u> of any changes to allow sufficient time to amend the SAS contract(s) to meet the changed needs. If an original request is changed significantly, the original SAS contract will be voided and the entire analysis effort will be resolicited, requiring additional time for resolicitation before sample shipment can take place.

d. User Provided Analytical Protocol

It is the responsibility of the RSCC Authorized Requestor to provide the applicable analytical protocol and associated QC procedures to be utilized for each SAS request. Before SMO can commence a SAS solicitation, the analytical methodology and QC requirements to be applied under the SAS must be provided or referenced <u>at the time of</u> request.

For SAS requests that are based on the use of amended RAS protocols, the AR must specify modifications or additions to these protocols at the time of request. If such changes are extensive, the AR must submit changes in written form <u>two to three weeks in advance of scheduled sample shipment</u> under the SAS. This additional lead-time is required for protocol distribution and review by solicited laboratories.

For SAS requests which require use of a non-RAS method that is not commonly available, the AR must submit the method to SMO <u>two to</u> <u>three weeks in advance of sample shipment</u>, to allow time for protocol distribution and review by solicited laboratories.

SAS requests which cite the application of well-known analytical publications do not require additional lead-time for distribution, since laboratories have immediate access to this information. Examples of such frequently-utilized method manuals are listed below. Additional analytical references are supplied in Appendix E.

- Methods for Chemical Analysis of Water and Waste, USEPA, 1983.
- <u>Test Methods for Evaluating Solid Waste</u>, Physical/Chemical Methods, SW-846, USEPA Office of Water and Waste Management, 1983.
- o <u>Standard Methods for the Examination of Water and Waste</u> Water, APHA, AWWA, WPCF, Current Edition.

The RSCC should contact SMO several weeks in advance if there is a question as to whether a particular method will require additional lead-time for distribution.

3. Procedures for Making Changes to Analytical Requests

The RSCC Authorized Requestor is responsible for immediately notifying the appropriate SMO Coordinator of all changes in sampling plans as they are identified. This includes any changes in sample matrices, numbers of samples, analyses requested, detection limits, shipping dates, postponements or cancellations. The RSCC Authorized Requestor must maintain this communication at all stages of the request – before, during and after shipment of samples to the laboratories. Likewise, the AR-designated logistical contact must notify the appropriate SMO Coordinator of any changes in sampling before and during the on-site sampling event and after shipment of samples to laboratories. Failure to notify SMO of such changes can result in: delay in sampling to accommodate scheduling changes, delay in start of analysis due to conflicts, unsuitability of a particular sample to an analytical program, and/or analysis data inappropriate for client purposes.

B. Regional Organic Allocation System

An allocation system has been established by the NPO to equitably apportion available organic laboratory capacity to the Regions during periods of heavy sampling activity when analysis capacity for all requests may not be available. Currently, capacity is available for the projected sample demand; however, when the allocation system is in effect, all organics RAS and RAS plus SAS Cases will be scheduled accordingly.

During the last month of each quarter (fiscal year), the NPO provides the RSCC with the Region's monthly allocation of organic sample analyses for the following quarter. The RSCC is responsible for planning the month's sampling activities in accordance with the NPO allocation.

Under the allocation system, each Wednesday preceding the week that samples are expected to be shipped, the RSCC requests sample analyses for all planned Regional sampling activities for that week, assigning a priority to each activity requested. (Analysis request procedures are delineated in the following sections of this chapter.)

Upon receiving the Region's sampling requests, SMO makes laboratory assignments for the week, scheduling requests received up to each Region's allocation limit. Requests for space in excess of the monthly allocation will not be processed by SMO until requests from all Regions, which fall within allocations, have been placed at a laboratory. At this time, any "excess" laboratory capacity for the week is determined. The NPO then prioritizes Regional sampling requests which exceed allocations, on a national basis. Following NPO prioritization, SMO makes laboratory assignments for sampling activities as prioritized by the NPO, utilizing available laboratory capacity.

For additional information concerning the allocation system, contact SMO's Group Leader for Analytical Services (see Appendix A).

C. Sample Documentation

Each sample processed by the CLP must be properly documented to ensure timely, correct and complete analysis for all parameters requested, and most importantly, to support use of sample data in potential enforcement actions concerning a site. The CLP documentation system provides the means to individually identify, track, and monitor each sample from the point of collection through final data reporting. As, used herein, a sample is defined as a representative specimen collected at a specific location of a waste site at a particular point in time for a specific analysis, and may reference field samples, duplicates, replicates, splits, spikes, or blanks, that are shipped from the field to a laboratory. Specific CLP sample documentation requirements are described in the following sections.

Whenever questions arise, samplers should contact SMO for direction and clarification concerning the proper completion and distribution of Case and/or SAS paperwork for a sampling effort.

1. Sample Traffic Report (TR)/Usage, Distribution, Ordering

The sample documentation system for the RAS organic and inorganic programs is based on the use of the EPA sample Traffic Report, a four-part carbonless form printed with a unique sample identification number. One Traffic Report and its preprinted identification number is assigned by the sampler to each sample collected. The two types of TRs currently in use are Organic and Inorganic. Copies of the two types of TRs are included in Appendix B, along with examples of properly completed TR forms. (High Concentration TRs are used when HC preparation and analysis is performed through the RAS plus SAS mechanism and will be used for the RAS HC Program when it is implemented.)

To provide a permanent record for each sample collected, the sampler completes the appropriate TR, recording the Case Number, site name or code and location and site/spill ID, analysis laboratory, sampling office, dates of sample collection and shipment, and sample concentration and matrix. Numbers of sample containers and volumes are entered by the sampler beside the analytical parameter(s) requested for particular sample portions.

After completing the TR, the sampler includes the bottom two copies in the sample shipment to the laboratory. Following sample shipment, the sampler returns the top copy of the completed TR to SMO. The second copy is the sampler's file copy. Upon receipt of samples, the analytical laboratory documents sample condition and signs the TR, returning the signed copy to SMO and keeping a laboratory file copy. Copies of the laboratory-signed TRs are provided to the RSCC as part of the data package.

A strip of adhesive sample labels printed with the TR sample number come attached to the TR for the sampler's use in labeling sample bottles. The sampler affixes one of these numbered labels to each container making up the sample. In order to protect the label from water and solvent attack, each label must be covered with clear waterproof tape. The sample labels, which bear the TR identification number, permanently identify each sample collected and link each sample component throughout the analytical process.

When a RAS sample is to be analyzed for RAS with SAS treatment (described in Chapter II as "RAS Plus SAS" request), TR forms are used for the "RAS Plus SAS" samples. A SAS Packing List is <u>not</u> required in addition to the TR. Both the RAS Case number and the SAS number must be entered on the TR line requesting "Case Number." Both numbers are required in order to clearly identify and track the sampling event. Caution is to be taken not to include the Case Number on "All SAS" samples taken at the same site. Additionally, the sampler must document a brief description of the SAS requirement on each TR. For example, "VOA - 1 ppb detection limit."

Traffic Report forms are provided by SMO to each Region through the RSCC. One of the RSCC ARs should contact SMO two or more weeks in advance to order additional TRs for the Region.

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2. Dioxin Shipment Record (DSR)/Usage, Distribution, Ordering

Sample documentation for the RAS dioxin program utilizes the CLP Dioxin Shipment Record, a four-part carbonless form. The DSR provides a record for one shipment batch of dioxin samples (up to 24 samples). Samples are individually numbered using the pre-printed labels provided by SMO with the supply of DSRs, and each sample number is entered on the DSR by the sampler. A copy of the DSR is included in Appendix C, along with an example of a properly completed DSR form.

To provide a permanent record of each sample collected, the sampler completes the DSR, first recording the appropriate CLP Case number and Batch/Shipment number. Header information pertinent to all samples is then entered, including: site name/code, tier designation, data turnaround (15 or 30 days), sampling office, sampling contact, sampling date, date of shipment, and analysis laboratory. Sample matrix and description information (e.g., soil/sediment field sample, solvent rinsate) is recorded for each sample by checking the appropriate box following each sample number.

After completion of the DSR, the sampler includes the bottom two copies with the sample shipment to the laboratory. Following sample shipment, the sampler returns the top copy of the DSR to SMO. The second copy is the sampler's file copy. Upon receipt of the sample shipment, the laboratory documents sample condition and signs the DSR, returning a copy to SMO and keeping a file copy. Copies of the laboratory-signed DSRs are provided to the RSCC as part of the data package.

As indicated, two strips of adhesive sample labels pre-printed with unique sample numbers are provided with the DSR for the sampler's use in labeling both the sample bottle and the outside of the paint can in which the sample is packed. In order to protect the labels from water and solvent attack, labels on both the sample container and the paint can are covered with clear, waterproof tape. The sample labels permanently identify each sample collected throughout the analytical process. Dioxin Shipment Record forms are provided by SMO to each Region through the RSCC. One of the RSCC ARs should contact SMO two or more weeks in advance to order additional DSRs.

3. SAS Packing List (PL)/Usage, Distribution, Ordering

Only for "All SAS" samples (as described in Chapter II), are samplers to utilize the SAS Packing List, a four-part carbonless form. The PL provides space to list up to 20 samples on one form. SAS samples are numbered using the SAS number followed by a hyphen and progressive numerical designation, starting with 1 (e.g., 2000E-1, 2000E-2, 2000E-3, etc.) If the sampling activity extends over several days and more than one PL is used, care must be taken not to repeat sample numbers. A copy of the SAS Packing List is included in Appendix C, along with an example of a properly completed PL form. Regions/samplers should consult SMO to verify that the PL is appropriate to use in their situation.

To provide a permanent record of each sample collected, the sampler completes the PL, recording the SAS number, site name and location, sampling date, shipment date, analysis laboratory, sampling office, sampler name and telephone number, individual SAS sample numbers, sample description and analytical parameters requested.

After completing the PL, the sampler includes the bottom two copies with the sample shipment to the analysis laboratory. Following sample shipment, the sampler sends the top copy to SMO. The second copy is the sampler's file copy. Upon receipt of samples, the analysis laboratory documents sample condition and signs the PL, returning a copy to SMO and keeping a laboratory file copy. Copies of the laboratory-signed PLs are provided to the RSCC as part of the SAS data package.

Adhesive sample labels must be provided by the sampler and marked with the appropriate SAS sample numbers using indelible ink. Labels are secured to each sample container, and covered with clear waterproof tape to protect the label from the effects of water and solvent(s). The sample label permanently identifies each sample collected and links each sample component throughout the analytical process.

SAS Packing Lists are provided by SMO to each Region through the RSCC. One of the RSCC ARs should contact SMO two or more weeks in advance to order additional SAS PLs.

4. Sample Tag

To render sample data valid for Agency enforcement uses, individual samples must be traceable continuously from the time of collection until the time of introduction as evidence during litigation. One mechanism utilized in the CLP to comply with this enforcement requirement is the use of the "sample tag." Each sample removed from a waste site and transferred to a laboratory for analysis is identified by a sample tag containing specific information regarding the sample, as defined by the EPA National Enforcement Investigations Center (NEIC). Following sample analysis, sample tags are retained by the laboratory as physical evidence of sample receipt and analysis, and may later be introduced as evidence in Agency litigation proceedings. Sample tags can be obtained through the Regional office.

The information recorded on an EPA sample tag includes:

- o CLP Case/SAS No(s). The unique number(s) assigned by SMO to identify the sampling event. (Entered under "Remarks" heading.)
- CLP Sample No. The unique sample identification number (from the TR, DSR or PL) used to document that sample. (Entered under "Remarks" heading.)
- o Project Code The number assigned by EPA to the sampling project.
- o Station No. A two-digit number assigned by the sampling team coordinator.
- o Date A six-digit number indicating the month, day and year of collection.

- Time A four-digit number indicating the military time of collection.
- o Station Location The sampling station description as specified in the project plan.
- o Samplers Signatures of samplers on the project team.
- Remarks Case/SAS and sample numbers are entered here, and any pertinent comments indicated.
- o Tag No. A unique serial number pre-printed or stamped on the tag.
- o Lab Sample No. Reserved for laboratory use.

Additionally, the sample tag contains appropriate spaces for noting that the sample has been preserved and indicating the analytical parameter(s) for which the sample will be analyzed. An example of a properly completed sample tag is included in Appendix C.

Each sample tag is completed and securely attached to the sample container. Samples are then shipped under chain-of-custody procedures as described in the following section.

5. Chain-of-Custody Record

Official custody of samples must be maintained and documented from the time of sample collection up to introduction as evidence in court, in accordance with Agency enforcement requirements. The following custody documentation procedure was developed by NEIC and is used in conjunction with CLP sample documentation (i.e., Traffic Report, Dioxin Shipment Record and SAS Packing List) for all samples processed through the CLP.

A sample is considered to be in an individual's custody if the following criteria are met: it is in your possession or it is in your view after being in your possession; or it was in your possession and then locked up or sealed to prevent tampering; or it is in a secured area. Under this definition, the team member actually performing the sampling is personally responsible for the care and custody of the samples collected until they are transferred
or dispatched properly. In follow-up, the sampling team leader reviews all field activities to confirm that proper custody procedures were followed during the field work.

The Chain-of-Custody Record is employed as physical evidence of sample custody. Chain-of-Custody Record forms can be obtained through the Regional office. The sampler completes a Chain-of-Custody Record to accompany each cooler shipped from the field to the laboratory.

Similar information to that entered on the sample tag is recorded on the Chain-of-Custody Record. Header information includes the project number, samplers' signatures and the CLP Case/SAS number (entered on the upper right of the form). Do not include the commonly known name of the site, as CLP laboratories may perform work for the responsible party of that site. For each station number, the sampler indicates: date, time, whether the sample is a composite or grab, station location, number of containers, analytical parameters, CLP sample number(s) (from TR, DSR or PL), and sample tag number(s). When relinquishing the samples for shipment, the sampler signs in the space indicated at the bottom of the form, entering the date and time the samples are relinquished. The sampler enters shipper name and airbill number under the "Remarks" section on the bottom right of the form. An example of a properly completed Chain-of-Custody Record is included in Appendix C.

The custody record is completed using waterproof ink. Any corrections are made by drawing a line through and initialing the error, then entering the correct information. Erasures are not permissable.

The top, original signature copy of the Chain-of-Custody Record is enclosed in plastic (with CLP sample documentation) and secured to the inside of the cooler lid. A copy of the custody record is retained for the sampler's files.

Shipping coolers are secured and custody seals are placed across cooler openings (see Section C., following). As long as custody forms are sealed

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inside the sample cooler and custody seals remain intact, commercial carriers are not required to sign off on the custody form.

Whenever samples are split with a source or government agency, a separate Chain-of-Custody Record should be prepared for those samples, indicating with whom the samples are being split and sample tag serial numbers from splits.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record to acknowledge receipt of the samples, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal log books and records that provide a custody record throughout sample preparation and analysis.

D. Sample Packaging and Shipment

1. Packaging Requirements

Samples processed through the CLP must be packaged for shipment in compliance with current U.S. Department of Transportation (DOT) and commercial carrier regulations. All required government and commercial carrier shipping papers must be filled out and shipment classifications made according to current DOT regulations. (Consult Appendix E for shipping references.)

Traffic Reports, Dioxin Shipment Records, SAS Packing Lists, Chain-of-Custody Records, and any other shipping/sample documentation accompanying the shipment, must be enclosed in a waterproof plastic bag and taped to the underside of the cooler lid.

Coolers must be sealed with custody seals in such a manner that the custody seal would be broken if the cooler were opened.

Shipping coolers must have clearly visible return address labels on the outside. Shipping coolers that are labeled in this manner will be returned to the sampler by the laboratory within 14 days following laboratory sample receipt.

Inside the cooler, sample containers must be enclosed in clear plastic bags through which sample tags and labels are visible. Dioxin samples as well as water and soil samples suspected to be of medium or high concentration or those suspected to contain dioxin must be enclosed in a metal can with a clipped or sealable lid (paint cans are normally used for this purpose) and surrounded by packing material such as vermiculite. The outer metal can must be labeled with the number of the sample contained inside.

Water samples for low or medium level organics analysis and low level inorganics analysis <u>must</u> be shipped cooled to 4^oC with ice. <u>No ice</u> is to be used in shipping: inorganic low level soil samples or

medium/high level water samples; or organic high level water or soil samples; or dioxin samples. Ice is not required in shipping soil samples, but may be utilized at the option of the sampler. All cyanide samples; however, must be shipped cooled to 4° C.

Low and medium level water samples for inorganic analysis require chemical preservation (reference Chapter II, Section B, for preservation techniques).

Waterproof, metal ice chests or coolers are the only acceptable type of sample shipping container. Shipping containers should be packed with noncombustible, absorbent packing material (vermiculite is recommended) surrounding the plastic-enclosed, labeled sample bottles (or labeled metal cans containing samples) to avoid sample breakage in transport. Sufficient packing material should be used so that sample containers will not make contact during shipment. Earth or ice should <u>never</u> be used to pack samples. Earth is a contaminant, and ice melts resulting in container breakage. Ice should be in sealed plastic bags to prevent melting ice from soaking packing material which, when soaked, makes handling of samples difficult in the lab.

Unless otherwise instructed through SMO in advance, the laboratory disposes of unused sample volume, sample bottles and packing materials 60 days following data submission.

A summary of correct sample packaging is illustrated in Appendix C.

2. Shipping Instructions

Samples for organics analysis must be shipped "Priority One/Overnight." If shipment requires more than a 24-hour period, sample holding times can be exceeded compromising the integrity of the sample analyses.

Samples for inorganics analysis should be held until sampling for the Case is complete and shipped "Standard Air" for two-day delivery. In the RAS inorganic program, three days is the recommended period for collection of a Case of samples.

All samples should be shipped through a reliable commercial carrier, such as Federal Express, Emery, Purolator, or equivalent. Sampling offices are responsible for sample shipping charges.

The NEIC/Denver and the ERT/Cincinnati hazardous waste site manuals (references provided in Appendix E), provide extensive information on EPA-approved sample packaging and shipment techniques. In addition, general questions concerning sample packaging and shipment may be directed to SMO.

3. Shipment Coordination

To enable SMO to track the shipment of samples from the field to the laboratory and ensure timely laboratory receipt of samples, the sampler must notify SMO of all sample shipments on the day of shipment. At that time, the sampler should provide the following information:

- o Sampler name and phone number.
- o Case Number and/or SAS Number of the project.
- o Site name/code.
- o Batch numbers (dioxin only)
- o Exact number(s), matrix(ces) and concentration(s) of samples shipped.
- o Laboratory(ies) samples were shipped to.
- o Carrier name and airbill number(s) for the shipment.
- o Method of shipment (e.g., overnight, two-day).
- o Date of shipment.
- o Suspected hazards associated with the samples or site.
- o Any irregularities or anticipated problems with the samples, including special handling instructions, or deviations from established sampling procedures.
- o Status of the sampling project (e.g., final shipment, update of future shipping schedule).

Sample shipments made after 5:00 PM EST should be called in to SMO at the start of business the next day (8:00 AM EST). SMO <u>must</u> be notified by 3:00 PM EST Friday concerning information on sample shipments going out Friday intended for Saturday delivery/pickup. CLP laboratories remain open to receive or pick-up Saturday shipments <u>only</u> upon advance notification by SMO and <u>only</u> when shipment information has been provided to SMO by the sampler.

The success of sample shipment coordination depends on the proper use and handling of the sample tracking forms and on timely and complete communication among the RSCC, samplers, SMO, and laboratories. Any postponements or cancellations, changes in the number or type of samples to be collected or shipping dates must be communicated to SMO as soon as this information is known, to facilitate this process. Appendix C contains a checklist for coordinating sample shipment.

E. **Procedures for Problem Resolution**

1. Resolving Problems Concerning Sample Shipment and Analysis

Program laboratories routinely notify SMO upon encountering problems with sample receipt or during sample analysis. (Examples of these types of problems are listed in Appendix C.) In response, SMO immediately contacts the RSCC to relay the problem and to assist in formulating a solution. SMO then contacts the laboratory involved to communicate the recommended action and to authorize processing of the sample(s) in question. The key to this type of problem resolution is <u>timeliness</u>, since delays impact sample holding times (contractual time requirements for sample extraction and analysis) and, if extreme, could invalidate the analyses.

General questions a user may have regarding sample shipment, sample analyses, laboratory contracts, or the status of data deliverables on a particular Case or SAS should be referred to the appropriate SMO personnel. Questions of a technical nature regarding contract analytical procedures should be referred to the appropriate NPO Project Officer or to the appropriate Regional Deputy Project Officer through the NPO. (Reference Appendix A, CLP Directory.)

2. Resolving Problems Concerning Analytical Data

In the CLP's Regional/Laboratory Communication System, authorized Regional personnel can contact specified laboratory personnel, <u>after</u> <u>laboratory data submission only</u>, to resolve questions regarding the final data package. This system may <u>never</u> be used to initiate additional analytical work to resolve data questions. All communications between laboratories and Regional contacts are recorded by each party on a Telephone Record Log, indicating the number of the Case and/or SAS concerned, the individuals making contact, the subject of the discussion and its resolution. In follow-up, copies of completed telephone logs are sent to SMO by both the Regional and laboratory parties and become a permanent part of the Case/SAS file. An example of the Telephone Record Log is included in Appendix C. Copies are available from SMO.

Prior to the laboratory's submission of the final data package, client queries regarding those analyses or data are handled through SMO. Depending on the nature of the question, SMO will respond or will direct the client to the appropriate NPO official for resolution. Comments regarding laboratory performance, whether positive or negative, should be directed in writing to the appropriate Regional DPO, with a copy provided to the NPO.

CHAPTER IV

AUXILIARY SUPPORT SERVICES

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In addition to the analytical programs, the CLP provides several supplementary services. These activities have developed as a natural adjunct to the program's analytical services. The purpose of this chapter is to provide the user with a description of each auxiliary program service and how the service may be accessed.

A. Sample Bottle Repository Program

1. Types and Quantities of Containers Available

Under the Sample Bottle Repository operation, ten types of sample containers are available to CLP clients for use in hazardous waste sampling activities of the Superfund Program. Containers provided through this program are precleaned and QC-tested according to prescribed procedures to ensure that no contamination exists that might affect sample data results. (Sample coolers and sample preserving agents are <u>not</u> supplied through the Repository program.)

The following chart lists the types of containers provided, the number of containers per carton, and the type(s) of samples appropriate for collection in each container type. Each container type is cleaned and QC tested by procedures directly related to the specific analyses that may be performed on samples collected in the container. Therefore, to ensure appropriate quality control, users are instructed to utilize containers <u>only</u> to collect sample types as listed on the following chart.

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2. Ordering Procedures

The Sample Bottle Repository program may be used by any organization scheduling Superfund sample analysis through the CLP, and is commonly accessed by Regional and remedial contractor clients. Two individuals from each organization are designated by SMO as Repository Authorized Requestors (RARs) and only these individuals may place container requests through the program. State personnel should access the program through their EPA Regional office.

Users should contact SMO initially to become authorized to order from a Repository and to obtain a supply of Delivery Request forms. Thereafter, the RAR requests containers directly from the Repository. Since the Repository can respond only to requests submitted by a SMO-designated RAR, users must promptly notify SMO of any change in RAR designations.

There are three types of container requests, defined by the amount of time between the date the order is placed and the requested delivery date:

0	Routine Request		Fiftee	n or i	more v	vorking	days	lead-	-time
			for de	livery	•				
0	Fast-Turnaround Request		More	than	three	days,	but	less	than
	fifteen days lead-time for delivery.								

o Emergency Request - Less than three days lead-time for delivery.

Routine requests are mailed to the appropriate Repository utilizing the Delivery Request (DR), a three-part carbonless form. The DR must be signed by an RAR. The top (original) copy of the completed DR is sent to the Repository at the address indicated on the form, the second copy is retained for the user's file, and the third copy is sent to SMO.

Fast-turnaround and emergency requests should be called in to the appropriate Repository, at the telephone number provided on the form, and the written Delivery Request distributed as outlined above, to confirm the order. When placing a telephone order, the RAR must give the Repository

SAMPLE BOTTLE REPOSITORY SERVICES

Container Type	Description	No. Per Carton	Used for RAS Sample Type'
A 80-oz amber glass bottle with teflon-lined black phenolic cap		6	Extractable Organics Low Concentration Water Samples
B	40-ml glass vial with teflon-lined silicon septum and black phenolic cap	72	Volatile Organics Low & Medium Concentration Water Samples
С	l-liter high-density polyethylene bottle with white poly cap	42	Metals, Cyanide Low Concentration Water Samples
D	120-ml wide mouth glass vial with white poly cap	72	Volatile Organics Low & Medium Concentration Soil Samples
E	16-oz wide mouth glass jar with teflon-lined black phenolic cap	48	Metals, Cyanide Medium Concentration Water Samples
F	8-oz wide mouth glass jar with teflon-lined black phenolic cap	96	Extractable Organics Low & Medium Concentration Soil Samples - and - Metals, Cyanide Low & Medium Concentration Soil Samples - and - Dioxin Soil Samples. - and - Organics & Inorganics High Concentration Liquid & Solid Samples

^{&#}x27;This column specifies the only type(s) of samples that should be collected in each container.

SAMPLE BOTTLE REPOSITORY SERVICES (continued)

Container Type	Description	No. Per Carton	Used for Sample Type'
G	4-oz wide mouth glass jar with teflon-lined black phenolic cap	120	Extractable Organics Low & Medium Concentration Soil Samples - and - Metals, Cyanide Low & Medium Concentration Soil Samples - and - Dioxin Soil Samples - and - Organic & Inorganic High Concentration Liquid & Solid Samples
н	l-liter amber glass bottle with teflon-lined black phenolic cap	24	Extractable Organics Low Concentration Water Samples
J	32-oz wide mouth glass jar with teflon-lined black phenolic cap	36	Extractable Organics Medium Concentration Water Samples
к	4-liter amber glass bottle with teflon-lined black phenolic cap	4	Extractable Organics Low Concentration Water Samples

This column specifies the only type(s) of samples that should be collected in each container.

the DR number for the request and provide the corresponding written DR in follow-up.

Users should submit requests a minimum of two weeks in advance of the required delivery date, whenever possible, to ensure timely and complete delivery of containers. Emergency and fast-turnaround requests are filled on an "as available" basis from the Repository's emergency inventory stock. It may not be possible to respond to all emergency and fast-turnaround requests, as response depends on Repository inventory and in-process requests.

In the event that a request is cancelled, the user must immediately contact the Repository to verbally cancel the request, and follow up with a cancellation memo to the Repository, sending a copy of the memo to SMO. Cancellation memos, as well as all other project-related correspondence, should cite the appropriate DR number.

3. Shipment Information

Upon receipt of the Delivery Request, Repository personnel schedule shipment and begin preparing the request. Repository personnel immediately notify the RAR if for any reason the request cannot be met in full by the requested delivery date. Often, partial shipments can be arranged over several days to meet the client's requirement. If concurrent requests are received at the Repository that cannot be filled in a timely manner and if partial shipments cannot be satisfactorily arranged, the Repository immediately notifies SMO, which coordinates with the involved Regional Sample Control Center(s) in determining the priority of container requests based on the Region's sampling needs.

Each carton in a Repository shipment is marked "Box _____ of ____," and a Repository Packing List (PL) is included in Box 1 of each shipment, so that the designee can verify that the entire shipment has been received. In addition, the Repository sends two copies of the shipping PL to the RAR at the time of shipment. The RAR confirms with the designee that the entire shipment was received in good condition, then enters the date of receipt

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and signs the packing list in the space indicated to confirm receipt. The RAR must return a copy of the signed packing list to SMO within seven days of shipment receipt.

4. Procedures for Problem Resolution

a. Resolving Problems Concerning Container Shipment

If there are problems relating to shipment (i.e., shipment does not arrive by scheduled date, shipment is incomplete or contents are damaged), the shipment designee or RAR (as appropriate to the situation) should contact the Repository immediately to resolve the problem. If the problem is not satisfactorily handled in this manner, the RAR should then contact SMO for resolution.

b. Resolving Problems Concerning Container Contamination

If a user has definitive cause to suspect that container contamination may have affected sample analysis results, the concerned RSCC should notify SMO by telephone and follow up with an explanatory memorandum directed to the appropriate NPO Project Officer (PO). The memorandum should include the following information: description of the problem. rationale for suspecting container contamination, supporting documentation (if available), and lot number(s) for all containers concerned. Container lot numbers must be provided before any corrective action can be taken. Prior to requesting corrective action, the user should verify to the extent possible that the contamination encountered is not a result of either improper field procedures (e.g., use of contaminated water for field blanks) or poor laboratory practice (e.g., background contamination) and include this information as part of the rationale in the memorandum submitted to the NPO.

After review of submitted information, the PO notifies SMO to initiate appropriate follow-up action. Upon notification by SMO, the Repository will first check the QC analysis record for the concerned

lot(s) of containers and verify that contract procedures were correctly followed and that the lot passed the QC analysis. Should an error be identified in this process, the Repository will notify SMO immediately.

As a second step, following PO authorization the Repository will pull the QC storage container for the bottle lot(s) and analyze the container(s) for suspected contaminants. SMO will notify the RSCC concerning the analysis results, so that if there is a contamination problem, analysis data from samples collected in other containers in that lot can be appropriately flagged. Should contamination be confirmed by analysis of the QC storage container, the Repository will immediately identify the problem and correct procedures as necessary to resolve it. Should a wide-spread problem be identified at any time, RARs would be notified in a timely manner so that containers could be pulled before use in the field.

5. Summary of Container Cleaning and Quality Control Procedures

Containers provided under this program are prepared in batches or lots of approximately 100 containers. (Exact lot sizes for each container type are determined, so that a container lot is not split between cases.) Containers are cleaned in lot groups, utilizing procedures specifically designed to remove any possible contaminants. Different cleaning procedures are employed according to the container material and the type(s) of samples that will be collected in the container.

Each container lot is assigned a unique identifying number. This lot number is permanently affixed to each container in the lot, recorded in the Repository logbook, and entered on the shipment Packing List when containers from that lot are shipped. For QA purposes, it is vital that each container's lot number be permanently associated with the sample collected in that particular container. Therefore, it is recommended that samplers record each container lot number and associated CLP sample numbers in their field records at the time that samples are collected. The Repository routinely performs QC analyses on one percent of the number of containers per lot. No lot is released for shipment until acceptable QC results are verified. QC analyses are performed by equivalent methods to those utilized in CLP RAS programs, and are specific to the types of samples that may be collected in the container. If a container fails to pass the QC check, the associated lot of containers is pulled and reprocessed through the system.

A QC release number is assigned to each lot of containers that passes QC analysis, and is marked on both the analysis and storage QC containers for each lot. The QC release number is cross-referenced with the lot number in Repository records, so that all QC records can be accessed based on the lot number identification.

In addition to the QC analysis check, an additional bottle is removed from each lot and stored for QC purposes. QC storage containers are kept in a contaminant-free area of the Repository which is monitored for volatile compounds. The QC storage containers are retained as a backup to recheck for cleanliness, should possible contamination of a lot of bottles come into questions at a later date.

B. Information Services

1. Regional Sample List Report

Upon request, SMO distributes a Regional Sample List Report to the Regional Deputy Project Officer (DPO). This computerized report provides a summary of the Region's use of CLP resources during a specified period of time. The following information is included in the Sample List Report:

- o Case number
- o Sample number
- o Laboratory name and contract number
- o Laboratory sample receipt date
- o Sample weight and components analyzed
- o Sample type
- o Data due date
- Days late/early calculations for contractually required deliverables
 (i.e., extraction, VOA analysis and sample data package).

This report is provided to the Region for use as a management and resource planning tool, as well as for verification of monthly sample receipts and analyses performed. An example of the Regional Sample List is contained in Appendix D.

2. Sample Status Information

In its sample management role, SMO schedules sample analysis and tracks samples from shipment through data reporting, maintaining manual and computerized tracking systems. SMO maintains ongoing communication with the DPOs, Regional Sample Control Centers, and laboratories regarding sample status, and responds to inquiries from concerned parties as appropriate. A backlog report is sent twice monthly to DPOs and laboratories listing each laboratory's samples and the number of days the samples have been in-house.

3. General Program Information

Under the direction of CLP management, SMO serves as the program's information center for both incoming calls, correspondence and dissemination of information. Upon request, SMO provides program participants and interested parties with information and material on program services and procedures, and refers callers to the proper sources for additional information as appropriate.

C. Enforcement Support

1. Generation of Enforcement Quality Data

One major objective of Superfund is to recover from responsible parties costs incurred in the investigation and cleanup of hazardous waste sites. The process by which these parties are identified and determined to be responsible often involves litigation, and frequently the Agency's case uses CLP analytical data generated from samples collected at a given site. The CLP supports these and other enforcement requirements of Superfund by ensuring that CLP-generated analytical data is controlled and available for litigation. The CLP, in cooperation with the EPA National Enforcement Investigations Center (NEIC), has established detailed procedures and documentation to ensure that CLP sample data meets Agency enforcement standards.

a. Chain-of-Custody and Document Control

Each CLP analysis contract requires the laboratory contractor to implement a comprehensive document control system and to employ strict chain-of-custody procedures in the receipt and handling of samples throughout the analytical and data reporting process. The laboratory must have written Standard Operating Procedures (SOPs) for: receipt and log-in of samples, maintenance of sample security after log-in, tracking the sample through all steps of preparation and analysis, and organization and assembly of all sample-related documentation on a Case-specific basis. Required document control and chain-of-custody records include, at a minimum: sample tags, custody records, sample tracking records, analyst logbook pages, bench sheets, chromatographic charts, computer printouts, raw data summaries, instrument logbook pages, correspondence and the document inventory.

Before a laboratory is awarded a CLP contract and continuing periodically throughout the life of the contract, each laboratory

facility is audited by NEIC to ensure compliance with these requirements. In addition to facility audits, laboratory data and evidence documentation are reviewed by NEIC on a regular basis, as described below.

b. NEIC Evidence Audits

Laboratories are contractually required to purge their files of all evidence and other documentation relating to sample analysis, and to submit a complete Case file purge package (as detailed in the previous section) to NEIC within 80 days after submission of analysis data. The Contractor Evidence Audit Team (CEAT) reviews all document control packages to verify that the documentation is complete and conforms to contractual requirements, and routinely audits a selected number of packages to determine adherence to procedure. A list of Case file purge materials is included in Appendix D.

NEIC evidence audits may involve production of sample profiles. A sample profile traces the path and handling of specific samples from the point of collection through shipping, laboratory receipt, chemical analysis and data reporting. This process is intended to identify all evidence and sequence of events necessary to reconstruct the sample history. The goal is to present to the case attorney a depiction of the sample integrity. Examples of NEIC sample profiles for organic and inorganic Cases are included in Appendix D.

Following review and/or audit, NEIC returns laboratory Case file purge packages to the originating Region, where the packages are filed with the analysis data and may be subject to additional Regional review. In addition to the routine generation of sample profiles in evidence audits, authorized Regional personnel and enforcement attorneys may request NEIC to prepare sample profiles for Cases to support enforcement activities.

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2. Additional CLP Enforcement Support

Enforcement activities frequently require direct CLP support. Court appearances and other mandated deadlines often do not allow sufficient time for completion of the normal Case file purge package submission, review and audit process. In this event, CLP assistance may be required. Also, data package evaluation and/or testimony from laboratory or CLP personnel may be needed.

The CLP has established procedures to meet these short-term requirements through SMO, which coordinates and responds to enforcement-related requests. This process is described in the following sections.

a. Request Procedures

Requests are originated by a Regional counsel, NEIC or other appropriately designated EPA personnel, and are submitted in a memorandum to the NPO Program Manager (PM). The PM reviews the memorandum, determines necessary CLP action and forwards the request along with his directions for action to SMO. If a request requires immediate response, the requestor should contact SMO directly by telephone and relay the request, following up with the written request memorandum to the PM.

b. Requestor Information Required

The following information must be provided by the requestor to initiate CLP action:

- Name and telephone number of Regional contact coordinating the enforcement activity
- o Case/SAS number(s) of specific site sampling(s)
- o Sample number(s)

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- o Date(s) of sample collection
- o Laboratory(ies) that performed the analysis
- o Type of support needed

Most requests can be met quickly, however a <u>two-week lead-time</u> is strongly recommended.

c. Documentaton/Support Provided by CLP

In responding to enforcement support requests, SMO provides the following support:

- Arranges for the timely delivery of all laboratory and evidence documentation relating to specific sample analyses (within a minimum of seven days of request, if designated).
- o Obtains information relating to sample analysis or handling not specifically required under laboratory contracts.
- o Arranges for expert testimony by laboratory or CLP personnel.
- o Augments Regional resources for analytical data review.

D. Cost Recovery Substantiation

The CLP provides documentation concerning program analytical costs to the EPA's Office of Waste Programs Enforcement (OWPE) in support of Superfund cost recovery efforts. Formal procedures have been developed to respond to Ágency requests for this information. Site-specific cost data, the information required to initiate this process, and cost documentation provided by CLP are described in the following sections.

1. Request Procedures

Requests for cost recovery (CR) documentation on a site must be made through OWPE, using the Cost Recovery Checklist. This checklist is designed to provide basic site information needed to compile cost documentation from the CLP and other sources. A copy of the OWPE Cost Recovery Checklist is included in Appendix D. Each requesting office must complete the CR Checklist, providing all information requested, and mail the completed checklist to OWPE.

In response to requests, OWPE collects and organizes cost-related documentation from the CLP and several other sources, such as the EPA Financial Management Division, the EPA Office of Emergency and Remedial Response, and REM/FIT, TAT and other Agency contractors. In case of conflicts, OWPE is responsible for prioritizing incoming requests.

A <u>minimum lead-time of four to six weeks</u> is required to complete this process and provide the requestor with a full site cost recovery report.

2. Requestor Information Required

Requestors are asked to supply the following information items on the CR Checklist to enable the CLP to prepare its cost documentation package. (Complete checklist information is required to obtain a full OWPE cost report, which contains information from other sources in addition to the CLP.)

- Identification number The appropriate CLP Case or SAS number must be entered here. If the Case or SAS number refers to more than one site, the specific sample numbers (from the Case Traffic Reports or SAS Packing List) related to the sites in question must be provided.
- o Name and location of site.
- Date the cost report is needed A minimum of four weeks from the date of request must be given. Six week lead-time is recommended whenever possible.

3. Documentation Provided by CLP

The CLP provides an information package to OWPE which is part of OWPE's full cost recovery report to the requestor. The CLP provides the following information to OWPE:

- Financial Summary for Cost Analysis This summary lists analytical and sample management costs on a Case and/or SAS basis, showing total expenses for a particular site. Information on how sample management costs are computed is included.
- Summary of Invoices, Vouchers, and Cancelled Checks This report lists all SAS laboratory invoice numbers and includes SAS cancelled check numbers. The summary is organized by SAS number and laboratory name.
- Routine Analytical Services (RAS) Cost Report This computerized report is organized by Case number and laboratory contract. It includes laboratory invoice numbers, net analysis costs, total of adjustments for late/early deliverables, and sample management costs; and lists total costs on a sample-by-sample, laboratory contract, and Case basis.
- Special Analytical Services (SAS) Cost Report This computerized report provides a brief description of the service provided, including the number of samples analyzed, data turnaround time, contract start date, laboratory receipt date, unit costs, sample management costs, and

contract status; and lists total contract costs on SAS and laboratory bases.

 Copies of all SAS-Related Cancelled Checks and Laboratory Invoices – CLP documentation, as described above, is assembled by SMO and submitted to OWPE. OWPE provides this CLP information, along with documentation gathered from other sources, to the Regional case development team in the full cost recovery package.

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E. Contract Compliance Screening

SMO performs Contract Compliance Screening (CCS) on all RAS data produced by the CLP. Modified CCS can also be performed on a case-by-case basis on mixed RAS-plus-SAS or All-SAS data.

CCS is a structured review which determines completeness of data deliverables and compliance of QA/QC parameters with contract specifications. Primary objectives of CCS are:

- o To resolve identified discrepancies in a timely manner.
- o To form the basis for the recommendation for payment made by the PO.

Structurally similar CCS procedures are applied to organics, inorganics and dioxin data. CCS results are produced on a fast-turnaround (15 day) basis and identify compliance discrepancies by code, criterion, fraction and sample. Results are distributed to the relevant laboratory and Region and to EMSL/LV. A reconciliation process deals with responses to CCS results. Data which meet all CCS criteria at initial receipt are recommended for 100% payment of the amount due. Data with CCS defects have some payment recommendation withheld, either temporarily or permanently, depending on the nature and extent of the defect identified.

CCS results are accumulated in the CCS Database in order to produce routine and on-demand summaries of laboratory performance and of compliance trends. Examples of CCS result forms are included in Appendix D.

F. Data Review Services

A full range of review services are used to assess CLP data. Objectives of the review services are:

- o To determine the usability and limitations of data given particular field or policy assessment criteria.
- To maximize the amount of usable data by identifying critical properties of data and by resolving or proposing solutions to analytical or quality control problems.
- o To provide systematic and standardized data quality assessment and status summary to determine method, laboratory, and program performance.

These review services are performed by a number of operations:

- Review for data usability is performed by Regional personnel and contractors as a service for the clients for whom sampling and analyses have been performed. Recommended review procedures have been standardized and organized into functional guidelines for evaluating CLP data. EPA Data Validation Work Groups have produced specific documents for review of organic, inorganic and dioxin analyses.
- Comprehensive QA review is performed by EMSL/LV on specific data packages. Review and assessment of some program-wide QA results is also performed by EMSL/LV to evaluate method and laboratory performance, and the quality of analytical data.
- Review of completeness and contract compliance of key criteria in CLP data is performed by SMO on all RAS data. Completeness of all SAS data is also determined. Results are used for payment recommendation purposes and also to provide summary information on program status and performance.
- Under direction of the CLP management, EMSL/LV and/or SMO may perform additional data review, to assess a problem Case or provide a second opinion on usability.

Regional client requests for Data Review Services should be directed to the Regional Deputy Project Officer (DPO). For SMO review, a copy of the request should be submitted to SMO (Attention: Data Review Team) and a copy should be provided to the Regional Sample Control Center. In follow-up, the DPO must notify SMO that the request is authorized. Alternatively, the DPO may choose to initiate all requests for the Region.

Upon authorization by the DPO, SMO schedules the review and notifies the requestor of the date the review is scheduled for completion. It should be noted that review cannot be initiated until all deliverables for the subject Case(s) have been received from the laboratory.

All requests should be placed using the SMO Data Review Request memorandum. An example of this memorandum is provided in Appendix D. Copies are available from SMO.

1. Requestor Information Required

In completing the Data Review Request form, the client must provide the following information for each Case for which review is requested:

- o SMO Case number
- o Site name
- o Analytical laboratory name(s)
- o Number of samples
- o Sample list
- o Type(s) of review requested
- o Requested date for review completion
- o User name and contact
- o Intended use of data

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A <u>minimum lead time of two weeks</u> is required for data review. However, review time is variable depending upon the number of samples involved and the nature of the review. If conflicts occur, the appropriate DPO(s) will be notified and asked to prioritize requests.

2. Documentation Provided by CLP

An evaluation report, including a sample/result matrix, and supporting statistics and documentation, is produced with each type of review.

The QA/QC Compliance Review report indicates for each sample fraction whether the data are considered: acceptable, acceptable given qualifications noted, or unacceptable. Reasons for the designation are discussed and completed data review forms for each of the areas of performance are included in the report to the client. Examples of data review forms used in the QA/QC Compliance Report are included in Appendix D.

The contents and format of reports for Problem Case, Applications and Consulting Reviews are determined by the nature of the data problem(s) being examined and/or the purpose for which the data will be used. Any statistical measures used to define data quality and the raw data supporting conclusions are appended to these reports.

CHAPTER V

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PROGRAM QUALITY ASSURANCE

CHAPTER V PROGRAM QUALITY ASSURANCE

The purpose of this chapter is to present a summary of the different aspects of quality assurance (QA) and to show their interrelationship within the overall structure of the program. This information is included to familiarize users with the program's basic QA principles and their application, and to facilitate a more complete understanding of the quality of CLP analytical data in terms of potential utilization.

A. Interface with Agency Quality Assurance

The primary role of the CLP is to support the Agency's Superfund investigation and cleanup efforts by producing analytical data of known and documented quality usable for Agency enforcement actions keyed to identification of pollutant sources and recovery of cleanup costs. Therefore, a comprehensive quality assurance program that reflects Agency QA objectives has been incorporated into all aspects of CLP operations. The CLP links three primary aspects of QA: (1) field QA, which includes field sampling operations and QA project planning; (2) laboratory QA, which is comprised of analytical method QC and external or program QA; and (3) post-laboratory QA to review laboratory and method performance and their impact on data quality.

Field operations include sampling activities performed by the EPA Regions and National Remedial Action/Field Investigation Team (REM/FIT) and Technical Assistance Team (TAT) contractors, which result in samples being processed through the CLP for analysis. The CLP NPO coordinates closely with these and other Agency sampling groups and Agency QA teams, in the development and application of Quality Assurance Program Plans and site-specific Quality Assurance Project Plans. These plans include the consistent use of Agencyspecified analytical procedures, containers, sampling techniques, sample preservation, sample tags and chain-of-custody documents, and adherence to DOT regulations in sample shipment. The CLP strongly supports the use of consistent field sampling, and sample packaging and shipment techniques, and specifies types of sample containers and required sample volumes for appropriate target analyses. Through its Sample Bottle Repository system, the CLP provides Superfund samplers with the precleaned sampling bottles for use in the field.

The CLP is directly involved in all aspects of laboratory QA. Analytical methods require extensive Agency-specified quality control (QC) procedures and documentation to ensure a complete data product that will withstand legal scrutiny. The CLP operates an extensive external QA program, which includes: pre-award and post-award laboratory performance evaluation sample analyses and laboratory facility evaluations, required submission of laboratory Standard Operating Procedures (SOPs) for analytical operations and documentation, continuous monitoring of lab performance by Headquarters POs and Regional DPOs, and a multi-level data review process to evaluate the validity of the data product, individual laboratory performance and methods performance.

The CLP, through a variety of mechanisms, continuously strives to improve the quality of program data by maintaining state-of-the-art analytical methods, refining the structure and requirements of analytical contracts, and strengthening laboratory operations. CLP QA activities are coordinated through the NPO QA Officer, to ensure that the CLP is operating in accordance with overall Agency QA mandates.

The application of field QA is addressed in Chapters II and III, where sample volume, container, preservation, packaging, shipment and documentation requirements are discussed. Analysis or method QC is addressed for each analytical program in Chapter II, which contains a description of contract analytical methods and QC requirements for each program. The following sections of this chapter describe the program's external laboratory QA activities.

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B. Laboratory Selection Process

1. Prebid Conference

The CLP prebid conferences are requested and chaired by the National Programs Office and are arranged by SMO to facilitate the invitation for bid (IFB) process for all interested laboratories. During each conference, the appropriate program officer will explain a particular aspect of the IFB. Areas of review include: contracting aspects; pre-award information; statement of work; and the role in the CLP of the Environmental Monitoring Systems Laboratory, SMO and the National Enforcement Investigations Center.

Whenever possible, a prebid conference is scheduled for each routine analytical service IFB series. Prebid conferences are announced and logistical information provided in the IFB. In addition, if time allows, the conference is advertised in advance in Chemical and Engineering News. Prebid conferences are generally held three to four weeks prior to the bid opening. The conferences are held at various U.S. cities.

2. Preaward PE Sample Analysis

The first criterion for laboratory selection is pre-award performance evaluation (PE) sample analysis. For some solicitations prior to bid opening, interested laboratories may request preaward PE samples through the contracting officer and submit a deposit that is returned upon completion of sample analyses and submission of the PE sample data package. For other solicitations, PE samples will be available only after bid opening and only to bidders determined by EPA to be in the competitive range.

PE samples are prepared by EMSL/LV and are representative of the types of field samples that the contractor would routinely be analyzing under the subject procurement. The laboratory is required to analyze PE samples according to contract procedures set forth in the IFB, and to report PE sample data according to IFB requirements, within a specified time period, usually 21 days. Bidders' PE sample data are evaluated by NPO and EMSL/LV personnel, in terms of compliance with contract requirements and accuracy of determination of compounds at the levels known to be in the PE samples. Analysis results are rated by a scoresheet developed by EMSL/LV. The PE sample score is a primary consideration in determining bidder responsiveness/responsibility for contract award.

3. Bid Price

The second criterion for laboratory selection is bid price. Following bid opening, bid abstracts are reviewed and evaluated by EPA NPO and EPA Procurement and Contracts Management Division (PCMD) officials. The lowest competitive bidders are selected to participate in pre-award bid confirmation, the process through which bidder responsiveness and responsibility for award are demonstrated and evaluated.

4. Preaward Bid Confirmations

Preaward bid confirmations may include: 1) bidder analysis of PE samples (discussed in 2, above), 2) bidder submission of Standard Operating Procedures (SOPs), and 3) site evaluation of the bidder's facility performed by EPA program management and PCMD personnel.

a. Standard Operating Procedures

Bidders are required to submit copies of all laboratory Standard Operating Procedures (SOPs) at the time of submission of PE sample data. SOPs are not required to coincide with each specific detail of the contract requirement, but must be representative of good laboratory practices and must demonstrate that the laboratory has a facility-wide quality assurance program in place and operating. Bidder SOPs are reviewed by NPO and EMSL/LV personnel and are utilized by EPA in performance of the site evaluation.

b. On-Site Laboratory Evaluation

EPA NPO, EMSL/LV, and NEIC personnel participate in on-site evaluations of laboratory facilities of bidders which scored acceptably on the PE sample analyses and are within the EPA determined competitive range. EPA personnel perform a walkthrough of the facility and perform a thorough on-site evaluation. The results of the on-site evaluation are considered in the final determination of bidder responsiveness/responsibility for contract award.
C. Laboratory Start-Up Process

Laboratories entering the program undergo a learning curve process during which they become fully familiarized and obtain expertise in application of program methodologies and quality control procedures. To reduce the learning curve period and bring laboratories "up to speed" in a timely manner, CLP management employs a series of laboratory start-up procedures which are utilized during the laboratory's initial contract operations and whenever laboratory problems are identified during contract performance.

1. Provision of Standards to Laboratory

Immediately following contract award, EMSL/LV arranges for the provision of Standard Reference materials (SRMs) to the contractor, through the Agency's contractor-operated QA Materials Bank. These SRMs are utilized by the laboratory as the official standards to which laboratory supplied standards must be traceable throughout contract performance.

2. PO Review of First Data Packages

Initial data packages are targeted for immediate review and evaluation by the NPO Project Officer (PO), EMSL/LV and the Region. This review is intensive and focuses on any problems the laboratory has, either in applying methodologies or in reporting the data. The PO then supplies feedback to the laboratory concerning the status of the data and works with the laboratory in identifying and remedying problems.

3. PO/DPO Laboratory Visits

Depending on the extent of the problems found during the review of an initial data package, the PO or DPO may visit the laboratory facility and work on-site with laboratory personnel in rectifying problems. This process also occurs on an ongoing basis during the life of the contract. On-site laboratory evaluations are performed yearly by EPA staff, EMSL/LV,

NEIC, and the PO and/or Deputy Project Officer (DPO), as well as on an as-needed basis to resolve performance problems.

4. PO/DPO/SMO/Laboratory Communication

Telephone communication is the most widely applied method for problemsolving and maintaining efficient laboratory operations, both during the laboratory start-up phase and throughout the performance of the contract. During the start-up period, communication links are established and the laboratory becomes familiarized with the communication process. In general, the laboratory notifies SMO immediately upon identification of any problem regarding the samples or any difficulties encountered in analysis. SMO routinely resolves sample-related problems in coordination with the Regional client, and refers technical problems to the contract PO or DPO, who contacts the laboratory and resolves the problem. The resolution and any specific actions taken are reported to the appropriate SMO personnel, who records this information as part of the permanent Case record. The laboratory also records the problem and resolution in the narrative portion of the sample data report, so that the Region considers this information in association with evaluating and using the data. Regional DPOs assist in the monitoring of contractor performance and play a major role in ongoing laboratory problem resolution in coordination with the PO.

D. Laboratory Performance Evaluation

1. Performance Evaluation Sample Analysis

Performance Evaluation (PE) samples are prepared by ORD EMSL/LV and sent to contractor laboratories for analysis, normally on a quarterly basis. Aqueous organic and inorganic PE samples are typically shipped as "double blind" samples (i.e., the PE samples are not discernable from routine field samples) to ensure that the laboratory processes the samples in a routine manner. Evaluation of PE sample data is performed by EMSL/LV and is used by the NPO in formally evaluating laboratory contract performance. Additionally, PE sample QC data are entered by EMSL/LV into the program's QA Data Base, and are utilized, along with other laboratory data, in trend analyses, and evaluation and revision of contract QC criteria.

2. On-Site Laboratory Evaluation

At least once a year, EPA NPO, NEIC, Regional and EMSL/LV personnel visit each contract laboratory facility and evaluate laboratory procedures. The evaluation reports which result from these on-site visits are utilized by the NPO in identifying and remedying laboratory performance problems. Repeat on-site visits are made on an as-needed basis throughout the year, to resolve laboratory problems.

3. Corrective Action

Upon identification of laboratory performance problems, the PO and DPO work closely with the laboratory to effect correction of the problems. Depending on the scope of the problems, the laboratory may be placed on temporary hold, whereby the laboratory does not receive additional samples for analysis until the problem has been corrected.

Should the contractor's non-compliance to contract performance or deliverable requirements continue, the EPA Contracting Officer is requested by the NPO to issue a Show Cause Notice to the contractor. This document requires the contractor, within a ten-day period of time, to present the government with any facts bearing on the issue, to be used in the government's determination regarding whether the contractor's failure to perform arose out of causes beyond the laboratory's control and without fault or negligence on the part of the contractor. The contractor, in response, must submit substantial evidence to demonstrate that the contract should not be terminated for default.

A recovery plan is generally included as part of the contractor's response to the Show Cause Notice. EPA Contracts and NPO officials review the contractor's response and proposed recovery plan, and determine whether the contractor has presented sufficient evidence to demonstrate timely remedy of the noncompliance. Following this review, if the contractor has presented acceptable evidence toward recovery, the government issues a Cure Notice to the contractor which delineates the government-accepted recovery plan that the contractor must follow to avoid contract termination. The government's recovery plan includes actions and time schedules for completion of each step of the recovery process, and specifies an overall time period acceptable for completion of recovery.

Should the contractor not comply with the recovery schedule, the next and final step may be contract termination by the government for default. In addition to terminating the laboratory's contract, this action impacts on evaluation of the contractor's responsiveness/responsibility for award under future CLP solicitations.

E. Sample Data Evaluation

1. Intercomparison Check Sample Studies

Intercomparison check sample studies are initiated by the EPA Regions on a periodic basis and involve simultaneous shipment of known samples to two or more CLP and/or Regional laboratories for analysis. Check samples are routinely shipped as "single blind" sample (i.e., the laboratory is aware samples are check samples but does not know sample composition). Analytical data from study participants are compiled by the Region and used in comparative data evaluation. The Region provides intercomparison sample study results to the NPO and EMSL/LV for use in programmatic applications. These studies differ from the PE sample program in that check sample data do not result in contractual evaluation of individual laboratory performance.

2. Regional Sample Split/Spike Programs

This Regionally directed program involves simultaneous sample analysis by two or more CLP and/or Regional laboratory facilities, and provides the Region with comparative data utilized in evaluating application of methods. In the sample split program, the Regions arrange to have field samples split and sent to different contractor and Regional laboratories for analysis. In the sample spike program, a known sample volume is prepared and divided into two or more equivalent portions. Each sample portion is then spiked with known levels of contaminants, and sent to different contractor and/or Regional laboratories for analysis. Results of split/spike sample analyses performed by CLP laboratories are provided to the NPO and EMSL/LV by the Region.

F. Analytical Data Review

Upon completion of analysis and data reporting, the laboratory simultaneously sends a copy of the complete data package to the CLP SMO, EMSL/LV and the Regional client. Each of these groups performs complementary aspects of data review.

1. EMSL/LV Data Review

On a routine basis, EMSL/LV performs a comprehensive QA audit on a statistically significant subset of CLP sample data packages using a Mil. Standard 105D approach. EMSL/LV also provides data audits, data evaluation and participates in special requests such as enforcement support, preparation/evaluation of data review SOPs and special projects (e.g., Dioxin Incineration Study, Love Canal Habitability Study.) Based on these reviews, EMSL/LV prepares a detailed report on the data packages, which is provided to the NPO, DPOs, Regional Data Reviewers, and to Regional clients by SMO. This review package is valuable to both program management and users in evaluating the suitability of the contract methods to the types of samples analyzed, the quality of the analytical data, and the performance of the contractor laboratories.

In addition, EMSL/LV maintains the program's QA/QC Data Base. This data base includes:

- o Spike recoveries
- o Blanks
- o Duplicates
- o Tuning
- o Calibration
- o Method of standard additions
- o ICP check
- o Analytical results for dioxin

These data are then statistically evaluated and utilized to determine and update contract QC acceptance windows for CLP-generated data and to characterize laboratory and method performance.

2. <u>Regional Data Review</u>

The Regional client reviews all data packages resulting from Regional sampling efforts. It is the responsibility of the Region, as the data user, to determine the applicability of each data package to its intended use, e.g., site investigation support, cleanup activities and/or enforcement actions. In this review, the Region applies the CLP data review Standard Operating Procedures and references the requirements of the contract Statement of Work under which the analyses were performed.

3. Contract Compliance Screening (CCS)

Every RAS CLP-generated data package is screened by SMO upon receipt on a fast-turnaround basis (See Chapter IV, Section E). CCS determines if all contractually required forms are included, that forms are completed according to contract specifications, and that QA/QC results meet contract specifications. CCS results are distributed to the laboratory, Region and EMSL/LV and are used to determine recommendation for payment. All CLP data packages are checked for completeness by SMO. If any missing information, incomplete forms or other problems with the data package are identified, the laboratory is contacted and instructed to submit the missing or incorrect portions of the data package. Other compliance discrepancies are addressed by the CCS reconciliation process.

4. SMO Data Review Services

Under direction of the NPO, SMO may perform additional data review, checking the data for compliance to contract QC procedures and parameters and for applicability to its intended uses. This review is provided on a limited basis in response to specific Regional requests. Consult Chapter IV, Section F, for a complete description of the data review services provided and appropriate request procedures.

G. Analytical Methodology Improvement/Development

1. Protocol Standardization and Improvement

Refining and improving analytical protocols to maintain state-of-the-art status and to reflect newly defined or changed requirements of the Superfund effort, is an ongoing activity for all CLP participants. This effort is accomplished through an established system of information transfer coordinated through the NPO. All program participants submit comments or recommendations to the NPO on an ongoing basis. The NPO reviews all submitted information and considers recommendations for program application on a periodic basis.

Since 1982, input on protocol improvements has come primarily through the CLP Technical Caucuses which involve NPO, EMSL/LV, EMSL/Cincinnati, EPA Regions, SMO, laboratories and other program support contractor personnel. Analytical methods and data reporting formats are reviewed and discussed in detail at the caucus sessions. EPA personnel then review caucus discussions and compile concensus recommendations for protocol changes. Following NPO approval of recommended changes, existing laboratory contracts are modified by the Contracting Officer to include recommended revisions, through contract change order actions. Whenever possible, all laboratory contracts within an analytical program are changed concurrently to maintain consistency across the program. NPO-approved protocol revisions are included in any new IFB solicitations.

2. <u>Method Development</u>

Development of new analytical methods may be initiated by a newly identified or redefined Agency analysis requirement, such as dioxin analysis. Analytical methods utilized in the CLP are based on EPA developed and approved methodologies. The NPO, EMSL/LV, EMSL/Cincinnati, EPA Regions and the contractor community have historically contributed to development of new program analytical methodologies. Regardless of the group responsible for method development, methods are reviewed by several sources and are tested prior to implementation, to the extent possible to meet program requirements.

APPENDICES TO

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CLP USER'S GUIDE

APPENDIX A

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CLP

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**AR, RAS Only

REGION VI (Continued) Data Submission

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*Debra Morey Technical DPO		
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Data Submission		v
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Lou Welzel

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> Pam Werntz Simons, Management Information Systems Manager Site/Cost Accounting, Invoicing and Reconciliation Production

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12/86

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Region VIII

Region IX

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A-26

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	Inorganics	Primary:	Wayne Wirtanen	617/861-6700
	Dioxin	Primary:	Wayne Wirtanen	617/861-6700
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	Inorganics	Primary:	John Birri	201/321-6709
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		Alternate:	Diana Pickens	301/224-2740
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	Dioxin	Primary:	Charles Hooper	404/250-3387
12/86

REGION V	Organics	Primary: Alternate:	Frank Thomas Dennis Wesolowski Jan Pels	312/886-1973 312/886-1971 312/886-1971
	Inorganics	Primary: Alternate:	Jay Thakkar Ida Levin	312/886-9087 312/886-9087
	Dioxin	Primary: Alternate:	Frank Thomas Dennis Wesolowski	312/886-1973 312/886-1971
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	Inorganics	Primary: Alternate:	Mahmond El Feky Keith Bradley	713/954-6771 214/767-9770
	<u>Dioxin</u>	Primary: Alternate:	Mel Ritter Keith Bradley	713/954-6771 214/767-9770
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	<u>Dioxin</u>	Primary: Alternate:	Debra Morey Bill Bunn	913/236-3881 913/236-3881
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	Inorganics	Primary: Alternate:	Ralph Allen Richard Cheatham (CDM)	303/757-5063 303/458-1311
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	•	- .	Steven Pope	206/442-0370
	Inorganics	Primary: Alternate:	Andrew Hafferty Bob Rieck	206/624-9537 206/442-0370
	<u>Dioxin</u>	Primary: Alternate:	Gerald Muth Joe Blazevich	206/442-0370 206/442-0370

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APPENDIX B

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RAS DELIVERABLES AND DATA REPORTING FORMS

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RAS ORGANICS

DELIVERABLES INDEX

I. Case Narrative

The Case narrative must contain: Case number, Contract number, summary of any QC, sample, shipment and analytical problems, and documentation of all internal decision tree processes used. Outline problems encountered and final solutions. Be as specific and detailed as necessary.

II. QC Summary

- A. Surrogate Percent Recovery Summary (Form II)
- B. Matrix Spike/Matrix Spike Duplicate Summary (Form III)
- C. Method Blank Summary (Form IV)

(If more than a single form is necessary, it must be arranged in chronological order.)

- D. GC/MS Tuning and Calibration Standard (Form V)
 - 1. DFTPP in chronological order; by instrument.
 - 2. BFB in chronological order; by instrument.

III. Sample Data

- A. Samples should be arranged in packets with the Traffic Report, the Organic Analysis Data Sheet (Form I), followed by the raw data for volatile, semivolatile and pesticide sample fractions. These sample packets should then be placed in increasing SMO sample number order.
 - 1. Copy of Sample Traffic Report
 - 2. HSL Results Organic Analysis Data Sheet (Form I)
 - 3. Tentatively Identified Compounds (Form I, Part B) Must be included even if no compounds are found; if so, indicate on form: "no volatile compounds found" and/or "no semi-volatile compounds found."
 - 4. Raw data in order: VOA, BNA, Pesticide
 - a. Reconstructed ion chromatogram(s) (GC/MS), chromatogram(s) (GC)
 - b. Data System Printout
 - o Quantitation report or legible facsimile (GC/MS)

- o Integration report or data system printout (GC)
- o Calibration plots (area vs. concentration) for 4,4'-DDT, 4,4'-DDD, 4,4'-DDE or toxaphene (where appropriate)
- c. Raw HSL mass spectra and the background subtracted HSL mass spectra with lab generated HSL standard spectra (dual display)
 - Data systems incapable of dual display shall provide spectra in order:
 - Raw HSL compound spectra
 - Enhanced or background subtracted spectra
 - Laboratory generated HSL standard spectra
- d. GC/MS library search spectra for Tentatively Identified Compound(s) (TIC)
- e. Quantitation/Calculation of TIC concentration(s)
- f. Manual work sheets
- g. GPC chromatograms (if appropriate)

IV. Standards Data

- A. Current list of laboratory calculated instrument detection limits for <u>all</u> HSL compounds
- B. Initial Calibration Data (Form VI) in order: VOA, BNA; by instrument if more than one instrument used.
 - 1. When more than one initial calibration is performed, the data must be put in chronological order. All initial calibration data must be included even for a specific Case.
- C. Continuing Calibration (Form VII) in order: VOA, BNA; by instrument if more than one instrument used.
 - 1. When more than one Continuing Calibration is performed, forms must be in chronological order.
- D. **Pesticide** forms in the following order:
 - 1. Form VIII Pesticide Evaluation Standards Summary (all GC columns)
 - 2. Form IX Pesticide/PCB Standards Summary (all GC columns)

- 3. Form X Pesticide/PCB Identification (only required for positive results)
- E. VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for both the initial (five point) and all continuing (12 hour) calibrations. Spectra are not required.
- F. BNA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for both the initial (five point) and all continuing (12 hour) calibrations. Spectra are not required.
- G. All pesticide Evaluation Standard(s) (A, B and C) chromatograms and data system printouts in chronological order by GC column type.
- H. All pesticide Individual Standard Mix (A or B) chromatograms and data system printouts in chronological order by GC column type.
- I. Pesticide Quantitation Standard(s) chromatograms and data system printouts.

V. Raw QC Data

- A. DFTPP (for each 12-hour period, for each GC/MS system utilized)
 - 1. Bar graph spectrum
 - 2. Mass listing
- B. BFB (for each 12-hour period, for each GC/MS system utilized)
 - 1. Bar graph spectrum
 - 2. Mass listing
- C. Blank Data
 - 1. Tabulated results (Form I)
 - 2. Tentatively Identified Compound(s) (TIC) (Form I, Part B), even if none found
 - 3. Raw Data in order: VOA, BNA, Pesticide
 - a. Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS)
 - b. Chromatogram(s) and data system printout(s) (GC)
 - c. HSL spectra with lab generated standard (dual display)
 - Data systems incapable of dual display shall provide spectra in order:

- Raw HSL compound spectra
- Enhanced or background subtracted spectra
- Laboratory generated HSL standard spectra
- d. GC/MS library search spectra for Tentatively Identified Compound(s) (TIC)
- e. Quantitation/calculation of TIC concentration(s)
- D. Matrix Spike Data
 - 1. Tabulated results (Form I) of non-spiked HSL compounds
 - a. Form I, Part B not required
 - 2. Raw Data in order: VOA, BNA, Pesticide
 - a. Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS)
 - o Spectra not required
 - **b.** Chromatogram(s) and data system printout(s) (GC)
 - o Both primary and confirmation column data is required
- E. Matrix Spike Duplicate Data
 - 1. Tabulated results (Form I) of non-spiked HSL compounds
 - a. Form I, Part B not required
 - 2. Raw Data in order: VOA, BNA, Pesticide
 - a. Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS)
 - o Spectra not required
 - b. Chromatogram(s) and data system printout(s) (GC)
 - o Both primary and confirmation column data is required

RAS ORGANIC DATA REPORTING FORMS

.

Organics Analysis Data Sheet

(Page 1)

Laboratory Name:	Case No:
Lab Sample ID No:	QC Report No:
Sample Matrix:	Contract No:
Data Release Authorized By:	Date Sample Received:

Volatile Compounds

Concentration:	Low	Medium	(Circle One)	
Date Extracted/P	repared	l:		
Date Analyzed:				
Conc/Dil Factor:		pł	H	-

Percent Moisture: (Not Decanted) _____

CAS Number		ug/l or ug/Kg (Circle One)
74-87-3	Chloromethane	
74-83-9	Bromomethane	
75-01-4	Vinyl Chloride	
75-00-3	Chloroethane	
75-09-2	Methylene Chloride	
67-64-1	Acetone	
75-15-0	Carbon Disulfide	
75-35-4	1, 1-Dichloroethene	
75-34-3	1, 1-Dichloroethane	
156-60-5	Trans-1, 2-Dichloroethene	
67-66-3	Chloroform	
107-06-2	1, 2-Dichloroethane	
78-93-3	2-Butanone	
71-55-6	1, 1, 1-Trichloroethane	
56-23-5	Carbon Tetrachloride	
108-05-4	Vinyl Acetate	
75-27-4	Bromodichloromethane	

CAS Number		ug/l or ug/Kg (Circle One)
78-87-5	1, 2-Dichloropropane	
10061-02-6	Trans-1, 3-Dichloropropene	
79-01-6	Trichloroethene	
124-48-1	Dibromochloromethane	
79-00-5	1, 1, 2-Trichloroethane	
71-43-2	Benzene	
10061-01-5	cis-1, 3-Dichloropropene	
110-75-8	2-Chloroethylvinylether	
75-25-2	Bromoform	
108-10-1	4-Methyl-2-Pentanone	
591-78-6	2-Hexanone	
127-18-4	Tetrachloroethene	
79-34-5	1, 1, 2, 2-Tetrachloroethane	
108-88-3	Toluene	
108-90-7	Chlorobenzene	
100-41-4	Ethylbenzene	
100-42-5	Styrene	
	Total Xylenes	

Data Reporting Qualifiers

С

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- Value If the result is a value greater than or equal to the detection limit, report the value.
- U Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g., 10U) based on necessary concentration /dilution action. (This is not necessarily the instrument detection limit.) The footnote should read: U-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample
- J Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero. (e.g., 10J). If limit of detection is 10 µg/l and a concentration of 3 µg/l is calculated, report as 3J.
- This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides≥10 ng/ul in the final extract should be confirmed by GC/MS.
- B This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.

•

Laboratory Name		
Case No:	·	

Sample Number

ug/lorug/Kg

Organics Analysis Data Sheet (Page 2)

Semivolatile Compounds

CAS

Concentration:	Low	Medium	(Circle One)
Date Extracted /P	repared.		
Date Analyzed: _			· ····
Conc/Dil Factor:	·		
Deserve Mainture	10		

GPC Cleanup DYes DNo Separatory Funnel Extraction

Yes

Continuous Liquid - Liquid Extraction DYes

Percent Moisture (Decanted)

CAS Number		ug/l or ug/Kg (Circle One)
108-95-2	Phenoi	
111-44-4	bis(-2-Chloroethyl)Ether	
95-57-8	2-Chlorophenol	
541-73-1	1. 3-Dichlorobenzene	
106-46-7	1, 4-Dichlorobenzene	
100-51-6	Benzyl Alcohol	
95-50-1	1. 2-Dichlorobenzene	
95-48-7	2-Methylphenol	
39638-32-9	bis(2-chloroisopropyl)Ether	
106-44-5	4-Methylpheno:	
621-64-7	N-Nitroso-Di-n-Propylamine	
67.72.1	Hexachloroethane	
98-95-3	Nitrobenzene	
78-59-1	Isophorone	
88-75-5	2-Nitrophenol	
105-67-9	2, 4-Dimethylphenol	
65-85-0	Benzoic Acid	
111-91-1	bis(-2-Chloroethoxy)Methane	
120-83-2	2, 4-Dichlorophenol	
120-82-1	1, 2, 4-Trichlorobenzene	
91-20-3	Naphthalene	
106-47-8	4-Chloroaniline	
87-68-3	Hexachlorobutadiene	
59-50-7	4-Chloro-3-Methylphenol	
91-57-6	2-Methylnaphthalene	
77-47-4	Hexachlorocyclopentadiene	
88-06-2	2, 4, 6-Trichlorophenol	
95-95-4	2, 4, 5-Trichlorophenol	
91-58-7	2-Chloronaphthalene	
88-74-4	2-Nitroaniline	
131-11-3	Dimethyl Phthalate	
208-96-8	Acenaphthylene	
99-09-2	3-Nitroaniline	

Number		(Circle One)
83-32-9	Acenaphthene	
51-28-5	2, 4-Dinitrophenol	
100-02-7	4-Nitrophenol	
132-64-9	Dibenzofuran	
121-14-2	2 4-Dinitrotoluene	
606-20-2	2. 6-Dinitrotoluene	
84-66-2	Diethylphthalate	
7005-72-3	4-Chlorophenyl-phenylether	
86-73-7	Fluorene	
100-01-6	4-Nitroaniline	
534-52-1	4, 6-Dinitro-2-Methylphenol	
86-30-6	N-Nitrosodiphenylamine (1)	
101-55-3	4-Bromophenyl-phenylether	
118-74-1	Hexachlorobenzene	
87-86-5	Pentachlorophenol	
85-01-8	Phenanthrene	
120-12-7	Anthracene	
84-74-2	Di-n-Butylphthalate	
206-44-0	Fluoranthene	
129-00-0	Pyrene	
85-68-7	Butylbenzylphthalate	
91-94-1	3, 3'-Dichlorobenzidine	
56-55-3	Benzo(a)Anthracene	
117-81-7	bis(2-Ethylhexyl)Phthalate	
218-01-9	Chrysene	
117-84-0	Di-n-Octyl Phthalate	
205-99-2	Benzo(b)Fluoranthene	
207-08-9	Benzo(k)Fluoranthene	
50-32-8	Benzo(a)Pyrene	
193-39-5	Indeno(1, 2, 3-cd)Pyrene	
53-70-3	Dibenz(a, h)Anthracene	
191-24-2	Benzo(g. h, i)Perylene	

(1)-Cannot be separated from diphenylamine

Laboratory Name:	Sample Number
Case No	
Organics Analysis Data Sheet	
(Page 3)	

Pesticide/PCBs

Concentration	Low	Medium	(Circle One)	C
Date Extracted	Prepared:		<u> </u>	5
Date Analyzed:			<u></u>	C

GPC Cleanup I Yes No Separatory Funnel Extraction I Yes Continuous Liquid - Liquid Extraction I Yes

Conc/Dil Factor: __

Percent Moisture (decanted)

CAS Number		ug/l or ug/Kg (Circle One)
319-84-6	Alpha BHC	
319-85-7	Beta-BHC	
319-86-8	Delta-BHC	
58-89-9	Gamma-BHC (Lindane)	
76-44-8	Heptachlor	
309-00-2	Aldrin	
1024-57-3	Heptachlor Epoxide	
959-98-8	Endosulfan I	
60-57-1	Dieldrin	
72 55-9	4, 4'-DDE	
72-20-8	Endrin	
33213-65-9	Endosulfan II	
72-54-8	4, 4'-DDD	
1031-07-8	Endosulfan Sulfate	
50-29-3	4, 4 - DDT	
72-43-5	Methoxychlor	
53494-70-5	Endrin Ketone	
57-74-9	Chlordane	
8001-35-2	Toxaphene	
12674-11-2	Aroclor-1016	
11104-28-2	Aroclor-1221	
11141-16-5	Aroclor-1232	
53469-21-9	Aroclor-1242	
12672-29-6	Aroclor-1248	
11097-69-1	Arocior-1254	
11096-82-5	Aroclor-1260	

V_i = Volume of extract injected (ul)

- V_s = Volume of water extracted (ml)
- W_s = Weight of sample extracted (g)

V1 -

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V_t = Volume of total extract (ul)

<u>_____</u>

V_s.

or W_s _

Sample Number

Laboratory Name: _____

Case No

Organics Analysis Data Sheet (Page 4)

Tentatively Identified Compounds

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/1 or ug/kg)
1				
2				
3				
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20				
30				

	Contract Laboratory						Contract No				
	- ' VOL/	ATILE	[s	EMI-VOLATIL	E]	PESTIC
TOLUENE-DB	BFB	I.2 DICHLORO- ETHANE-D4	NITRO- BENZENE-DS	2-FLUORO- BIPHENYL	TERPHENYL - DI4			PHENOL-D5	2-FLUORO- PHENOL	2.4.6 TRIBROMO- PHENOL	DIBUT
(88-110)	(88-115)	(78-114)	(35-114)	(43-116)	(33-141)			(10-94)	(21-100)	(10-123)	(24-15
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	TOLUENE-DB (58-110)	VOL.	TOLUENE-DB BFB I.2 DICHLORO- ETHANE-D4 (88-110) (88-115) (76-114)	TOLUENE-DB BFB I.2 DICHLORO- ETMANE-D4 MITRO- BENZENE-D5 (88-110) (88-115) (78-114) (35-114)	TOLUENE - DB BFB I.2 DICHLORO- ETHANE-D4 MITRO- BENZENE-D5 2-FLUORO- BIPHENYL (88-110) (88-115) (76-114) (35-114) (43-116)	VOLATILE TOLUENE-DB BFB I.2 DICHLORO- ETMANE-OB MITRO- BENZENE-DS 2-FLUORO- BIPMENYL TERPHENYL- DIA (88-110) (88-115) (76-114) (35-114) (43-116) (33-141)	VOLATILE VICATILE VICATILE VICATILE Second constraints Second cons	VOLATILE SEMI-VOLATIL TOLUENE-DB BFB 1.2 DICHLONO- ETMANE-DG 2FLUCBO- BRIZENE-DS TERPHENVL BIPMENVL (43-118) TERPHENVL- DI 4 (80-110) (80-112) (78-114) (33-114) (33-141) Image: Distribution of the structure of the		Production Product	SEMI-VOLATILE SEMI-VOLATILE

WATER SURROGATE PERCENT RECOVERY SUMMARY

B-11

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FORM II

Low		Medium						·· ··· ·····				
	[VOL	ATILE	I			SE	MI-VOLATILE]	-PESTICI
SMO TRAFFIC NO.	TOLUENE-D8	BFB	1.2 DICHLORO- ETHANE-D4	NITRO - BENZENE-DS	2 ~ FLUORO - BIPHENYL	TERPHENYL - 014			PHENOL-D5	2-FLUORO - PHENOL	2.4.6 TRIBROMO- PHENOL	DIBUTYL
	(81-117)	(74-121)	(70-121)	(23-120)	(30-115)	(18-137)			(24-113)	(25-121)	(19-122)	(20-150
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VALUES	ARE OUTSIE	de of con	TRACT REQ	uired QC LI	MITS	Volati	les:	out of	;	outside of C	C limits	
ADVISOF	Y LIMITS ON	LY				Semi-	Volatiles:	out of	;	outside of Q	C limits	
						Pestic	ides:	out of	;	outside of Q	C limits	
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SOIL SURROGATE PERCENT RECOVERY SUMMARY

EDACTION	COMPOUND	CONC. SPIKE	SAMPLE RESULT	CONC.	% REC	CONC. MSD	% REC	BPD	OC LIMITS*		
PRACTICI	COMPOUND	ADDED (ug/L)		MS				HPO	RPD	RECOVERY	
VOA	1,1-Dichloroethene								14	61-145	
SMO SAMPLE NO.	Trichloroethene	······································							14	71.120	
	Chlorobenzene								13	75-130	
	Toluene								13	76-125	
	Benzene								11	76-127	
	1,2,4-Trichlorobenzene								28	39.98	
8/N	Acenaphthene								31	46-118	
SMO	2,4 Dinitrotoluene							· · · · · · · · ·	38	24.96	
SAMPLE NO.	Pyrene								31	26-127	
	N-Nitroso-Di-n-Propylamine								38	41-116	
	1,4-Dichtorobenzene								28	36-97	
4.010	Pentachlorophenol								50	9-103	
ACID	Phenol	·							42	12.89	
SMU	2-Chlorophenol								40	27-123	
SAMPLE NU.	4-Chloro-3-Methylphenol	·							42	23.97	
	4-Nitrophenol								50	10.80	
	Lindane					·			15	56-123	
PEST	Heptachlor								20	40-131	
SMO	Aldrin								22	40-120	
AMPLE NO.	Dieldrin								18	52-126	
j	Endrin								21	56-121	
	44'-DDT								27	38.127	

WATER MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Case No. _____ Contractor _____ Contract No. _____

ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

 RPD:
 VOAs ______ out of _____;
 outside QC limits

 B/N ______ out of _____;
 outside QC limits

 ACID ______ out of _____;
 outside QC limits

 PEST ______ out of _____;
 outside QC limits

RECOVERY: VOAs_____out of_____; outside QC limits B/N _____out of _____; outside QC limits ACID _____out of _____; outside QC limits PEST _____out of ____; outside QC limits

Comments: _____

FORM III

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7/85

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Case No._____ Contractor _____ Contract No._____

Low Level______ Medium Level _____

FRACTION	COMPOUND	CONC. SPIKE ADDED (ug/Kg)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	OC RPD	
VOA	1,1-Dicholorethene								22	59-172
SMO	Trichloroethene								24	62-137
SAMPLE NO	Chlorobenzene								21	60-133
SAMPLE NO.	Toluene								21	59-1 39
	Benzene			•					21	66-142
	1,2,4-Trichlorobenzene								23	38-107
B/N	Acenaphthene								19	31-137
SMO	2.4 Dinitrotoluene								47	28-89
SAMPLE NO.	Pyrene								36	35-142
	N-Nitrosodi-n-Propylamine								38	41-126
;	1,4-Dichlorobenzene					_			27	28-104
ACID	Pentachlorophenol								47	17-109
ACID	Phenol					· ·			35	26-90
	2-Chlorophenol								50	25-102
SAMPLE NU.	4-Chloro-3-Methylphenol								33	26-103
	4-Nitrophenol								50	11-114
	Lindane	·							50	46-127
PEST	Heptachlor								31	35-130
SMO	Aldrin	•							43	34.132
SAMPLE NO.	Dieldrin								38	31-134
	Endrin								45	42-139
	4,4'-DDT								50	23-134

*ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

RPD:	VOAs out of;	outside QC limits	RECOVERY:	VOAsout of;	outside QC limits
	B/N out of;	outside QC limits		B/N out of ;	outside QC limits
	ACID out of;	outside QC limits		ACIDout of;	outside QC limits
	PEST out of;	outside QC limits		PEST out of ;	outside QC limits
Comm	ente:				

FORM III

7/85

METHOD BLANK SUMMARY

150 NO	Reç	jion		Contractor			or Contract No			
FILE ID	DATE OF ANALYSIS	FRACTION	MATRIX	CONC. LEVEL	INST. ID	CAS NUMBER	COMPOUND (HSL,TIC OR UNKNOWN)	CONC.	UNITS	CRO
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						·				
mments:										

B-15

FORM IV

GC/MS TUNING AND MASS CALIBRATION

Bromofluorobenzene (BFB)

Case No.	Contractor	Contract No.
Instrument ID	Date	Time
Lab ID	Data Release Authorized By:	

m/e ION ABUNDANCE CRITERIA

%RELATIVE ABUNDANCE

50	15.0 - 40.0% of the base peak		
75	30.0 - 60.0% of the base peak		•
9 5	Base peak, 100% relative abundance		
96	5.0 - 9.0% of the base peak		
173	Less than 1.0% of the base peak		
174	Greater than 50.0% of the base peak		
175	5.0 - 9.0% of mass 174	()1
176	Greater than 95.0%, but less than 101.0% of mass 174	() 1
17 7	5.0 - 9.0% of mass 176	 () 2

THIS PERFORMANCE TUNE APPLIES TO THE FOLLOWING SAMPLES, BLANKS AND STANDARDS.

¹Value in parenthesis is % mass 174. ²Value in parenthesis is % mass 176.

SAMPLE ID	LAB ID	DATE OF ANALYSIS	TIME OF ANALYSIS
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	Deca	afluorotriphenyl <mark>j</mark>	phosphine (DFTPP)		
Case N	lo	Contractor	Contract	No	
Instru	ment ID	Date	Time		<u></u>
Lab IC)	Data Release Authorize	d By:		
m/e	ION ABUNDANCE CRI	TERIA	%RELATIVE ABUNDANC	E	
51	30.0 - 60.0% of mass 19	98	:		
68	less than 2.0% of mass	69		()1
69 .	mass 69 relative abunda	ince			
70	less than 2.0% of mass	69		(י(
127	40.0 - 60.0% of mass 1	98			
197	less than 1.0% of mass	198			
198	base peak, 100% relativ	e abundance			
199	5.0 · 9.0% of mass 198]
275	10.0 - 30.0% of mass 1	98			
· 365	greater than 1.00% of r	nass 198			
441	present, but less than n	nass 443			
442	greater than 40.0% of r	nass 198			
443	17.0 - 23.0% of mass 4	42		() ² .

GC/MS TUNING AND MASS CALIBRATION

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THIS PERFORMANCE TUNE APPLIE'S TO THE FOLLOWING1 Value in parenthesis is % mass 69.
2 Value in parenthesis is % mass 442.SAMPLES, BLANKS AND STANDARDS.2 Value in parenthesis is % mass 442.

SAMPLE ID	LAB ID	DATE OF ANALYSIS	TIME OF ANALYSIS
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Initial Calibration Data Volatile HSL Compounds

Case No:	Region	Instrument I D:
Contractor:		Calibration Date:
Contract No:		

Minimum RF for SPCC is 0.300 (0.25 for Bromoform) Maximum % RSD for CCC is 30%

Laboratory ID			,					
Compound	RF ₂₀	RF ₅₀	RF ₁₀₀	RF150	RF200	R F	% RSD	CCC• SPCC••
Chloromethane								* *
Bromomethane			1		1		j	
Vinyl Chloride				1	t		}	*
Chloroethane			l					
Methylene Chloride			·					
Acetone			1					
Carbon Disulfide			;					
1, 1-Dichloroethene			· · · · · · · · · · · · · · · · · · ·					*
1, 1-Dichloroethane			· · · · · · · · · · · · · · · · · · ·					* *
Trans-1, 2-Dichloroethene			· · · · · · · · · · · · · · · · · · ·					
Chioroform		11						*
1, 2-Dichloroethane			·		1		· · · · ·	
2-Butanone								
1, 1, 1-Trichloroethane			l		 			
Carbon Tetrachloride		11	·					
Vinyl Acetate								
Bromodichiloromethane			,					
1, 2-Dichloropropane		1 1			· · · ·			*
Trans-1, 3-Dichloropropene		1	·					
Trichloroethene		1 1						
Dibromochloromethane			1					
1, 1, 2-Trichloroethane								•
Benzene								
cis-1, 3-Dichloropropene								
2-Chloroethylvinylether								
Bromoform								• • •
4-Methyl-2-Pentanone								
2-Hexanone				·				
Tetrachloroethene								
1, 1, 2, 2-Tetrachloroethane								* *
Toluene		1						*
Chiorobenzene								* *
Ethylbenzene								*
Styrene		· · · · · · · · · · · · · · · · · · ·	1					
Total Xylenes								

RF -Response Factor (subscript is the amount of ug/L) RF -Average Response Factor %RSD -Percent Relative Standard Deviation CCC -Calibration Check Compounds (+)

SPCC -System Performance Check Compounds (++)

Initial Calibration Data Semivolatile HSL Compounds

(Page 1)

Case No: Region:	Instrument ID:
Contractor:	Calibration Date:
Contract No:	

Minimum RF for SPCC is 0.050

Maximum % RSD for CCC is 30%

7

Laboratory ID								
Compound	RF ₂₀	RF ₅₀	RF ₈₀	RF ₁₂₀	RF ₁₆₀	RF	% RSD	CCC+ SPCC++
Phenol								*
bis(-2-Chloroethyl)Ether								
2-Chlorophenol		1						
1, 3-Dichlorobenzene		1						
1, 4-Dichlorobenzene								*
Benzyl Alcohol			1					
1, 2-Dichlorobenzene								
2-Methylphenol								
bis(2-chloroisopropyl)Ether								
4-Methylphenol			[
N-Nitroso-Di-n-Propylamine			1					* *
Hexachloroethane								
Nitrobenzene	1							
Isophorone								
2-Nitrophenol			1					*
2, 4-Dimethylphenol		1						
Benzoic Acid	+							
bis(-2-Chloroethoxy)Methane		1			·			
2, 4-Dichlorophenol			-					
1, 2, 4-Trichlorobenzene			1					
Naphthalene								
4-Chloroaniline								
Hexachlorobutadiene			_	1				*
4-Chloro-3-Methylphenol			1	[*
2-Methylnaphthalene								
Hexachlorocyclopentadiene				[* *
2, 4, 6-Trichlorophenol				1	1			*
2, 4, 5-Trichlorophenol	Ŧ	1	1.	1				
2-Chloronaphthalene		1	1					
2-Nitroaniline	ŧ		1		1			
Dimethyl Phthalate			1					
Acenaphthylene		1		1				
3-Nitroaniline	†	1						
Acenaphthene		1		1	1			*
2, 4-Dinitrophenol	+		1	i			I	* *
4-Nitrophenol	+	1	1	1	1			* *
Dibenzofura n		1	1	1	1	[1	

Response Factor (subscript is the amount of nanograms) RF - Average Response Factor %RSD - Percent Relative Standard Deviation CCC - Calibration Check Compounds (+) SPCC -System Performance Check Compounds (++) † -Not detectable at 20 ng

Initial Calibration Data Semivolatile HSL Compounds

(Page 2)

Case No: _____ Region: _____ Instrument ID: _____

Contractor:

Contract No:

Minimum RF for SPCC is 0.050

Maximum % RSD for CCC is 30%

Calibration Date: _____

Laboratory ID]		
Compound	RF ₂₀	RF ₅₀	RF ₈₀	RF ₁₂₀	RF160	नन	% RSD	CCC+ SPCC++
2, 4-Dinitrotoluene								
2, 6-Dinitrotoluene								
Diethylphthalate			1					
4-Chlorophenyl-phenylether			I	1				
Fluorene								
4-Nitroaniline	Ť		1					
4, 6-Dinitro-2-Methylphenol	T T		[1				
N-Nitrosodiphenylamine (1)					1			*
4-Bromophenyl-phenylether			[
Hexachlorobenzene								
Pentachlorophenol	Ŧ							*
Phenanthrene								
Anthracene								
Di-N-Butylphthalate	,				· · ·	· ·		
Fluoranthene								*
Pyrene			[
Butylbenzylphthalate	К							
3, 3'-Dichlorobenzidine	:							
Benzo(a)Anthracene								
bis(2-Ethylhexyl)Phthalate								
Chrysene								
Di-n-Octyl Phthalate								
Benzo(b)Fluoranthene								
Benzo(k)Fluoranthene								
Benzo(a)Pyrene				·				*
Indeno(1, 2, 3-cd)Pyrene								
Dibenz(a, h)Anthracene								
Benzo(g, h, i)Perylene								

Response Factor (subscript is the amount of nanograms) AF -Average Response Factor %RSD -Percent Relative Standard Deviation CCC -Calibration Check Compounds (+) SPCC -System Performance Check Compounds (++) † - Not detectable at 20 ng

(1) -Cannot be separated from diphenylamine

Continuing Calibration Check Semivolatile HSL Compounds (Page 1)

Case No: Region:	Calibration Date:
Contractor:	Time:
Contract No:	Laboratory ID:
Instrument ID:	Initial Calibration Date:

Minimum RF for SPCC is 0.050

Maximum %D for CCC is 25%

Compound	ŘF	RF ₅₀	% D	000	SPCC
Phenol		•		*	
bis(-2-Chloroethyl)Ether					
2-Chlorophenol					
1, 3-Dichlorobenzene					
1. 4-Dichlorobenzene				*	
Benzyl Alcohol					
1, 2-Dichlorobenzene					
2-Methylphenol					
bis(2-chloroisopropyl)Ether					
4-Methylphenol					
N-Nitroso-Di-n-Propylamine					* *
Hexachloroethane					
Nitrobenzene					
Isophorone	·				
2-Nitrophenol				+	
2, 4-Dimethylphenol					
Benzoic Acid	†				
bis(-2-Chloroethoxy)Methane					
2, 4-Dichlorophenol				*	
1, 2, 4-Trichlorobenzene					
Naphthalene					
4-Chloroaniline					
Hexachlorobutadiene			· .	*	
4-Chloro-3-Methylphenol					
2-Methylnaphthalene					
Hexachlorocyclopentadiene					* *
2, 4, 6-Trichlorophenol				*	
2, 4, 5-Trichlorophenol	1				
2-Chloronaphthalene					
2-Nitroaniline	†				
Dimethyl Phthalate					
Acenaphthylene					
3-Nitroaniline	1				
Acenaphthene				*	
2, 4-Dinitrophenol	+ 1		1		* *
4-Nitrophenol	1		T		* *
Dibenzofuran	-		1		

RF₅₀ Response Factor from daily standard file at concentration indicated (50 total nanograms)

^o_oD -Percent Difference

CCC -Calibration Check Compounds (+)

RF - Average Response Factor from initial calibration Form VI

+-Due to low response, analyze

at 80 total nanograms

SPCC - System Performance Check Compounds (++)

Continuing Calibration Check Semivolatile HSL Compounds

(Page	2)
-------	----

Case No: Region:	Calibration Date:
Contractor:	Time:
Contract No:	Laboratory ID:
Instrument ID:	Initial Calibration Date:

Minimum RF for SPCC is 0.050

Maximum %D for CCC is 25%

Compound	A R	RF50	% D	CCC	SPCC
2, 4-Dinitrotoluene					
2, 6-Dinitrotoluene			1	1	
Diethylphthalate			1	1	
4-Chlorophenyl-phenylether					
Fluorene					
4-Nitroaniline	+			1	
4, 6-Dinitro-2-Methylphenol	†				
N-Nitrosodiphenylamine (1)				+	
4-Bromophenyl-phenylether					
Hexachiorobenzene				1	
Pentachlorophenol	1			+	
Phenanthrene					
Anthracene					
Di-N-Butylphthalate		·			
Fluoranthene	1			*	·
Pyrene					
Butylbenzylphthalate					
3, 3'-Dichlorobenzidine					
Benzo(a)Anthracene					
bis(2-Ethylhexyl)Phthalate					
Chrysene					
Di-n-Octyl Phthalate			·	+	
Benzo(b)Fluoranthene					
Benzo(k)Fluoranthene					
Benzo(a)Pyrene				*	
Indeno(1, 2, 3-cd)Pyrene					
Dibenz(a, h)Anthracene					
Benzo(g, h, i)Perylene					

RF₅₀ -Response Factor from daily standard file at concentration indicated (50 total nanograms)

RF -Average Response Factor from initial calibration Form VI %D -Percent Difference

+-Due to low response, analyze

at 80 total nanograms

CCC - Calibration Check Compounds (+)

SPCC - System Performance Check Compounds (++) (1) - Cannot be separated from diphenylamine

Continuing Calibration Check Volatile HSL Compounds

Case No: Region:	Calibration Date:
Contractor:	Time:
Contract No:	Laboratory ID:
instrument ID:	Initial Calibration Date:

Minimum RF for SPCC is 0.300 (0.25 for Bromoform)

Maximum %D for CCC is 25%

Compound	RF	RF ₅₀	% D	CCC	SPCC
Chloromethane					* *
Bromomethane					
Vinyl Chloride		1		*	
Chloroethane					
Methylene Chloride					
Acetone					
Carbon Disulfide					
1, 1-Dichloroethene					
1, 1-Dichloroethane					* *
Trans-1, 2-Dichloroethene					
Chloroform	· · · · ·			*	
1, 2-Dichloroethane					
2-Butanone					
1, 1, 1-Trichloroethane					
Carbon Tetrachloride					
Vinyl Acetate					
Bromodichloromethane					
1, 2-Dichloropropane	•			*	
Trans-1, 3-Dichloropropene					
Trichloroethene					
Dibromochloromethane					
1, 1, 2-Trichloroethane					
Benzene					
cis-1, 3-Dichloropropene					
2-Chloroethylvinylether					
Bromoform			•		* *
4-Methyl-2-Pentanone					
2-Hexanone					
Tetrachloroethene					
1, 1, 2, 2-Tetrachloroethane					* *
Toluene				*	
Chiorobenzene					* *
Ethylbenzene				*	•
Styrene					
Total Xylenes					

 $\rm RF_{50}$ -Response Factor from daily standard file at 50 ug $^{\prime}$ I RF -Average Response Factor from initial calibration Form VI

%D -Percent Difference CCC -Calibration Check Compounds (+) SPCC -System Performance Check Compounds (++)

Pesticide Evaluation Standards Summary

(Page 1)

Case No:Region	Laboratory:
Contract No:	GC Column:
Date of Analysis:	Instrument ID:

Evaluation Check for Linearity

Laboratory ID				
Pesticide	Calibration Factor Eval. Mix A	Calibration Factor Eval. Mix B	Calibration Factor Eval. Mix C	% RSD (
Aldrin				
Endrin				
4,4'- DDT ⁽¹⁾				
Dibutyl Chlorendate				

Evaluation Check for 4,4'- DDT/Endrin Breakdown (percent breakdown.expressed as total degradation)

· · · · · · · · · · · · · · · · · · ·	Laboratory I.D.	Time of Analysis	Endrin	4,4'- DDT	Combined ⁽²⁾
Eval Mix B 72 Hour					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
· Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					

(1) See Exhibit E, Section 7.5.4

(2) See Exhibit E, Section 7.3.1.2.2.1 B-24

Pesticide Evaluation Standards Summary (Page 2) Evaluation of Retention Time Shift for Dibutyl Chlorendate Report all standards, blanks and samples

SMO Sample No.	Lab I.D.	Time of Analysis	Percent Diff.	SMO Sample No.	Lab I.D.	Time of Analysis	Percent Diff
·····							
						· · · · · · · · · · · · · · · · · · ·	
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PESTICIDE/PCB STANDARDS SUMMARY

Case No. _____ Laboratory _____ Contract No. _____ GC Column _____ GC Instrument ID _____ DATE OF ANALYSIS _____ DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ TIME OF ANALYSIS LABORATORY ID _____ LABORATORY ID RETENTION CALIBRATION CONF. CONF. CALIBRATION PERCENT OR COMPOUND RT RT OR FACTOR FACTOR DIFE ** WINDOW QUANT. QUANT. alpha - BHC beta - BHC delta - BHC gamma-BHC Heptachlor Aldrin Heptachlor Epoxide Endosulfan I Dieldrin 4.4'-DDE Endrin Endosulfan II 4.4'-DDD Endosulfan Sulfate 4.4'-DDT Me thox ychlor Endrin Ketone Tech. Chlordane alpha-Chlordane# gamma-Chlordane* Toxaphene Aroclor - 1016 Aroclor - 1221 Aroclor - 1232 Aroclor - 1242 Aroclor - 1248 Aroclor - 1254 Aroclor - 1260

SEE EXHIBIT E, PART 7



B-26

FORM IX

Pesticide/PCB Identification

Case No. _____

Laboratory_____

Contract No.

SAMPLE ID	PRIMARY COLUMN	PESTICIDE/ PCB	RT OF TENTATIVE ID	RT WINDOW OF APPROPRIATE STANDARD	CONFIRMATION COLUMN	RT ON CONFIRMATORY COLUMN	RT WINDOW OF APPROPRIATE STANDARD	GC/MS CONFIRMED (Y or N)
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FORM X

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RAS INORGANIC

DELIVERABLES INDEX

- A. <u>Weekly Progress Reports</u> Tabulation of samples received, date of receipt, and a tabulation of problems encountered.
- B. <u>Sample Traffic Report</u> (Original Lab Copy for Return to SMO) Copy of SMO Sample Traffic Report with lab receipt information and original Contractor signature.
- C. <u>Sample Data Package</u> Data report package for analyses of each sample (including all required QA/QC-Exhibit E) must be complete before submission and shall include:
 - 1) Copies of completed SMO Sample Traffic Reports with receipt of information completed for all samples reported in data package.
 - 2) The cover page for the inorganic data package (Exhibit B), including general comments, Statement of Work (SOW) Number, QC Report #, sample EPA cross reference numbers, footnotes used in the data package, and the statement on use of ICP background and interelement corrections for the samples. The SOW number defines the Statement of Work used to obtain the reported values.
 - Tabulated results in ug/L for aqueous samples or mg/kg for solid samples 3) (identification and quantity) of specified chemical constituents (Exhibit C) by the specified analyses (Exhibit D), validated and signed in original signature by the Laboratory Manager, and reported on Form I.* The results for solid samples will be reported on a dry weight basis. Percent solids are not required on aqueous samples. If the value or the result is greater than or equal to the Instrument Detection Limit (IDL), corrected for dilutions, as determined in Exhibit E, report the value and indicate the analytical method used for the metals. Use P for ICP, A for Flame AA and F for Furnace AA. All dilutions not required by the contract and affecting the IDL, must be noted on an element by element basis on Form I. If the value is less than the Contract Required Detection Limits (CRDL) in Exhibit C, put the value in brackets e.g., [10]. If the element was analyzed for but not detected, report the instrument detection limit value with a "U" (e.g., 10U). Use an "E" as the footnote to indicate an estimated value or value not reported due to the presence of interference, and an explanatory note must be included on the cover page. If the duplicate sample analysis is not within the control limits, flag it with an asterisk (*). Use "S" as a footnote to indicate a value determined by Method of Standard Additions (MSA). If the correlation coefficient (r) for method of standard additions is less than 0.995, flag the value with a plus sign (+). If duplicate injection precision criteria specified for Furnace AA analysis in Exhibit E cannot be met, flag the data with an "M". If the spike sample recovery is not within control limits, flag the data with the letter N. Report results to two significant figures for values from 0 to 100 and three significant figures for results greater than 100, with the exception of

^{*}In the event the Laboratory Manager cannot validate all data reported for each sample, he/she will provide a detailed description of the problems associated with the sample.

Mercury (see Mercury Methods – Exhibit D). For rounding rules, follow the EPA Handbook of Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/4-79-019).

Under the comments section on Form I, provide a brief physical description of the sample using the following guidelines:

- A. Water samples Coloration and clarity
- B. Solid samples Coloration, texture and artifacts

Recommended Descriptive Terms

<u>Coloration</u>: Red, blue, yellow, green, orange, violet, white, colorless, brown, grey, black

Clarity: Clear, cloudy, opaque

<u>Texture</u>: Fine (powdery), Medium (sand), Coarse (large crystals or rocks)

Also note any significant changes that occur during sample preparation (i.e., coloration shifts, emulsion formation).

- 4) Analytical results for samples and spikes, duplicates, standards, ICP Interference Check Samples, reagent blanks, laboratory control samples, instrument detection limits and holding times on QA Forms II, III, IV, V, VI, VII, VIII, IX and X. Multiple forms require identification (i.e., Form IIA, Form IIB etc.). Summarize each full method of standard addition performed on Form VIII.
- 5) Legible photocopy of raw data (sequential measurement readout record) clearly labeled with sufficient information to unequivocally identify:
 - a) Calibration standards (including prep date).
 - b) Calibration blanks and preparation blanks.
 - c) Initial and continuing calibration verification standards and interference check samples.
 - d) Diluted and undiluted samples (by EPA number) and all weights, dilutions and volumes used to obtain the reported values. If the volumes, weights and dilutions are consistent for all samples in a given Case, a general statement outlining these parameters is sufficient.
 - e) Duplicates
 - f) Spikes (indicating standard solutions used, final spike concentrations, and volumes involved).
 - g) Any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values.

- h) All information for furnace analysis contained in Form VIII (Exhibit E), clearly identified on the raw data, including sample #, initial single spike data, % recovery, full MSA data, MSA correlation coefficient, slope and intercept of linear fit, and final sample concentration (standard addition concentration).
- 6) Copies of digestion logs for the ICP, flameless AA and Hg preparations and of the distillation log for cyanide. These logs must include: 1) date, 2) sample weights and volumes, 3) sufficient information to unequivocally identify which QC samples (i.e., LCS, preparation blank) correspond to each batch digested, 4) comments describing any significant sample changes or reactions which occur during preparation, and 5) indication of pH<2 or >12, as applicable.
- 7) The order of raw data in the data package shall be: 1) ICP, 2) Flame AA, 3) Furnace AA, 4) Hg, 5) CN, 6) Digestion and Distillation logs, and 7) Percent Solids. All raw data must include intensities (ICP) and absorbances (AA) unless instrument direct readout is in concentration units.
- 8) For each reported value, the data package must contain all raw data from the instrument used to obtain that value and the QA/QC values reported. All AA and ICP instruments must provide a hard copy of the instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct instrument readout must be included in the raw data package.

Information shall include a key to abbreviations, with response units stated and a cross reference to EPA sample numbers.

- D. <u>Results of Intercomparison/Performance Evaluations (PE) Sample Analyses</u> Tabulation of analytical results for intercomparison/PE sample analyses include all requirements specified in C above.
- E. <u>Complete Case File Purge</u> (formerly termed the "Document Control and Chain-of-Custody Package") — The Complete Case File Purge package must include all laboratory records received or generated for a specific case that have not been previously submitted to EPA as deliverables. These items include, but are not limited to: sample tags, custody records, sample tracking records, analysts logbook pages, bench sheets, instrument readout records, computer printouts, raw data summaries, instrument logbook pages (including instrument conditions), correspondence and the document inventory.

Shipment of each Complete Case File Purge package by first class mail, overnight carrier, priority mail or equivalent is acceptable. Custody seals, which are provided by EPA, must be placed on shipping containers and a document inventory and transmittal letter included. The laboratory is not required to maintain any documents for a case after shipment; however, it is recommended that you maintain a copy of the document inventory and transmittal letter.

- F. Quarterly Verification of Instrument Parameters The Contractor must perform and report quarterly verification of instrument detection limits by methods specified in Exhibit E and report type and model # for each instrument used on this contract. For the ICP instrumentation and methods, the Contractor must also report quarterly: linearity range verification, interelement correction factors, wavelengths used and integration times. This information is reported using Forms XI, XII and XIII. Submissions of Quarterly Verification of Instrument Parameters must include the raw data used to determine those values reported.
- G. <u>Results of Solid Laboratory Control Samples (LCS)</u> Tabulation of analytical results and QC for solid LCS analysis, as specified in Exhibit E.

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RAS INORGANIC DATA REPORTING FORMS

U.S. EPA Contract Laboratory Program Sample Management Office P.O. Box 818 - Alexandria, VA 22313 703/557-2490 FTS: 8-557-2490

	COVER	PAGE ES DATA PACKAGE	
Lah Name		Case No.	
SOW No.		0.C. Report	No.
	<u></u>		
	Sample !	Numbers	
EPA No.	Lab ID No.	EPA No.	Lab ID No.
	1		
		·····	
	<u> </u>		<u></u>
			<u> </u>
			
Comments:		<u> </u>	<u> </u>
		<u></u>	
			· · · · · · · · · · · · · · · · · · ·
ICP interclonent and	hadround correctio	ne applied? Yes	No
If was corrections a	ackground correctio	or after	
Footpotes:			inclation of law data.
NR - Not required	by contract at this	time	
Form I:			
Value - If the result detecion lim: report the va method used v U - Indicates ele	: is a value greater it but less than the alue in brackets (i.e with P (for ICP), A ement was analyzed fo	than or equal to the contract-required of , [10]). Indicate (for Flame AA) or F r but not detected.	ne instrument detection limit, e the analytical F (for Furnace AA). 6 Report with the
instrument de E - Indicates a v	etection limit value value estimated or no	(e.g., 10U). t reported due to t	the presence of
interference - Indicates val	Explanatory note i lue determined by Met	ncluded on cover pa	age. lition.
N - Indicates sp:	ike sample recovery i	s not within control	ol limits.
 * - Indicates dup + - Indicates the 	plicate analysis is n e correlation coeffic	ot within control 1 ient for method of	limits. standard addition is
less than 0.	995	Letter vor meenou or	
M - Indicates du	plicate injection res	ults exceeded cont	rol limits.

Indicate method used: P for ICP; A for Flame AA and F for Furnace.

Date _____

	Form I			
U.S. EPA Contract Laboratory Prog Sample Management Office P.O. Box 818 - Alexandria, VA 223 703/557-2490 FTS: 8-557-2490	ram EPA Sample No.			
τηρεσι	TC ANALYSIS DATA SHEFT			
LAR NAME	CASE NO			
SOW NO.	Lab Receipt Date			
LAB SAMPLE ID. NO.	QC REPORT NO.			
Elements	Identified and Measured			
Concentration: Low	Medium			
Matrix: Water Soil	Sludge Other			
ug/L or i	mg/kg dry weight (Circle One)			
l. Aluminum	13. Magnesium			
2. Antimony	14. Mangane se			
3. Arsenic	15. Mercury			
4. Barium	16. Nickel			
5. Beryllium	17. Potassium			
6. <u>Cadmium</u>	18. Selenium			
7. <u>Calcium</u>	19. Silver			
8. Chromium	20. Sodium			
9. Cobalt	21. Thallium			
10. Copper	22. Vanadium			
11. <u>Iron</u>	23. <u>Zinc</u>			
12. Lead	Precent Solids (%)			
Cyanide				
Footnotes: For reporting results as defined on Cover Pa results are encouraged and contained on Cover	to EPA, standard result qualifiers are use age. Additional flags or footnotes explain d. Definition of such flags must be explic r Page, however.			
	· · · · · · · · · · · · · · · · · · ·			
	T al Marazza			
	Lab Manager			
		For	<u>n II</u>	
----	----	--------	-------------	---------
Q.	С.	Report	No.	<u></u>

-

LAB NAME	<u></u>	SOW NO.											
DATE					UNITS: ug/L								
Compound	-		Initia	al Calib '	lib.1 Continuing Calibration ²								
Metals:	ĺ	True	Value	Found	<u>%R</u>	True	Value	Found	<u>%R</u>	Found	<u>%R</u>	Method ⁴	
l. <u>Alumi</u>	num				ļ				ļ				
2. Antim	iony				<u> </u>	ļ							
3. <u>Arsen</u>	ic		·····									ļ	
4. <u>Bariu</u>	ım												
5. Beryl	lium		. <u> </u>						ļ				
6. <u>Cadmi</u>	um		- <u></u>		[
7. <u>Calci</u>	um												
8. Chron	nium												
9. <u>Cobal</u>	t												
0. <u>Coppe</u>	r								Í			<u> </u>	
l. Iron			,										
2. Lead							· .						
3. Magne	sium												
4. Manga	nese												
5. Mercu	ry					Π							
6. Nicke	1												
7. Potas	sium						•						
8. Seler	ium												
9. Silve	r				1						1		
0. Sodiu	ım				1	11							
l. Thall	ium					11					1		
2. Vanad	lium				<u></u>	11							
3. Zinc											1		
ther:						11			ł	• •			
					1	<u> </u>			<u></u>		1		
vanide						††			i				
l Inded - 1			on Co	1	I	<u>ц</u> 2	Contin		1	ion form	<u>+</u>	L.J	

⁴ Indicate Analytical Method Used: P - ICP; A - Flame AA; F - Furnace AA

Form III

Q. C. Report No.

BLANKS

LAB NAME _____

CASE NO.

UNITS

DATE _____

	<u>Initial</u>	Cont	inuing Ca	alibrati	on	Preparat	ion Blank
Compound	Calibration Blank Value		Blank V	Value	 /.	Matrix:	Matrix:
compound	DIGHK VALUE	1	2	J	4	1	2
Metals:							
l. Aluminum							
2. Antimony							
3. Arsenic							
4. Barium							
5. Beryllium							
6. Cadmium							
7. Calcium							
8. Chromium							
9. Cobalt					· · · · ·		
10. Copper							·
ll. Iron							
12. Lead							
13. Magnesium							
14. <u>Mangane</u> se							
15. <u>Mercury</u>							
16. Nickel							
17. Potassium							
18. <u>Selenium</u>							
19. Silver		•					
20. Sodium							
21. Thallium							
22. Vanadium							
23. Zinc							
Other:							
		<u> </u>					- .
Cyanide							

		Fo	<u>rm IV</u>							
Q. C. Report No.										
		ICP INTERFERE	NCE CHECK	SAMPLE						
LAB NAME				CASE NO.						
				Check Samp	ole I.	D				
DATE				Check Samp	ple So	urce				
				Units:	ug/L					
			TT	1						
	Control	Limits ¹		Initial	(U.D.)	Final	<u>1</u>			
Compound	Mean	Std. Dev.	True ²	Observed	~R	Observed	%R			
Metals:										
1. Aluminum		· · · · · · · · · · · · · · · · · · ·	_							
2. Antimony										
3. Arsenic			<u> </u>							
4. <u>Barium</u>			<u> </u>	· · · · · · · · · · · · · · · · · · ·						
5. Beryllium										
6. <u>Cadmium</u>	······································									
7. <u>Calcium</u>			<u> </u>		ļļ					
8. Chromium						<u> </u>				
9. Cobalt										
10. Copper			· · ·	ļ						
11. <u>Iron</u>										
12. Lead										
13. Magnesium	. <u> </u>									
14. Manganese										
15. Mercury					ļ		ļ			
l6. <u>Nickel</u>										
17. Potassium						<u> </u>	 			
18. <u>Selenium</u>					 					
19. <u>Silver</u>	<u></u>	•								
20. <u>Sodium</u>						ļ	 			
21. Thallium				ļ		ļ	ļ			
22. Vanadium				<u> </u>		ļ	 			
23. Zinc	······································				 		ļ			
Other:	·			· · · · · · · · · · · · · · · · · · ·			ļ			
		<u> </u>				<u> </u>	<u> </u>			

1 Mean value based on n = ____.

 2 $\,$ True value of EPA ICP Interference Check Sample or contractor standard.

Form V

Q. C. Report No.

SPIKE SAMPLE RECOVERY

LAB NAME _____

DATE _____

CASE	NO.		
EPA	Sample	No.	
Lab	Sample	ID No.	
Unit	s		

Matrix _____

		Control Limit	Spiked Sample	Sample	Spiked	
Com	pound	%R	Result (SSR)	Result (SR)	Added (SA)	%R ^l
Meta	als:					
1.	Aluminum	75-125				
2.	Antimony					
3.	Arsenic					
4.	Barium	**				
5.	Beryllium					
6.	Cadmium	••				
7.	Calcium	57				
8.	Chromium	11				
9.	Cobalt	11				
10.	Copper	••				
11.	Iron				· · · · · · · · · · · · · · · · · · ·	
12.	Lead					
13.	Magnesium	••				
14.	Manganese					
15.	Mercury					
16.	<u>Nickel</u>		·			
17.	Potassium	**				
18.	Selenium	**				
19.	Silver	**				
20.	Sodium					
21.	Thallium					
22.	Vanadium					
23.	Zinc	**				
0th	er:					
Cya	nide	**				

 $\frac{1}{R} = [(SSR - SR)/SA] \times 100$

"N"- out of control

"NR" - Not required

Comments:

		Form VI		
-	Q. C. R	eport No		
		DUPLICATES		
LAB NAME			CASE NO.	
DATE			EPA Sample No.	
URIL			Units	
	Matri	x		
Compound	Control Limit ¹	Sample(S)	Duplicate(D)	rpd ²
Metals:				
1. <u>Aluminum</u>	·····			
2. Antimony				
3. Arsenic				·
4. Barium		·		
5. Beryllium		·		
6. <u>Cadmium</u>	· · · · · · · · · · · · · · · · · · ·			
7. Calcium	·			
8. Chromium				
9. Cobalt				
10. Copper				
ll. Iron		·		
12. Lead				
13. Magnesium	•			
14. Manganese				
15. Mercury				
16. Nickel				
17. Potassium				
18. Selenium				
19. Silver				
20. Sodium		<u></u>		
21. Thallium				
22. Vanadium	· · · · · · · · · · · · · · · · · · ·	· · · · · · · · · · · · · · · · · · ·		
23. Zinc				
Other:				
Cvanide				
* Out of Control	I	.	1	

¹ To be added at a later date. NC - Non calculable RPD due to value(s) less than CRDL $\frac{2 \text{ RPD} = [|S - D|/((S + D)/2)] \times 100}{2 \text{ RPD} = [|S - D|/((S + D)/2)] \times 100}$

Form VII

Q.C. Report No.

INSTRUMENT DETECTION LIMITS AND

LABORATORY CONTROL SAMPLE

LAB NAME _____ CASE NO. ____ DATE _____

LCS NO.

Com	pound	Required Detection Limits (CRDL)-ug/1	Instrumer Limits (ICP/AA ID#	nt Detection IDL)-ug/1 Furnace ID#	Lab Co <u>ug/L</u> (c True	Lab Control Sample ug/L mg/kg (circle one) True Found %R	
lieta	als:						
1.	Aluminum	200				┼──┼	
2.	Antimony	60					
3.	Arsenic	10			1		
4.	Barium	200	ļ			├ ─── ├	
5.	Beryllium	5			<u> </u>		
6.	Cadmium	5	_	<u> </u>	ļ		
7.	Calcium	5000			ļ	<u> </u>	
8.	Chromium	10			· ·	ļ	
9.	Cobalt	<u>50</u>		· ·			
10.	Copper	25					
11.	Iron .	,100					
12.	Lead	5					
13.	Magnesium	5000				t -	
14.	Manganese	15					
15.	Mercury	0.2					
16.	Nickel	40					
17.	Potassium	5000					
18.	Selenium	5					
Í9.	Silver	10					
20.	Sodium	5000					
21.	Thallium	10					
22.	Vanadium	50		11			
23.	Zinc	20				1	
Oth						11	
Jen			1	11	1		
	nido.	10	NR	NR	1		

NR - Not required

Form VIII

Q.C.	Report	No.	
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STANDARD ADDITION RESULTS

LAB NAME						CASE NO.					
DATE	TE						UNIT	S: U	ıg/L		
EPA Sample #	Element	Matrix	0 ADD ABS.	1 A CON.	ADD ABS ²	2 A CON.	ADD ABS. ²	3 A CON.	ABS. ²	FINAL CON. ³	r*
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- ¹ Matrix abbreviations: Low Solid, LS; Medium Solid, MS; Low Aqueous, LA; Medium Aqueous, MA.
- 2 CON is the concentration added, ABS. is the instrument readout in absorbance or concentration.
- 3 Concentration as determined by MSA
- *"r" is the correlation coefficient.
- + correlation coefficient is outside of control window of 0.995.

Form	IX

Q. C. Report No.

ICP SERIAL DILUTIONS

LAB NAME

DATE _____

CASE NO.	
EPA Sample No.	
Lab Sample ID No.	
Units: ug/L	

Matrix _____

Com	pound	Initial Sample Concentration(I)	Serial Dilution ¹ Result(S)	% Difference ²
Meta	als:			
1.	Aluminum			
2.	Antimony			
3.	Arsenic			
4.	Barium			
5.	Beryllium			
6.	Cadmium			
7.	Calcium			
8.	Chromium			
·у.	Cobalt			
10.	Copper			
11.	Iron			L
12.	Lead			
13.	Magnesium			
14.	Manganese		· · · · · · · · · · · · · · · · · · ·	
15.	Nickel		·	
16.	Potassium			
17.	Selenium			
18.	Silver			
19.	Sodium			-
20.	Thallium			
21.	Vanadium			
22.	Zinc			
0th	er:			

¹ Diluted sample concentration corrected for 1:4 dilution (see Exhibit D) ² Percent Difference = $\frac{|I - S|}{I} \times 100$

NR - Not Required, initial sample concentration less than 10 times IDL NA - Not Applicable, analyte not determined by ICP

Form X

QC Report No.

HOLDING TIMES

LAB NAME

DATE

1

CASE NO. _____

EPA	[Date	Mercury	Mercury	CN Prep	CN
Sample No.	Matrix	Keceived	Prep Date	Holding Time l	Date	Holding Time ¹
				(Days)		(Days)
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 $^{\rm l}{\rm Holding}$ time is defined as number of days between the date received and the sample preparation date.

IFB Amend One.

Form XI (Quarterly) INSTRUMENT DETECTION LIMITS

LAB NAME _____ DATE _____

ICP/Flame AA (Circle One) Model Number_____ Furnace AA Number_____

Element	Wavelength (nm)	CRDL (ug/L)	IDL (ug/L)	Element	Wavelength CRDL (nm) (ug/L)		IDL (ug/L)
l. Aluminum		200		13. Magnesium		5000	
2. Antimony		60		14. Manganese		15	
3. Arsenic		10		15. Mercury		0.2	
4. Barium		200		16. Nickel		40	
5. Beryllium		5		17. Potassium		5000	
6. Cadmium		5		18. Selenium		5	
7. Calcium		5000		19. Silver		10	
8. Chromium		10		20. Sodium		5000	
9. Cobalt		50		21. Thallium		10	
10. Copper		25		22. Vanadium		50	ļ
ll. Iron		100		23. Zinc	 	20	
12. Lead		5					

Footnotes: • Indicate the instrument for which the IDL applies with a "P" (for ICP), an "A" (for Flame AA), or an "F" (for Furnace AA) behind the IDL value.

> • Indicate elements commonly run with background correction (AA) with a "B" behind the analytical wavelength.

• If more than one ICP/Flame or Furnace AA is used, submit separate Forms XI-XIII for each instrument.

COMMENTS:

Lab Manager

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Form XII (Quarterly)

ICP Interelement Correction Factors

LABORATORY_____ ICP Model Number_____

DATE

			Interelement Correction Factors for									
	Analyte	Analyte Wavelength (nm)	Al	Ca	Fe	Mg	/1					
1.	Antimony											
2.	Arsenic											
3.	Barium											
4.	Beryllium									<u> </u>		
5.	Cadmium											
6.	Chromium		<u> </u>									
7.	Cobalt	· · · · · · · · · · · · · · · · · · ·										
8.	Copper		<u> </u>		 	 -				-		
9.	Lead				 							
10.	Manganese			ļ 								
11.	Mercury		ļ		 							
12.	Nickel	: 	ļ		ļ	. 						
13.	Potassium		l	ļ	ļ							
14.	Selenium			 	ļ			. i				
15.	Silver			[
16.	Sodium			ļ	 	ļ						
17.	Thallium		<u> </u>	ļ	ļ				 			
18.	Vanadium			,				· · · · · · · · · · · ·	ļ			
19.	Zinc					l			Ĺ			
Сом	MENTS:											

Lab Manager_____

Form XII (Quarterly) (cont'd)

ICP Interelement Correction Factors

ICP Model Number_____

			1		Interel	amont C	orrecti	n Fast	ore	
			1		Interer	ement C	or	on race	brs	
	Analyte	Analyte Wavelength (nm)								
1.	Antimony									
2.	Arsenic	•				 		!		
3.	Barium				ļ					
4.	Beryllium	<u></u>		_						
5.	Cadmium					 				
6.	Chromium									
7.	Cobalt									
8.	Copper			_						
9.	Lead					 				
.0.	Manganese	# <u></u>	-						ļ	
1.	Mercury		_		ļ				 	
2.	Nickel									
3.	Potassium	<u> </u>								
.4.	Selenium	·····					 			
5.	Silver		_						 	
6.	Sodium				4		ļ			
7.	Thallium		-							
.8.	Vanadium									
9.	Zinc									

Lab Manager_____

Form XIII (Quarterly) ICP Linear Ranges

LAB NAME _____ ICP Model Number _____

DATE _____

Analyte	Integration Time	Concen- tration	Analyte	Integration Time	Concen- tration
	(Seconds)	(ug/L) ,		(Seconds)	(ug/L)
1. Aluminum			13. Magnesium		· · · · · · · · · · · · · · · · · · ·
2. Antimony			14. Manganese		
3. Arsenic			15. Mercury		
4. Barium			16. Nickel		
5. Beryllium			17. Potassium		
6. Cadmium			18. Selenium		
7. Calcium			19. Silver		
8. Chromium			20. Sodium		
9. Cobalt			21. Thallium		
10. Copper			22. Vanadium		
11. Iron			23. Zinc		
12. Lead					

Footnotes: • Indicate elements not analyzed by ICP with the notation "NA".

COMMENTS:

Lab Manager

RAS DIOXIN DELIVERABLES INDEX

I. Case Narrative

The case narrative must contain: Case number; contract number; summary of any QC, sample, shipment, and analytical problems; and documentation of all internal decision tree processes used. Outline problems encountered and final solutions. Be as specific and detailed as necessary.

- II. Tabulated Data (Form B-1 to Form B-4)
- III. Standard Data By Instrument
 - A) All quantitation reports, and SIM mass chromatgrams for concentration calibration solutions

 Organized in order: Triplicate runs of CC1 Triplicate runs of CC2 Triplicate runs of CC3 etc.

- B) All quantitation reports, and SIM mass chromatograms for routine calibration
 - 1. Performance check solutions organized in chronological order
 - Concentration calibration solution #1 organized in chronological order
- IV. Sample Data
 - A) All quantitation reports, and SIM mass chromatograms for method blank(s) organized in chronological order
 - B) All quantitation reports, and SIM mass chromatograms for samples (organized by increasing EPA number)
- V. Chain of Custody and In House Laboratory Control Documents
 - A) EPA Chain of Custody Records
 - B) SMO Dioxin Shipment Records
 - C) Sample Log-in Sheets
 - D) In-House Dioxin Bench Sheet
 - E) GC/MS Standard and Sample Run Logs

RAS DIOXIN DATA REPORTING FORMS

Case/Batch	No:				Column:						
Instrument	ID:	<u></u>									
EPA Sample No.	Extr. Date	Wet wt	ug/kg Meas.	g/kg TCDD as. MPC	GC/MS Date	Analysis Time	Surr. S/N Ratio	* % REC(IS)			
							· · · · · · · · · · · · · · · · · · ·				
						-					
							<u> </u>				
								·			
MB = Met N = Nat	hod Bla ive TCI	ink)D Spike	2			FB IS	Field Bl Internal	lank L Standard			

FORM B-1S. TCDD SOIL DATA REPORT FORM Page 1 of 2

Lab:

Report Date:_____

RS = Recovery Standard ND = Not Detected

RR = Rerun

1

D = Duplicate/Fortified Field Blank

PE = EMSL-LV Performance Evaluation Sample

MPC = Maximum Possible Concentration *Note: Relative to ${}^{13}C_{12}-1,2,3,4-TCDD$

FORM B-1S. TCDD SOIL DATA REPORT	FORM
----------------------------------	------

Lab:	<u></u>	
Case/Batch No:		

Report Date:_____

Column:____

Instrument ID:

		Rela										
EPA	Response Ratios						Response (Area)					
Sample	320/332/		332/									
Number	322	334IS	334RS	259	320	322	328	332IS	334IS	332RS	334RS	
	-								فساري بير معندا المقسمي			
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MB = Method Blank

N = Native TCDD Spike

D = Duplicate/Fortified Field Blank

PE = EMSL-LV Performance Evaluation Sample

MPC = Maximum Possible Concentration

FB = Field Blank

IS = Internal Standard

- RR = Rerun
- ND Not Detected
- RS = Recovery Standard

FORM B-1W. TCDD WATER DATA REPORT FORM Page 1 of 2

Lab: _____

Case/Batch No:_____

Instrument ID:

			_			_						
EPA	•	Extr.		ng/L	TCDD	GC/MS	Analysis	Sı	ırr.			*
Sample	No.	Date	volume	Meas.	MPC	Date	Time	S/N	Ratio	7	REC(I	<u>S)</u>
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MB = Method Blank

N = Native TCDD Spike

D = Duplicate/Fortified Field Blank

PE = EMSL-LV Performance Evaluation Sample

- MPC = Maximum Possible Concentration *Note: Relative to ${}^{13}C_{12}$ -1,2,3,4-TCDD
- FB = Field Blank

IS = Internal Standard

RR = Rerun

RS = Recovery Standard

9/86

ND = Not Detected

Report Date:_____

Column:

FORM	B-1W.	TCDD	WATER	DATA	REPORT	FORM
------	-------	------	-------	------	--------	------

Lab:

Report Date:_____

Column:___

Instrument ID:

Case/Batch No:

		Rela	•								
EPA	Respo	onse Ra	atios				Respons	e (Area)		
Sample	320/	332/	332/					The second se			
Number	322	334TS	334RS	259	320	322	328	33215	334IS	332RS	334RS
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MB = Method Blank

N = Native TCDD Spike

D = Duplicate/Fortified Field Blank

- PE = EMSL-LV Performance Evaluation Sample
- MPC = Maximum Possible Concentration
- FB = Field Blank
- IS = Internal Standard
- RR = Rerun
- ND = Not Detected
- RS = Recovery Standard

A. TCDD REPORT FORM (Form B-1)

This form is used for tabulating and reporting case results.

Complete the header information at the top of the page including instrument ID, laboratory name, case/batch number, report date, and column used.

EPA sample number is tabulated along with date sample was extracted, and weight (wet) extracted to the nearest tenth (0.1) of a gram or volume extracted (water) to the nearest 10 milliliters.

Calculate the concentration of 2,3,7,8-TCDD using the formula:

$$C_{\mathbf{x}} = \frac{A_{\mathbf{x}} \cdot Q_{\mathbf{IS}}}{A_{\mathbf{is}} \cdot RRF_{\mathbf{n}} \cdot W}$$

- $C_x = 2,3,7,8$ -TCDD concentration in ug/kg or ug/L
- A_X = the sum of integrated ion abundance detected for m/z 320 and 322
- A_{1S} = the sum of integrated ion abundances detected for m/z 332 and 334 (characteristic ions of ${}^{13}C_{12}$ -2,3,7,8-TCDD the internal standard).
- Q_{is} = quantity (in ng) of ${}^{13}C_{12}$ -2,3,7,8-TCDD added to the sample before extraction
- RRF_n = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to ${}^{13}C_{12}$ -2,3,7,8-TCDD
 - W = The weight (in g) of soil/sediment extracted or volume of water extracted (in mL)

Positive samples are quantitated with values >10.0 ug/kg or 100 ng/L recorded to three (3) significant figures and those values <10.0 ug/kg or 100 ng/L reported to two (2) significant figures.

For samples in which unlabeled 2,3,7,8-TCDD was not detected calculate the estimated maximum possible concentration, which is the concentration required to produce a signal with a peak height of 2.5 times the background signal height.

Use the formula:

$$MPC = \frac{2.5 \cdot H_x \cdot Q_{is}}{H_{is} \cdot RRF_n \cdot W}$$

where: MPC = maximum possible concentration of unlabeled 2,3,7,8-TOUP required to produce H_x .

- H_x = peak height for m/z 320 or 322 in the same group of >5 scans used to measure A_{1s} .
- H_{is} = peak height for the appropriate ion characteristic of the internal standard, m/z 332 when 320 is used to determine A_x , and m/z 334 when 322 is used to determine A_x .
- Q_{is} = quantity (in ng) of ${}^{13}C_{12}$ -2,3,7,8-TCDD added to the sample before extraction.
- RRF_n = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to ${}^{13}C_{12}$ -2,3,7,8-TCDD.
 - W = weight (in g) of wet soil/sediment sample or volume of water extracted (in mL).

Report GC/MS Instrument ID, the date and time the analysis was performed, and the signal to noise ratio for the surrogate compound.

FORM B-2

INITIAL CALIBRATION SUMMARY

Laboratory:

CC Solution Alternative:

Case/Batch No.: _____

Instrument ID: _____

						AREA			
Date	Time	Sol. ID	320	322	328	332IS	334IS	332RS	334RS
		CC1 CC1 CC1							
		CC2 CC2 CC2							
		CC3 CC3 CC3			*				
		CC4 CC4 CC4		•	t / t / / t / t	-			

Solution ID Codes:

CCl = Concentration calibration solution #1 CC2 = Concentration calibration solution #2 CC3 = Concentration calibration solution #3 CC4 = Concentration calibration solution #4

* Not present in CC Solution Alternative One. FORM B-2

INITIAL CALIBRATION SUMMARY

Laboratory: _____ CC Solution Alternative: _____

Case/Batch No.: _____

Instrument ID:

Date	Time	Sol. ID	Measured RRF _n	Mean RRF _n	Measured RRF ₁	Mean RRF <u>i</u>
		CC1 CC1 CC1				
		CC2 CC2 CC2				
		CC3 CC3 CC3				
		CC4 CC4 CC4				

Solution ID Codes:

- CC1 = Concentration calibration solution #1
- CC2 = Concentration calibration solution #2
- CC3 = Concentration calibration solution #3
- CC4 = Concentration calibration solution #4

ZRSD: RRF _n	RRFi			
CC1=				
CC2=				
CC3=				
CC4=		Native Mean	IS Me	ean
		of Means:	of Me	ans:

B. Initial Calibration Summary (Form B-2)

Record all routine calibrations (PCS and CCl) performed during initial calibration on form B-3.

Complete all header information including laboratory, case/batch number, and instrument ID and EPA CC Solution Alternative.

Date and time along with response for each ion is recorded for each calibration solution. The response factors are calculated with the following equations:

RRF_n (native Response Factor) RRF₁ (internal Standard Response Factor)

$$RRF_{n} = \frac{A_{x} \cdot Q_{is}}{A_{is} \cdot Q_{n}} \qquad RRF_{i} = \frac{A_{is} \cdot Q_{rs}}{A_{rs} \cdot Q_{is}}$$

Where:

- A_x = the sum of integrated ion abundance of m/z 320 and 322 for unlabeled 2,3,7,8-TCDD
- $A_{is} =$ the sum of integrated ion abundances of m/z 332 and m/z 334 for ${}^{13}C_{12}-2,3,7,8-TCDD$
- A_{rs} = the sum of integrated ion abundance of m/z 332 and m/z 334 for ${}^{13}C_{12}$ -1,2,3,4-TCDD

$$Q_n = quantity of unlabeled 2,3,7,8-TCDD injected$$

 Q_{is} = quantity of ${}^{13}C_{12}-2,3,7,8-TCDD$ injected

$$Q_{rs} = quantity of {}^{13}C_{12}^{-1,2,3,4-TCDD}$$

Calculate the mean RRF and the percent relative standard deviation for the triplicate runs of each calibration solution.

$$%$$
RSD = $\frac{SD}{x}$ 100

B-58

Where:

SD =
$$\sqrt{\frac{N}{\frac{1}{2}} \frac{(X_{1} - \overline{X})^{2}}{\frac{1}{1} - \frac{1}{N} - 1}}$$

 \overline{X} = mean of each of the three Response Factors respectively

From the 4 mean native response factors and 4 mean internal standard response factors: calculate the mean of means for each respective RRF's.

FORM	B-3
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ROUTINE CALIBRATION SUMMARY

Laboratory: _____ CC Solution Alternative:

Case/Batch No.: _____ Instrument ID: _____

(CC1) (PCS) PERFORMANCE CHECK SOL. CON. CALIB. SOL. #1 Date Time Response 259 320 322 328 332IS 334IS 332RS 334RS Ratios 320/322 332/334IS 332/334RS . • _____ -------RRFn RRF1 7 Valley

C. Routine Calibration Summary (Form B-3)

Complete the header information including the laboratory, instrument ID Case/Batch number and EPA CC Solution Alternative.

For each performance check solution analyzed complete the date and time of analysis, the response for m/z 259, 320, and 322 for unlabeled 2,3,7,8-TCDD, 328 for ${}^{37}Cl_4-2,3,7,8-TCDD$, and 332 and 334 for ${}^{13}Cl_{12}-2,3,7,8-TCDD$ and ${}^{13}Cl_{12}-1,2,3,4-TCDD$.

Ion ratios for m/z 320/322, m/z 332/334 for ${}^{13}C_{12}$ -2,3,7,8-TCDD and m/z 332/334 for ${}^{13}C_{12}$ -1,2,3,4-TCDD are to be calculated and recorded.

Response factors are to be calculated as in the Initial Calibration Summary (Section B).

For calculation of valley percent see Section D, Section 9.2.6.1.

For each Concentration Calibration Solution #1 used in Routine Calibration, complete all the above information.

FORM B-4

QUALITY CONTROL SUMMARY

Laboratory Name		Case/Batch No	
Instrument ID			
	SOIL		
Accuracy, Fortified/ Spike Field Blank:		EPA Sample Number:	
Relative Difference (%), Duplicate Analysis:		EPA Sample Number:	
	WATER		
Accuracy, Fortified/ Spike Field Blank:		EPA Sample Number:	
Relative Difference (%), Duplicate Analysis:		EPA Sample Number:	

.

D. QC Summary

Complete all the header information.

Report the sample number for the fortified field blank and the % accuracy of the fortified/spike field blank by using the following equation:

amount measured 2 accuracy = ----- x 100 1.0

Record the sample used for duplicate and the Relative Percent Difference which is calculated as follows:

$$RPD = \frac{|s_1 - s_2|}{\frac{s_1 - s_2}{\frac{s_1 - s_2}{2}}} \times 100$$

Where:

 S_1 and S_2 represent sample and duplicate sample results.

APPENDIX C

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SAMPLE INFORMATION AND DOCUMENTATION

ORGANIC SAMPLE COLLECTION REQUIREMENTS



SOIL/SEDIMENT SAMPLES	REQUIRED	CONTAINER TYPE
EXTRACTABLE ANALYSIS (LOW OR MEDIUM LEVEL*)	6 OZ.	1 × 8-0Z. WIDE-MOUTH GLASS JAR
		OR
		2 × 4-0Z. WIDE-MOUTH GLASS JARS
VOLATILE ANALYSIS (LOW OR MEDIUM LEVEL*)	240 ML	2 ×120-ML WIDE-MOUTH GLASS VIALS

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

INORGANIC SAMPLE COLLECTION REQUIREMENTS

WATER SAMPLES	REQUIRED VOLUME	CONTAINER TYPE
METALS ANALYSIS (LOW LEVEL)	1 LITER	1 × 1-LITER POLYETHYLENE BOTTLE
METALS ANALYSIS (MEDIUM LEVEL*)	16 OZ.	1 × 16-OZ. WIDE-MOUTH GLASS JAR
CYANIDE (CN ⁻) ANALYSIS (LOW LEVEL)	1 LITER	1 × 1-LITER POLYETHYLENE BOTTLE
CYANIDE (CN ⁻) ANALYSIS (MEDIUM LEVEL*)	16 OZ.	1 × 16-OZ. WIDE-MOUTH GLASS JAR

SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME	CONTAINER TYPE
Metals and cyanide (CN ⁻) Analysis (Low or medium level*)	6 OZ.	1 × 8-OZ. WIDE-MOUTH GLASS JAR OB
		2 × 4-OZ. WIDE-MOUTH
		GLASS JARS

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

DIOXIN SAMPLE COLLECTION REQUIREMENTS

WATER SAMPLES	REQUIRED	 CONTAINER TYPE
2,3,7,8-TCDD ANALYSIS (MULTI-CONCENTRATION)	2 LITERS	2 × 1-LITER AMBER GLASS BOTTLES
SOIL/SEDIMENT SAMPLES	REQUIRED	 CONTAINER TYPE
2,3,7,8-TCDD ANALYSIS (MULTI-CONCENTRATION)	4 OZ.	1 × 4-OZ. WIDE-MOUTH GLASS JAR
		OR 1 × 8-OZ. WIDE-MOUTH GLASS JAB

HIGH HAZARD SAMPLE COLLECTION REQUIREMENTS

LIQUID OR SOLID SAMPLES	REQUIRED		CONTAINER	TYPE
ORGANIC AND INORGANIC ANALYSIS	6 OZ.		1 × 8-OZ. WIE GLASS .	DE-MOUTH JAR
*All Medium Level Samples To) be sealed in	METAL PAINT CAN I	FOR SHIPMENT	

SPECIAL ANALYTICAL SERVICES

Client Request

	Regional Transmittal	Telephone Request
А.	EPA Region/Client:	
в.	RSCC Representative:	
c.	Telephone Number:)	
D.	Date of Request:	
E.	Site Name:	

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):

3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):

Estimated date(s) of collection: 4. Estimated date(s) and method of shipment: 5. 6. Number of days analysis and data required after laboratory receipt of samples: _____ 7. Analytical protocol required (attach copy if other than a protocol currently used in this program): . 8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): . ····· 9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion. 10. Other (use additional sheets or attach supplementary information, as needed): . Name of sampling/shipping contact: 11. Phone: ()

12. Data Requirements

	Parameter	Detection Limit	Precision Desired (* % or Concentration)
	······································		
	······································		
10			
13.	<u>Audits Required</u>	Frequency of Audits	Limits (Percent or Concentration)
	·	·	
14.	Action Required if Limits	are Exceeded	
·			

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

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CONTRACT LABORATORY PROGRAM RAS RE-ANALYSIS REQUEST/APPROVAL RECORD

SEC	TIOI	NA							
#1.	Ca	se No.		SAS No	#	2. DPO or R	scc		
#3.	De	tails of R	le-Analysis I	Request:					
	0	Laborato	ory Name:	· · · · · · · · · · · · · · · · · · ·					
	0	Sample I	No(s). + Frac	:tion(s):					
	0	Reason f	for Re-Analy	/sis:					
	o	Procedu	re for Re-A	nalysis:					
#4.	Na	me of PC REQU) Contacted	Approved	Not	Approved _	Date _	/	_/
		RE-A	NALYSIS:	Billable	Not	Billable			
∦ 5.	Na	ime of SN	10 Contact:				Date		
SEC	TIOI	NB (ТО	BE COMPL	ETED BY SMO)					
#1.	Da	te of Lab	oratory Not	ification (Verbal)	/	<u> </u>			
∦ 2.	Re	-Analysis	s Start Date			#3. Data [Due Date _	/	_/
SEC	TIOI	NC (PR	OJECT OFF	ICER CONCURRE	INCE)				
Con	curr	ence By	Project Offi	cer Signature	<u> </u>		Date	/_	_/
				Return intac	t form 1	to:			
				Sample Manage P.O. Bo Alexandria, Vi	ement O x 818 rginia	ffiœ 22313			
I	Distr	ribution:	(1) PO Cor	y (2) DPO/RSCC	Сору	(3) SMO Fil	e Copy (4) Lab (Сору
									7/22/86

CONTRACT LABORATORY PROGRAM SAS RE-ANALYSIS REQUEST/APPROVAL RECORD

SECT	ION	IA						
#1.	SA:	S No #2. DPO or RSCC						
# 3.	De	tails of Re-Analysis Request:						
	0	Laboratory Name:						
	0	Sample No(s). + Parameter(s):						
	o	Reason for Re-Analysis:						
	0	Procedure for Re-Analysis:						
#4.	Na	me of SMO Contact: Date _/ / REQUEST: Approved Not Approved						
		RE-ANALYSIS: Billable Not Billable						
SECT		VB (TO BE COMPLETED BY SMO)						
#1.	Da	te of Laboratory Notification (Verbal)						
∦ 2.	Re	-Analysis Start Date // #3. Data Due Date //						
SECI	101	IC (COORDINATOR CONCURRENCE)						
Conc	urre	Ence By Date / /						
		Return intact form to:						
	Sample Management Office P.O. Box 818 Alexandria, Virginia 22313							
		Distribution: (1) DPO/RSCC Copy (2) SMO File Copy (3) Lab Copy						
		7/22/86						

SAMPLE DOCUMENTATION

.

ORGANI			ORT		
(1) Case Number:	② SAMPLEC	ONCENTRATI Check One)	ON (C) Ship To:	
Sample Site Name/Code:	Low (Medi	Concentration um Concentratio	an		
	3 SAMPLEN	ATRIX		Ättn: Hen anne anne (
	(Check (Water Soil/S	one) Sediment	1	Fransfer Ship To:	
(5) Regional Office: Sampling Personnel:	6 For each sam of containers on each bottle	uple collected sp used and mark v a.	ecify mimb colume leve	ear 1	
(Name)		Number of Containers	Approxim Total Volu	ate	
(Phone) Sampling Date:	Water (Extractable)				
(Begin) (End)	- Water (VOA)				
(7) Shipping Information	Soil/Sediment				
	Water (Ext/VOA)				
Name of Carrier	Other				
Date Shipped:					
		•			
Airbill Number:	· · · · · · · · · · · · · · · · · · ·		······		
(8) Sample Description			9 Sample	Location	
Surface Water	Mixed Media				
Ground Water ′_	Solids				
Leachate	Other (specify).			-	
10 Special Handling Instru	uctions:				

D Case Number: 5859	② SAMPLE C	VIC REP(ONCENTRATIOn heck One)	ORT) Shi An	PTO: NAL, LAB		
Sample Site Name/Code:	Low C	Concentration um Concentratio	an	10 An	D MAIN ST. AY TOWN, MA		
DRUM SITE SMALL TOWN, ME #01	(3) SAMPLE M (Check C 	LATRIX me) ediment	Attn: GC MASSPEC Transfer Ship To:				
B) Regional Office: Image: Complex state ampling Personnel: Image: Complex state JOE SAMPLER (Name) Image: Complex state	6 For each same of containers on each bottle	ple collected sp used and mark v	olume leve	ber al nate	(1) Analysis Lab: Rec'd by: Date Rec'd: Sample Condition on Receipt (e.g., broken, no		
<u>6/7/555-/2/2</u> (Phone) Sampling Date:	Water (Extractable)	Containers	Total Volu 80 o z	ime	ice, Chain-of-Custody, etc.)		
Begin) (End)	(VOA)	2	80 m	١			
) Shipping Information	Soil/Sediment (Extractable)						
EDERAL EXPRESS	Soil/Sediment (VOA)						
11 / 4 / 86 Date Shipped:	Other						
1234567890 Airbill Number:	•						
		· · · · · · · · · · · · · · · · · · ·			<u></u>		
Surface Water	Mixed Media		(9) Sample Location				
Called Water Solids Leachate Other (specify)				# GKL6RO 12			
10 Special Handling Instructions: (e.g., safety precautions, hazardous nature) - PEST/PCB/VOA ONLY - MATCHES INORGANIC SAMPLE MAA 001							

*Form to be revised

U.S. ENVIRONMENTAL PROTECTION AGENCY HWI Sample Management Office PO. Box BIB, Alexandria, VA 22313-703 /557-2490 FTS 557-2490 INORGANICS TRAFFIC REPORT						
Case Number: Sample Site Name/Code:	SAMPLE CONCENTRATION (Check One) Low Concentration Medium Concentration SAMPLE MATRIX (Check One) Water Scil/Sediment	Ship To: Attn: Transfer Ship To:				
Sampling Office: Sampling Personnel:	(Shipping Information: Name Of Carrier:					
(Name) (Phone) Sampling Date: (Begin) (End)	Date Shipped: Airbill Number:					
Sample Description: (Check One) Surface Water Ground Water Leachate Mixed Media Solids Other	Mark Volume Level On Sample Bottle Check Analysis required Total Metals Cyanide					

U.S. ENVIRONMENTAL PR PO. Box 818, Alexandria, VA 2231 INORGANICS	OTECTION AGENCY HWI Sample Man 3–703/557-2490 •FTS/557-2490 S TRAFFIC REPORT	agement Office Sample Number MAA 001
(1) Case Number: <u>5859</u> Sample Site Name/Code: <u>DRUM SITE</u> <u>SMALLTOWN, ME</u> # 01	SAMPLE CONCENTRATION (Check One) Low Concentration Medium Concentration SAMPLE MATRIX (Check One) Water Soil/Sediment	(a) Ship To: METALS LAB 100 MAIN STREET ANY TOWN, CA 54321 Attn: AA FERNAS Transfer Ship To:
Sampling Office:	 Shipping Information: Name Of Carrier: FEDERAL EXPRESS Date Shipped:	ANALYSIS LAB: Recd by: Date Recd: 10 Sample Condition
Sample Description: (Check One) Surface Water Ground Water Leachate Mized Media Solids Other	Mark Volume Level On Sample Bottle Check Analysis required Total Metals Cyanide	On Receipt: (eg. broken, leakage, chain of custody, etc.)

* Form to be revised

Sample Management	Office	rainia	2231	3			C,	ASE NO:		8	ATCH NO:	
FTS 8-557-2490 70	3/557-24	90	(CLP C		SHIPM	ENT	RECORD)	S	AS NO:	
Site Name:		Reg	ion l	Number	r:]	Ship To:					
City & State:		Sampling Contact:							-			
FPA Site Sn (11 T						Date	Shipped:		-			
EPA Site Spill I.D.:			(name) /(company)							- 2		
Tier: 1 2 3 4 5	67 •e)										No. V. Contraction	
Instructions: 1) Ship 2) Send rinsste sample 3) Sample Requirements <u>Aqueous</u> : 2 Liters/s sample/batch for la	all samples in TCE s in TCE sample in th QC.	les as or he ed: 4 amber	: Mediu xane o oz./sa glass	m level rganic mple in . Send	, in pair solvents glass ja one 4 L	nt cans. er iter						
	MAT (chec	RIX	ne/sa	mple)		DESCA	PTION		SAS	ONLY		
SAMPLE NUMBERS	SOIL/ Sediment	Αςυεούς	EQUIP RINS (ORG SOLV)	OTHER (SAS ONLY)	SAMPLE TO (c ^{SPIKE} one)	SAMPLE TO DUPLICATE (check one)	SAMPLE	LOCATION (or other field desc)	SPECIFY ADDI TIONAL SAS	ANALYSES paramaters)		
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* Form to be revised

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U.S. ENVIRONMENTAL PROTECTION AGENCY CLP Sample Management Office PO Box 816 - Alexandra Vergina 22313 Phone 703/557/2490 - FTS 557/2490

IGH HAZARD TRAFFIC REPORT

FIELD SAMPLE RECORD

Ε

6402

Case Number: Sample Site Name/Code:	Pield Sample Description: Drum Aqueous Liquid Studge Solid Oil Other 	3) Ship To:Attn:
(4) Sampling Office:	5 Known or Suspected Hazards:	6 Sample Location:
Sampling Personnel:		
(name)	·	
(phone)		
Sampling Date:	7 Preparations Requested:	
(begm) (end)	(check below) Sample Volume:	
⁽⁸⁾ Shipping Information:	- Organics Volatile Organics Base/Neutral, Acid.	
(name of carrier)	Pesticides, PCB	
(date shipped)	Inorganics Total Metals Total Mercury Strong Acid Anions	
(airbil number)		
(9) Special Handling Instruct	tions:	
	SMO Copy	

C-17

	FIELD SAMPLE RECORD	
1) Case Number: /872 Sample Site Name/Code: D v M P	(2) Field Sample Description: X Drum Aqueous Liquid Sudge Solid Oli Other	3 Ship To: Anal.Lab IDO Main St. Anytown, MD 12345 Attn: Steve Kunen
4) Sampling Office:	5 Known or Suspected Hazards:	6 Sample Location: B 7039
Sampling Personnel: John Angelo (name) 312 (66-5432 (phone)	Inert Liquid	S 0808
Sampling Date: 3/16/96 (begin) (end)	Preparations Requested: (check below) Sample Volume: <u>4 o z</u>	
8) Shipping Information: <u>Federal Express</u> (name of camer)	X. Volatile Organics X. Base/Neutral, Acid, TCDD X. Pesticides, PCB	
<u>7/14/86</u> (date shipped) <u>288 4/17 121</u> (airbill number)	 Inorganics Total Metals Total Mercury Strong Acid Anions CN Confirmation Sulfide Confirmation 	

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



SPECIAL ANALYTICAL SERVICE

PACKING LIST

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

Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
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2.		
3.	· · · · · · · · · · · · · · · · · · ·	
4.		
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19.		
20.		
		For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

SAS Number 1000 - A

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

SPECIAL ANALYTICAL SERVICE

PACKING LIST

Sampling Office: <u>Region I</u> Sampling Contact: <u>JIM SAMPLER</u> (name) <u>GI7/555-/J/J</u> (phone)	Sampling Date(s): 11/2 - 11/4/86 Date Shipped: 11/4/86 Site Code: # O1	Ship To: SAS LAB 100 MAIN STREET ANY TOWN, CO 98765 Attn: TP SELVEKS	For Lab Use Only Date Samples Rec'd: Received By:
Sample Numbers 1. <u>1000 A - 0 1</u>	Samp i.e., Analysis, <u>Low CoNC, w</u> A	De Description Matrix, Concentration ATER - 24-D; 24,5-TP	Sample Condition on Receipt at Lab
2. <u>1000A -02</u>	<u> </u>	<u> </u>	·
4. 1000A-04			
5. 1000A-05	13	• 1	
6. 1000A-06		• • •	
7. 1000A-07		•1	
8. 1000 A-08	<u> </u>	L İ	
9			
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11		<u> </u>	
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White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

Custody Seal



Sample Tag





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ENVIRONMENTAL PROTECTION AGENCY

Office of Enforcement

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SAMPLE PACKAGING AND SHIPMENT

SAMPLE PACKAGING SUMMARY



- ENCLOSE ALL SAMPLE CONTAINERS IN CLEAR PLASTIC BAGS.
- PACK ALL MEDIUM AND HIGH LEVEL WATER AND SOIL SAMPLES IN METAL PAINT CANS.
- LABEL PAINT CANS WITH SAMPLE NUMBER OF SAMPLE CONTAINED INSIDE.
- SURROUND CONTENTS OF CAN WITH NON-COMBUSTIBLE, ABSORBENT PACKING MATERIAL.
- USING FREEZER PACKAGES OR ICE SEALED IN PLASTIC BAGS, COOL ORGANIC LOW OR MEDIUM SAMPLES AND INORGANIC SAMPLES TO BE ANALYZED FOR CYANIDE TO 4°C.
- ICE IS NOT REQUIRED IN SHIPPING LOW LEVEL SOIL SAMPLES, BUT MAY BE UTILIZED AT THE DISCRETION OF THE SAMPLER.
- DO <u>NOT</u> COOL DIOXIN, INORGANIC LOW LEVEL WATER, INORGANIC MEDIUM/HIGH LEVEL WATER OR SOIL, OR ORGANIC HIGH LEVEL WATER OR SOIL SAMPLES.
- PACK SEALED PAINT CANS OR PLASTIC-ENCLOSED SAMPLE BOTTLES IN SHIPMENT CONTAINER.
- USE A METAL ICE CHEST FOR SHIPMENT (DO <u>NOT</u> USE CARDBOARD OR STYROFOAM CONTAINERS TO SHIP SAMPLES).
- SURROUND CONTENTS WITH NON-COMBUSTIBLE, ABSORBENT PACKING MATERIAL (DO <u>NOT</u> USE EARTH OR ICE PACKING MATERIALS).
- TAPE PAPERWORK IN PLASTIC BAGS UNDER COOLER LID.
- CLOSE COOLER AND SEAL WITH CUSTODY SEALS.

SAMPLE SHIPMENT COORDINATION CHECKLIST

IMMEDIATELY UPON SHIPMENT OF SAMPLES, SAMPLERS CALL SMO AT (703/557-2490), WITH THE FOLLOWING INFORMATION:



- CASE AND/OR SAS NUMBER
- NAME OF LABORATORY
- DATE OF SHIPMENT
- CARRIER, AIRBILL (SHIPMENT) NUMBERS AND TYPE OF SERVICE
- NUMBER AND MATRICES (WATERS, SOILS, ETC.) OF SAMPLES SHIPPED
- INFORMATION ON COMPLETIONS, CHANGES, DELAYS, CONTINUATIONS, ETC., PERTINENT TO THE CASE
- SAMPLER'S NAME, REGION, AND PHONE NUMBER
- SMO <u>MUST</u> BE NOTIFIED BY 3:00 PM ON FRIDAY FOR SAMPLES INTENDED FOR SATURDAY DELIVERY/PICKUP

POTENTIAL PROBLEMS WITH SAMPLE SHIPMENT AND ANALYSIS

- INCORRECT OR INCOMPLETE PAPERWORK
- LABORATORY RECEIPT OF INCORRECT SAMPLES
- INSUFFICIENT VOLUME FOR ANALYSIS REQUESTED
- o BROKEN OR LEAKING SAMPLES.
- MATRICES OTHER THAN WATER OR SOIL (I.E., ROCKS, LEAVES, STICKS, OIL, ETC.)
- NON-HOMOGENEOUS/MULTI-PHASE WATER OR SOIL SAMPLES
- ANALYTICAL PROBLEMS WITH SAMPLES
- LABORATORY ACCIDENTS INVOLVING SAMPLES

IF ANY OF THESE PROBLEMS ARE ENCOUNTERED, CONTACT SMO IMMEDIATELY

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In Reference to Case No(s):

Contract Laboratory Program REGIONAL/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

Date of Call:	•	· · · · · · · · · · · · · · · · · · ·	
Laboratory Name: Lab Contact:			
Region: Regional Contact:			
Call Initiated By:	Laboratory	Region	
In reference to data for the	e following sample nu	mber(s):	
Summary of Questions/Issu	es Discussed:		
·····	· · · · · · · · · · · · · · · · · · ·		· · · · · · · · · · · · · · · · · · ·
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Summary of Resolution:			
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Signati	Ire	· · · · · · · · · · · · ·	Date

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy

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CONTRACT LABORATORY PROGRAM

Deputy Project Officer Communication Summary

Date DPO Notified of Issue:	DPO Notified By:
Subject Laboratory:	Case/Sas No:
Contact for Resolution:	(Laboratory or PO)
Date of Contact:	Call or Visit (Circle One
Summary of Issues & Resolutions:	
Document the issue(s), resolution(s), an	nd action deadlines, if any.
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Signature	Date
Region	

1. Lab DPO Copy 2. SMO Copy 3. Lab Copy 4. Regional DPO Copy 5. Project Officer Copy 8/11/86

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SAMPLE MANAGEMENT OFFICE

Follow-Up Notice of Request for Corrective Action

SAS No				
Laboratory, Name:				
Date of Verbal Notice:	<u> </u>	Corrective Ac	tion Start Date:/	/
Sample No(s)/Parameters:	<u> </u>			
Direction Given:			·····	
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			······································	
Direct Inquiries to:			703/557-2490	
White: Lab Yello	w: SAS File	Pink: Region	Gold: SMO Invoice Log	
				7/22/86

.

APPENDIX D

.

AUXILIARY SUPPORT SERVICES DOCUMENTATION

· · · · ·

CLP SAMPLE BOTTLE REPOSITORY SUPERFUND DELIVERY REQUEST

-

Date of Request:	Type of Request: Routine	
	Routine	
	Fast Turnaround Emergency	(date/time request called in)
		(date/time request called in)
FROM (Name):		-
Affiliation:		<u> </u>
Telephone:		
AR Signature:	• <u>•</u> ••••••••••••••••••••••••••••••••••	-

Ship the following items for arrival by:	(date)
(If applicable) Ship to arrive no earlier than:	(date)

Item	Description	No. of Items Per Case	No. of Cases Requested
A	80-ounce amber glass bottle	6	
B	40-mL glass vial	72	
С	I-L polyethylene bottle	42	
D	120-mL wide-mouth glass vial	72	
E	16-oz wide-mouth glass jar	48	
F	8-oz wide-mouth glass jar	96	
G	4-oz wide-mouth glass jar	. 120	
н	l-L amber glass bottle	24	
J	32-oz wide-mouth glass jar	36	
к	4-L amber glass bottles	4	
	SHIP TO:		
	(provide		
	street address)		<u> </u>
	Attention:		
DIS	TRIBUTION: White - Repository Copy Pink - SMO Copy	Yellow - Request	tor File Copy

	SAMPLE B SUPERFU	OTTLE REPOSITOR Y IND PACKING LIST
REPO	DSITORY	DELIVERY REQUEST NO.
		Request Date:
	:	Type of Request: R FTA E Required Delivery Date:
DEST	INATION (from Delivery Request)	The materials listed below have been
Name:Address:		Data Shianada
		Mode of Shipment:
		Shipment ID No.
Telep	phone No:	
Туре	of Shipment: Complete Pa	rtial Partial/Completes Request
Item No.	No of. Cases Description Shipped	Lot QC Clearance Number(s) Number(s)
A	80-oz glass	
в	40-mL glass	
С	1-L poly	
D	120-mL glass	
E	16-oz glass	
F	8-oz glass	
G	4-oz glass	
н	1-L glass	
J	32-oz glass	
К	4-L glass	
AUT	HORIZED REQUESTOR USE ONLY	•
Sign the p	below and forward the yellow copy ink copy for your file.	y to SMO within 7 days of shipment receipt.
The a	above request was received by the o	designee, inspected, and accepted.
Date	of Receipt:	Requestor Signature:
Send	yellow PL copy to: USEPA P.O. Bo Alexand	Sample Management Office (SMO) ox 818 Iria, Virginia 22313
Distr	ibution: White - Shipment/Designee Copy	Yellow - Requestor Copy for Return to S Pink - Requestor File Copy

U.S ENVIRONMENTAL PROTECTION AGENCY HAZARDOUS WASTE INVESTIGATION SAMPLE MANAGEMENT OFFICE

DATE: 02/24/86

**** REGIONAL SAMPLE LIST **** SAMPLES RECEIVED FROM REGION I FROM 08/01/85 TO 08/31/85 LABORATORY: CONTRACT :

PROGRAM : GC SCREEN, GC/MS ANALYSIS

DAY LATE/EARLY CODES: N/A : NOT APPLICABLE N/R: NO CHRONICLE RECEIVED N/D: NOT DUE

CASE NO/ REGION/ TYPE1 INV TYPE2 INV	SAMPLE NO	LAB RECEIVED	SAMPLE WEIGHT	C 	COMP CODE	SAMPLE Type	DATA DUE	DATA Received	DAYS Data	LATE/E EXT	ARLY VOA
**************************************	AB969	08/01/85	0.80	EB EF	э вр	OLN	08/31/85	08/31/85	0	-2	 N/A
00002186	AB970	08/01/85	0.80	EB EF	В Р	OLW	08/31/85	08/31/85	0	-2	N/A
00002186	AB971	08/01/85	0.80	EB EF	, B b	OLW	08/31/85	08/31/85	0	-2	N/A
00002186	AB972	08/01/85	0.80	EB EF	, B b	OLW	08/31/85	08/31/85	0	-2	H/A
00002186	AB973	08/01/85	. 0.80	EB EF	вр	OLW	08/31/85	08/31/85	0	-2	N/A
00002186	AB974	08/01/85	0.80	EB EF	• B P	OLH	08/31/85	08/31/85	0	-2	N/A
00002186	AB975	08/01/85	0.80	EB EF	• B P	OLH	08/31/85	08/31/85	0	-2	N/A
00002261	AB975-X	08/01/85	0.40	EB	В	OLH	09/14/85	09/14/85	0	0	N/A
00002186	AB976	08/01/85	0.80	EB EF	• B P	OLN	08/31/85	08/31/85	0	-2	N/A
00002186	AB976-M	08/01/85	0.80	EB EF	• B P	OLW	08/31/85	08/31/85	0	-2	N/A
00002186	AB976-D	08/01/85	0.80	EB EF	• B P	OLH	08/31/85	08/31/85	0	-2	N/A
00002186	AB977	08/01/85	0.80	EB EP	9 B P	DLW	08/31/85	08/31/85	0	-2	N/A
00002186	AB978	08/01/85	0.80	EB EP	• B P	OLH	08/31/85	08/31/85	0	-2	N/A
**************************************	AC584	08/14/85	1.00	FU	NL	OLS	09/13/85	09/25/85	12	-2	-1
00002326	AC584-M	08/14/85	1.00	FU	JLL	OLS	09/13/85	09/25/85	12	-2	-1
00002326	AC584-D	08/14/85	1.00	FU	πL	OLS	09/13/85	09/25/85	12	-2	-1

PAGE:

CASE FILE PURGE MATERIALS

INCLUDE, BUT ARE NOT LIMITED TO:

SAMPLE TAGS

CHAIN-OF-CUSTODY RECORDS

COPIES OF SAMPLE TRACKING RECORDS

ANALYSTS' LOGBOOK PAGES

INSTRUMENT LOGBOOK PAGES (INCLUDING INSTRUMENT CONDITIONS)

BENCH SHEETS

INSTRUMENT READOUT RECORDS

COMPUTER PRINTOUTS

CHROMATOGRAPHIC CHARTS

RAW DATA SUMMARIES

CORRESPONDENCE MEMOS

DOCUMENT INVENTORY

QA/QC COMPLIANCE REPORT

U.S. ENVIRONMENTAL PROTECTION AGENCY Contract Laboratory Program

MEMORAND	UM	
DATE:		, 1986
TO:		
	USEPA Region	
FROM:		
	SMO Data Review Team	
SUBJECT:	QA/QC Compliance Revie Organic Sample Data Pac	ew Summary for a Contract Laboratory Program ckage: Case No
As requ noted have be the following	uested, quality control and period and compared to general areas were evaluated	performance measures for the data packages to EPA standards for compliance. Measures for d:
Data Co Spectra Surroga Matrix S Calibra	ompleteness Matching Quality te Spikes Spikes/Duplicates tion	Blanks DFTPP and BFB Tuning Chromatography Holding Times Compound ID (HSL, TIC)
Any sta that the revie	tistical measures used to sup w may be reviewed by others	pport the following conclusions are attached so
<u>Summai</u>	ry of Results	I II III Volatiles <u>B/N/A Pesticide</u>
A	cceptable as Submitted	
A	cceptable with Comments	
U	nacceptable, Action Pending	
	naccentable	
U	nacceptable	
U Data Review	ed by:	Date:
U Data Review Review Autho	ed by:	Date: Date:
U Data Review Review Autho Signature:	ed by:	Date: Date:
U Data Reviewo Review Autho Signature: Area Code/Pl	ed by: prized By: hone No.:	Date: Date:
U Data Reviewe Review Autho Signature: Area Code/Pl FTS Line:	ed by: orized By: hone No.:	Date: Date: Date:
U Data Review Review Autho Signature: Area Code/Pl FTS Line:	ed by: orized By: hone No.:	Date: Date:

NARRATIVE

Case No.

Site Name			
Laboratory Name			
Introduction			
The laboratory samples collected or	's portion of this C	ase consisted of , 1985.	
The laborator samples.	y reported	problem(s) with	h the receipt of the
The laboratory co	reported	problems with the	analyses of

The evaluator has commented on the criteria specified under each fraction heading. All criteria have been assessed, but no discussion is given where the evaluator has determined that criteria were adequately performed or require no comment. Details relevant to these comments are given on the forms in Appendix A. Amounts of detected compounds are summarized in Appendix B.

Evaluation by Fraction

Holdir	ng Times		MS/MSD
GC/M	S Tuning		Compound ID (HSL, TIC)
Calibr	ration, Initial		Spectra Quality
Calibr	ation, Continuing		Standards
Blank			Chromatography
Surrog	gate Recovery		Data Completeness
Comments:			
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/Neutral/Acids	
_ Holding Times	MS/MSD
_ GC/MS Tuning	Compound ID (HSL, TIC)
_ Calibration, Initial	Standards
_ Calibration, Continuing	Chromatography
Blank	Data Completeness
_ Surrogate Recovery	
ments:	
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III. Pesticides/PCBs

Holding Times	Calibration Linearity
Instrument Performance	Calibration, Continuing
DDT RT/12 Minute?	Blank
Retention Time Window	Surrogate Recovery
Analytical Sequence	MS/MSD
DDT/Endrin Degradation	Compound ID (HSL, TIC)
RT Check for DBC	Standards
Resolution Check	Chromatography
	Data Completeness
Comments.	
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REVIEW MATRIX APPENDIX B - VOA COMPOUNDS

Page ____ of ____

Case No.

Laboratory Name

	Samples					Blanks				
Compounds				Ţ	1	[
	+	_	<u> </u>	<u>↓</u>	+	<u> </u>	<u> </u>			
Chioromethane			ļ	ļ		ļ				
Bromomethane	·		<u> </u>				ļ	 	ļ	L
Vinyl Chloride			ļ		L	ļ	<u> </u>			
Chloroethane						ļ	L			
Methylene Chloride				ļ	ļ				L	
Acetone				<u> </u>						
Carbon Disulfide										
1,1-Dichloroethene										
1,1-Dichloroethane										
Trans-1,2-Dichloroethene										
Chloroform										
1,2-Dichloroethane										
2-Butanone										
1,1,1-Trichloroethane										
Carbon Tetrachloride		1.]	ļ				
Vinyl Acetate					1					
Bromodichloromethane										
1,2-Dichloropropane										
Trans-1,3-Dichloropropene										
Trichloroethene			1							
Dibromochloromethane										
1,1,2-Trichloroethane						[
Benzene										
cis-1.3-Dichloropropene	1 1									
2-Chloroethylvinylether	1		1							
Bromoform										
4-Methyl-2-Pentanone	1 1		1							
2-Hexanone	1									
Tetrachloroethene	T									
1,1,2,2-Tetrachloroethane	1									
Toluene	1				1					
Chiorobenzene	1				1					
Ethylbenzene										
Styrene	1				t					
Total Xylenes	1				1					
	†			Sample/	Blank Arec	ciation				

REVIEW MATRIX APPENDIX B - PESTICIDE/PCB COMPOUNDS

.

Page ____ of ____

Case No.	 Laboratory Name									
	 Samples						Blanks			
Compounds Alpha-BHC										
				[I		
Beta-BHC		ļ	L	l	ļ	ļ	<u> </u>	ļ		
Delta-BHC	 	ļ		L	ļ		ļ	L		
Gamma-BHC			ļ	L			<u> </u>	ļ		
Heptachlor				ļ	L	l		<u> </u>		
Aldrin								ļ		
Heptachlor Epoxide		1	[<u> </u>	[[1	
Endosulfan I										
Dieldrin							<u> </u>			
4,4-DDE										
Endrin								· ·	<u> </u>	
Endosulfan II					i					
4,4-DDD								L		
Endosulfan Sulfate								L		
4,4-DDT									1	
Methoxychlor		<u> </u>	<u> </u>							
Endrin Ketone										
Chlordane							<u> </u>	ļ		
Toxaphene										
Aroclor-1016										
Aroclor-1221										
Aroclor-1232		i					•			
Aroclor-1242			<u> </u>				<u> </u>		<u> </u>	
Aroclor-1248										
Aroclor-1254										
Aroclor-1260										
	Sample/Blank Association									
REVIEW MATRIX

APPENDIX B - BNA COMPOUNDS

Case No.

Laboratory Name

				Samp	les					Blanks	
Compounds											
2											
Phenol							1				
bis(2-Chloroethyl)Ether											
2-Chiorophenol						1					
1.3-Dichlorobenzene											
1.4-Dichlorphenzene				1		1					L
Repard Alcohol						<u> </u>					
1.3. Disblasharman				ł	<u> </u>						┝╍╧╍╍╼╾┥
1,2-Dichlorobenzene											
2-Methylphenol				L	ļ	L					
bis(2-Chloroisopropyl)Ether				[I	ļ					
4-Methylphenol						ļ					
N-Nitroso-Di-n-Propylamine				<u> </u>	I						
Hexachioroethane											
Nitrobenzene											
Isophorone											
2-Nitrophenol						1					
2 A Dimethylphenol											
Receipt A sid						<u> </u>					
penzoic Acia						<u> </u>					
bis(2-Chloroethoxy)Methane						<u> </u>					
2,4-Dichlorophenol						L	· · ·				d
1,2,4-Trichlorobenzene						L					
Naphthalene				· · · · · · · · · · · · · · · · · · ·		I					
4-Chloroaniline											
Hexachlorobutadiene											
4-Chloro-3-Methylphenol											
2-Methylnaphthalene						1					
Hexachiorocyclopentadiene						f					
2 4 6 Trichlorophenol	-										
2 4 5 Trichlorophenol											
				····					<u> </u>		
2-Chloronaphthalene											
Z-Nitroaniline									ļ		
Dimethyl Phthalate											
Acenaphthylene						·					
3-Nitroaniline											
Acenaphthene											
2,4-Dinitrophenol		_									
4-Nitrophenol											
Dibenzofuran				•							
7.4-Dinitrotoluene											
2 6 Dinistrataluana											
Dischula haba laca											
Dieuryiphutalate											
4-Chiorophenyl-phenylether											
Fluorene							ļ				
4-Nitroaniline											
4,6-Dinitro-2-Methylphenol											
N-Nitrosodiphenylamine(1)											
4-Bromophenyl-phenylether											
Hexachlorobenzene											
Pentachlorophenol											
Phenanthrene	-										
Anthracene											[
Di-n-Burylohthalate											
Elverenthene											
Pyrane											
Pyrelie But it an anti-bat - tata											_
Butylbenzylphthalate											
3,3'-Dichlorobenzidene											
Benzo(a)Anthracene								L			
bis(2-Ethylhexyl)Phthalate			·								
Chrysene											
Di-n-Octyl Phthalate											•
Benzo(b)Fluoranthene											
Benzo(k)Fluoranthene											
Benzo(a)Purane											{
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Dibenzia-n/Anthracene	<u> </u>	L									
Benzo(g,h,i)Perylene							L		<u> </u>		
					Sample/	Blank Asso	ciation			-	Τ
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REVIEW MATRIX

APPENDIX B - TIC COMPOUNDS

Page ____ of ____

Case No.

Laboratory Name

CONTRACT COMPLIANCE SCREENING

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LAB NAME	:												PREPARED BY:
	 c.m	I DA	TE		ASSOC-	ASSOC-	CALIB	RATION	і на 1 на	LTS	NO	SURR.	
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CONTRACT COMPLIANCE SCREENING FOR ORGANIC VOA PAGE: ____ OF ____

CASE:						SAMPLES	;						DATI	E:LOG NO
LAB NAMI	E:			~									PREI	PARED BY:
 Sampi F			DATE		ANALY-	ASSOC-	ASSOC-	CALIB	RATION	н	ITS			 DDABIEME
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CONTRACT COMPLIANCE SCREENING FOR ORGANIC B/N/A PAGE: ____OF ____

CONTRACT COMPLIANCE SCREENING FOR ORGANIC PESTICIDES/PCBS

PAGE: ____ OF ____

CASE:

LAB NAME:

SAMPLES:

DATE:_____LOG NO.____

PREPARED BY:

•••			DA	T E	COLUMN	1		·	COLUMN	2				
	SAMPLE				ANALYSIS	ASSO- Ciate	CALIB	RATION	ANALYSIS Date	ASSO- CIATE	CALIB	RATION	NO. Of Hits	PROBLEMS
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CASE:

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LAB NAME:

SAMPLES: **REGION:**

DATE: _____ LOG NO.____

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N P L E		H O L D	T U N E	B L K S	I N I T	C O N T		S U R R	M S / D	C O M P	H O L D	H O L D		T U N E	8 L K S	I N I T	C O N T		S I U I R I R I	M S / D	C O M P	H O L D			B L K S	D D T 		R T 	A N A L	D E G) 3 2 1	M S / D	1 N 1 T		C 0 N T	C O M P
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APPENDIX E

REFERENCES

NOTE: The references in this Appendix are supplied for general information purposes and do not necessarily represent methods or procedures utilized in the CLP.

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