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# REMEDIAL INVESTIGATION/FEASIBILITY STUDY
# QUALITY ASSURANCE PROJECT PLAN ADDENDUM
# LENZ OIL SERVICE, INC.
# LEMONT, ILLINOIS

REVISION: 1

SUBMITTED BY:

LENZ OIL SETTLING RESPONDENTS

DECEMBER 30, 1991

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PROJECT NO. 0252

**RI/FS QUALITY ASSURANCE PROJECT PLAN ADDENDUM**
**LENZ OIL SITE, LEMONT, ILLINOIS**

**December 30, 1991**

**Revision: 1**

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## 1.0 PROJECT DESCRIPTION

This Quality Assurance Project Plan (QAPP) Addendum has been prepared by ERM-North Central, Inc. (ERM-North Central) to supplement the approved November 12, 1990 QAPP for the Remedial Investigation/Feasibility Study (RI/FS) prepared for the Lenz Oil, Inc. Site, located in Lemont, Illinois. This Addendum is being submitted to modify the approved QAPP for the additional RI/FS Phase II activities as directed by the U.S. Environmental Protection Agency (USEPA). The project description provided in the approved QAPP, dated November 12, 1990, is applicable as presented except that the following additional activities will be performed during Phase II:

- A neighboring residential well will be sampled and analyzed,
- The nonaqueous phase liquid (NAPL) will be sampled and analyzed,
- A shallow soil boring and possibly a shallow monitoring well will be installed,
- At least one residential well will be abandoned,
- A second round of ground water samples from the monitoring wells will be collected and analyzed,

- The surface water in the drainage ditch northwest of the site will be resampled and analyzed for the TAL metals to replace rejected metals data from the Phase I samples,

- Four of the Phase I soil borings will be resampled and analyzed.

- Background soil samples will be collected and analyzed, and

- Additional on-site soil samples will be collected and analyzed.

The Phase II investigative tasks are intended to further characterize the nature and extent of soil, surface water, and ground water contamination at the site. The scope of this QAPP Addendum applies only to the Phase II tasks described in the Phase II Work Plan (Parts A and B), dated October 7, 1991. Therefore, only the necessary modifications and/or additions to the existing approved QAPP are presented.

### 1.1 Site Description and History

The site description and history are presented in the approved Technical Memorandum No. 1, dated May 1, 1991.

### 1.2 Target Compounds

The discussion of target compounds provided in the approved QAPP, dated November 12, 1990, is applicable as presented with the following exceptions:

The NAPL matrix will be analyzed for Target Compound List (TCL) organics, Target Analyte List (TAL) total inorganics, and Toxicity Characteristics Leaching Procedure (TCLP) organics and inorganics. Table 1-1 in the approved QAPP lists the TCL organic and TAL inorganic target parameters and their associated Contract Required Detection Limits for soil/sediment and water samples. Table 1-1 in this document lists the TCLP target parameters for the NAPL and the associated project-required detection limits.

The Round Two ground water samples from the monitoring wells will be analyzed for TCL organics and TAL inorganics. Tentatively Identified Compounds (TICs) will be included in the Contract Laboratory Program (CLP) Routine Analytical Service (RAS) Statement of Work (SOW) for volatile and semivolatile analyses. Ground water samples from the residential well will be analyzed for TCL organics and TAL inorganics using the lower detection limits listed on Table 1-2. The monitoring well samples will be analyzed for both total metals and dissolved metals; however, the residential well samples will be analyzed for total metals, but not dissolved metals.

Samples analyzed for TCL organics will be analyzed in accordance with the CLP SOW OLM01.0 including the revisions in OLM01.1, or the most current SOW for TCL organic analysis. Samples analyzed for TAL inorganics will be analyzed in accordance with CLP SOW ILM01.0 or the most current SOW for TAL inorganics analysis.

Samples analyzed for TCLP organics will be analyzed in accordance with SW-846 Methods: 8240, 8270, 8080, and 8150. Samples analyzed for TCLP inorganics

will be analyzed in accordance with CLP SOW ILM01.0 or the most current SOW.

## 1.3 Project Objectives

The project objectives provided in the approved QAPP dated November 12, 1990 are applicable as presented except for the information added to the following subsections.

### 1.3.1 Specific Objectives

The general objectives provided in the approved QAPP, dated November 12, 1990, are applicable as presented; however, the following objectives are specific to the Phase II investigation.

- Reevaluating the stratigraphy in the vicinity of well cluster G104 to further define ground water flow.

- Collecting sufficient data to characterize the physical and chemical nature of the NAPL.

- Collecting organic ground water data of sufficient quantity and quality to characterize the nature and extent of any site-derived inorganic ground water contamination.

- Collecting inorganic ground water data of sufficient quantity and quality to characterize seasonal variations in the extent of any site-derived organic ground water contamination.

- Collecting ground water analytical data of sufficient quality to evaluate the potential risk to human health posed by drinking ground water from a neighboring residential well.

- Minimizing the spread of ground water contamination by closing and abandoning contaminated private wells.

- Collecting organic and inorganic soil data of sufficient quantity and quality to characterize the nature and extent of site-derived soil contamination.

- Collecting inorganic surface water data of sufficient quantity and quality to characterize the nature of any site-derived inorganic contamination in the drainage ditch.

### 1.3.2 Intended Data Uses

The discussion of intended data uses provided in the approved QAPP, dated November 12, 1990, is applicable as presented with the following additions:

- Specific gravity and viscosity - used to determine the physical character of the NAPL to establish remedial requirements.

- TCLP of the NAPL - used to determine the chemical nature of the NAPL to identify remediation alternatives.

- Laboratory analysis of residential well samples - used to verify the extent of ground water contamination northeast of the site and to evaluate the potential risk to human health caused by drinking ground water from a neighboring residential well.

### 1.3.3 Data Quality Objectives

The Data Quality Objectives provided in the approved QAPP, dated November 12, 1990, are applicable as presented with the following additions:

- TCLP extraction of NAPL - Level III.

- Laboratory analysis of TCLP extract - Level IV.

- Specific gravity and viscosity of NAPL - Level III.

- Laboratory analysis of TCL organics and TAL inorganics in ground water samples from a residential well - Level V.

## 1.4 Sample Network and Rationale

The Phase II sample network and rationale are provided in the Phase II Work Plan (Parts A and B), dated October 7, 1991. Specific sampling locations for each of the sample matrices are shown on figures in the Phase II Work Plan. A summary of the Phase II Sampling and Analysis Program is provided in Table 1-3.

## 1.5 Project Schedule

The project schedule provided in the approved QAPP, dated November 12, 1990, is not applicable and should be replaced with the project schedule provided in the Phase II Work Plan-Part A, dated October 7, 1991. Work progress reports will be prepared and submitted to the USEPA during the course of the project according to the schedule specified in the Administrative Consent Order.

## 2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The discussion of the project organization and responsibility provided in the approved QAPP, dated November 12, 1990, is applicable as presented with the following additions to the appropriate subsections.

### 2.1 Management

Section 2.1 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following personnel changes and additions:

- Dave Dollins, the IEPA Project Manager, has been replaced by Tracy Fitzgerald of IEPA.

- Lisa Snow, the Laboratory Quality Assurance (QA) Director for Skinner & Sherman, Inc. will coordinate and oversee the laboratory inorganic analyses of monitoring well and soil samples.

- Gary Torf, the Laboratory QA Director for Rocky Mountain Analytical Laboratory (RMAL) will coordinate and oversee the laboratory organic analyses of monitoring well samples and all laboratory analyses of NAPL.~~and residential well samples.~~

- Robert Whitehead, the Laboratory QA Director for CompuChem Laboratories, Inc. (CompuChem) will coordinate and oversee the

laboratory organic and inorganic analysis of residential well samples.

## 2.2 Field Activity

Section 2.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following additions to the lists of the ERM-North Central and IEPA subcontractors:

**ERM-North Central Subcontractors**

| Subcontractor | Service |
|---|---|
| Skinner & Sherman, Inc. | Analytical services for CLP RAS inorganics in monitoring well samples. |
| Rocky Mountain Analytical Laboratory | Analytical services for CLP RAS organics for all matrices; ~~non CLP organics and inorganics of ground water;~~ and TCLP, viscosity, and specific gravity of the NAPL. |

| | |
|---|---|
| ARDL, Inc. | Analytical services for CLP RAS volatile organics in monitoring well samples. |
| CompuChem Laboratories, Inc. | Analytical services for non-CLP organic and inorganic analysis of groundwater (low concentration analysis). |

**IEPA Subcontractors**

| Subcontractor | Service |
|---|---|
| Skinner & Sherman, Inc. | Analytical services for CLP RAS inorganics in soils. |

### 2.3 Laboratory Analysis

Section 2.3 of the approved QAPP, dated November 12, 1990, is not applicable to the Phase II work and should be replaced with the following:

> RMAL, located in Arvada, Colorado, will perform the ~~viscosity and specific gravity analyses of the NAPL in accordance with ASTM methods and the~~ TCLP analysis of the NAPL in accordance with their Standard Operating Procedures

(SOPs) (Appendix A). RMAL will subcontract the viscosity and specific gravity analysis of the NAPL to Hauser Laboratory in Boulder, Colorado who will perform the analysis in accordance to ASTM Methods (Appendix A). ~~RMAL~~ CompuChem Laboratories, Inc. will also perform the TCL organic and TAL inorganic analyses of the residential well sample in accordance with the CLP SOW OCL01.0 for organics and or the most current revision. The inorganics portion of the residential well sample will be analyzed by CompuChem's subcontractor, Warzyn in Madison, Wisconsin, using the CLP SOW ILC01.0 or the most current revision. ~~their SOP (Appendix A).~~ All TCL organic analyses of ground water samples from the monitoring wells will be completed in accordance with the CLP RAS SOW OLM01.0, with ARDL, Inc. in Mount Vernon, Illinois conducting the TCL volatile organic analyses and RMAL conducting the TCL semivolatile organic and pesticide/polychlorinated biphenyl (PCB) analyses. The TCL organic and TAL inorganic analysis of the NAPL will be performed by RMAL. Skinner and Sherman, Inc., in Waltham, Massachusetts will perform the TAL inorganic analyses of the monitoring well samples. For the soil samples, ARDL will analyze for TCL organics and Skinner and Sherman will analyze for TAL inorganics. The surface water samples will be analyzed by ARDL for those TAL inorganics rejected during the Phase I investigation.

## 2.4 Quality Assurance Organization

Section 2.4 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

Internal Laboratory Audits: RMAL, Skinner & Sherman, and ARDL QA Officers

## 2.5 Performance and System Audits

Section 2.5 of the approved QAPP, dated November 12, 1990, is applicable as presented.

# 3.0 QUALITY ASSURANCE OBJECTIVES

Section 3.0 provided in the approved QAPP, dated November 12, 1990, is applicable as presented.

## 3.1 Level of Quality Control Effort

Section 3.1 of the approved QAPP, dated November 12, 1990, is applicable as presented.

## 3.2 Accuracy, Precision, and Sensitivity of Analyses

Section 3.2 of the approved QAPP, dated November 12, 1990, is applicable as presented except for the additional information that is included in the following subsections.

### 3.2.1 Field Instruments

Section 3.2.1 of the approved QAPP, dated November 12, 1990, is applicable as presented.

### 3.2.2 Laboratory Instruments

Section 3.2.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

> The Phase II activities will use the CLP SOW OLM01.0 for organics analysis and the CLP SOW ILM01.0 for inorganics analysis or the most current SOWs in effect at the time sampling begins. The accuracy and precision requirements for

the TCL and TAL parameters are specified in the CLP SOWs. The accuracy and precision of the low concentration organic and inorganic parameters are specified in the CLP SOW's OLC01.0 and ILC01.0. A summary of the project-specific accuracy and precision requirements for all Phase II laboratory analyses is presented in Table 3-1.

### 3.3 Data Completeness, Representativeness, and Comparability

Section 3.3 of the approved QAPP, dated November 12, 1990, is applicable as presented.

### 3.4 Documentation

Section 3.4 of the approved QAPP, dated November 12, 1990, is applicable as presented.

### 3.5 Quality Control Requirements

Section 3.5 of the approved QAPP, dated November 12, 1990, is applicable as presented.

# 4.0 SAMPLING PROCEDURES

The sampling and analysis procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented except for the additions and clarifications presented in the following subsections.

## 4.1 Sample Locations and Frequency

The discussion of sample locations and frequency presented in the approved Sampling and Analysis Plan, dated November 12, 1990, is applicable as presented with the following changes:

Tables 3-1 and 3-2 in the approved Sampling and Analysis Plan are superseded by Table 1-3 in the QAPP Addendum.

### 4.1.1 Monitoring Well Sampling

The Phase II monitoring well sample locations and the rationale for selecting these locations are presented in Sections 2.3 and 4.3 of the Phase II Work Plan-Part A, dated October 7, 1991.

### 4.1.2 On-Site Soil Sampling

The Phase II on-site soil sampling locations and the rationale for selecting these locations is presented in Section 3.2 of the Phase II Work Plan-Part B, dated October 7, 1991.

### 4.1.3 Background Soil Sampling

The Phase II background soil sampling locations and the rationale for selecting these locations is presented in the Phase II Work Plan-Part B, dated October 7, 1991.

### 4.1.4 Surface Water Sampling

The surface water sample locations and the rationale for selecting these locations presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented.

### 4.1.5 NAPL Sampling

The NAPL sample location and the rationale for selecting this location is presented in Sections 2.3 and 4.2 of the Phase II Work Plan-Part A.

### 4.1.6 Residential Well Sampling

The residential well sample location and the rationale for selecting this location is presented in Sections 2.4 and 4.4 of the Phase II Work Plan-Part A.

## 4.2 Sample Designation

The sample designation procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented except for the modifications presented in Section 3.3.1 of the Phase II Work Plan-Part B.

## 4.3 Sampling Equipment and Procedures

The discussion of sampling equipment and procedures presented in the approved Sampling and Analysis Plan is applicable as presented with the additions to the following subsections.

### 4.3.1 Monitoring Network Design and Well Installation

The monitoring network design and well installation procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented except for the changes outlined in Section 4.1 of the Phase II Work Plan-Part A, dated October 7, 1991.

### 4.3.2 Ground Water Sampling

The ground water sampling procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable are presented except for the changes presented in Section 4.3 of the Phase II Work Plan-Part A, dated October 7, 1991.

### 4.3.3 On-Site Soil Sampling

The on-site soil sampling procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented except as modified by Section 3.2.2 and 3.2.3 of the Phase II Work Plan-Part B, dated October 7, 1991.

### 4.3.4 Background Soil Sampling

The background soil sampling procedures are presented in Section 3.2.3 of the Phase II Work Plan-Part B, dated October 7, 1991.

### 4.3.5 Surface Water Sampling

The surface water sampling procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented.

### 4.3.6 NAPL Sampling

The NAPL sampling procedures are presented in Section 4.2 of the Phase II Work Plan-Part A, dated October 7, 1991.

### 4.3.7 Residential Well Sampling

The residential well sampling procedures are presented in Section 4.4 of the Phase II Work Plan-Part A, dated October 7, 1991.

## 4.4 Sampling Handling and Analysis

The sampling handling and analysis procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented.

### 4.4.1 Sample Preparation, Handling, and Shipment

The sample preparation, handling, and shipment procedures presented in the approved Sampling and Analysis Plan, dated November 12, 1990, are applicable as presented except as modified in Sections 4.1 to 4.5 of the Phase II Work Plan-Part A and Sections 3.3.3 to 3.3.5 of the Phase II Work Plan-Part B, dated October 7, 1991. Table 4-1 summarizes the sample preservation, handling, and bottle requirements.

# 5.0 SAMPLE CUSTODY

The sample custody procedures provided in the approved QAPP, dated November 12, 1990, are applicable as presented except as noted in the following subsections.

## 5.1 Field Custody Procedures

Section 5.1 and its subsections provided in the approved QAPP, dated November 12, 1990, are applicable as presented.

## 5.2 Laboratory Custody Procedures

Section 5.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

> The chain-of-custody procedures to be followed by Skinner & Sherman, ~~and~~ RMAL and CompuChem are presented in Appendices B, C, and D, respectively.

## 5.3 Final Evidence File

Section 5.3 of the approved QAPP, dated November 12, 1990, is applicable as presented.

# 6.0 CALIBRATION PROCEDURES

The calibration procedures provided in the approved QAPP, dated November 12, 1990, are applicable as presented with the exception of the following additions to specific subsections.

## 6.1 Field Instruments

Section 6.1 of the approved QAPP, dated November 12, 1990, is applicable as presented.

## 6.2 Laboratory Equipment

Section 6.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

> RMAL, CompuChem, and Skinner & Sherman will be responsible for the calibration and maintenance of the analytical equipment used for their laboratory analyses. RMAL and Skinner & Sherman will follow exactly the procedures outlined in the CLP SOW OLM01.0 for organics and the CLP SOW ILM01.0 for inorganics or the most current versions at the date sampling begins. The SOPs that RMAL will be using for TCLP ~~and residential ground water sampling~~ are included in Appendix A. CompuChem will follow exactly the procedures outlined in the CLP SOW OLC01.0 for organics and ILC01.0 for inorganics or the most current versions at the date sampling begins. Discussions of the calibration frequency and methodology for non-CLP analyses (e.g., specific gravity, viscosity, TCLP, and residential well sample analyses) are presented in Appendix A. ~~Viscosity analysis will follow the American Standards for Testing Materials (ASTM) method DZ669 for calibration frequency and methodology.~~

## 7.0 ANALYTICAL PROCEDURES

The analytical procedures provided in the approved QAPP, dated November 12, 1990, are applicable as presented except for the following additions:

The CLP TCL and TAL analyses will be conducted by RMAL, ARDL, and Skinner & Sherman using methods specified in the RAS SOW OLM01.0 ~~and the RAS SOW OLM01.0~~ for organics and the RAS SOW ILM01.0 for inorganics, or the most current versions that are used by CLP laboratories at the time sampling begins.

As stated in RMAL's SOP for TCLP extraction and analysis (refer to Appendix A), the TCLP extracts will be analyzed using SW846 Methods (8420, 8270, 8080, and 8150)~~ the exact methods specified in the RAS SOW OLM01.0~~ for organics and the RAS SOW ILM01.0 for inorganics, or the most current version at the time sampling begins. The RMAL SOPs for analyzing specific gravity, and viscosity~~ TCL organics and TAL inorganics at lower detection limits~~ are presented in Appendix A. ~~Viscosity will be analyzed using the ASTM DZ669 method.~~

In conducting their residential well analysis, CompuChem will follow the analytical procedures and methods specified in the CLP SOW OLC01.0 for organics and CLP SOW ILC01.0 for inorganics, or the most current versions that are used by CLP Laboratories at the time sampling begins.

# 8.0 DATA REDUCTION, VALIDATION, AND REPORTING

The data reduction, validation, and reporting discussion provided in the approved QAPP, dated November 12, 1990, is applicable as presented except for the following:

> RMAL, CompuChem, and Skinner & Sherman will likewise be responsible for reducing their own analytical data (RMAL's SOP for data reduction, data validation, and reporting are presented in Appendix A). Environmental Standards, Inc. will validate the Round Two ground water, NAPL, and residential well data.

## 8.1 Documentation

Section 8.1 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

> Laboratory documentation procedures utilized by Skinner & Sherman, RMAL and CompuChem are provided in Appendices B, C, and D respectively. Non-CLP procedures are provided in Appendix A.

## 8.2 Data Reduction

Section 8.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

Analytical data reduction will be carried out in-house by RMAL, CompuChem, ARDL, and Skinner & Sherman on their respective data sets. Reduction of all TCL-organic and TAL-inorganic data will be completed by RMAL, ARDL, and Skinner & Sherman in exact accordance with the CLP SOW OLM01.0 and CLP SOW OLM01.1 for organics and the CLP SOW ILM01.0 for inorganics, or the most recent SOW versions. Because the TCLP extract will be analyzed in strict accordance with the SW846 Methods ~~CLP SOW OLM01.0~~ for organics and SOW ILM01.0 for inorganics, the TCLP data will be reduced by RMAL using the applicable procedures outlined in the RMAL SOPs and CLP SOWs. Reduction of the TCL organic, TAL inorganic, and TCLP data ensures that the actual quantities reported are accurate and appropriately qualified. The reported quantities will be as directed, qualified or not, by the laboratory. Compounds detected in blanks will not be subtracted from the analytical results of investigative samples and will be reported separately. Data reduction procedures used by RMAL for the non-CLP analyses are presented in Appendix A.

## 8.3 Data Validation

The data validation description provided in the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

RMAL, ARDL, CompuChem and Skinner & Sherman will perform laboratory analyses as part of the Phase II RI. The TCLP extract data as well as the TCL organic and TAL inorganic data with low detection limits will be validated by Environmental Standards, Inc. using the USEPA documents "Laboratory Data Functional Guidelines for Evaluation of Organic Analyses" (2/88) and

"Laboratory Data Functional Guidelines for Evaluation of Inorganics Analyses" (7/88) or the most current versions at the time sampling begins. The viscosity and specific gravity results will not be validated.

## 8.4 Reporting

The reporting requirements provided in the approved QAPP, dated November 12, 1990, are applicable as presented except as follows:

The CLP SOW OLM01.0 for organics and CLP SOW ILM01.0 for inorganics or the most current SOWs available at the time sampling begins will be followed as the guides for reporting requirements.

Non CLP analyses (TCLP sample) will be reported using RMAL's standard raw data deliverable (SOP is presented in Appendix A).

# 9.0 INTERNAL QUALITY CONTROL CHECKS

The internal quality control checks provided in the approved QAPP, dated November 12, 1990, are applicable as presented with the following additions to the applicable subsections.

## 9.1 Field Quality Control

Section 9.1 of the approved QAPP, dated November 12, 1990, is applicable as presented.

## 9.2 Laboratory Quality Control

Section 9.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

> RMAL, ARDL, and Skinner & Sherman will perform the internal quality control checks, laboratory performance, and system audits specified in the CLP RAS SOWs (OLM01.0 and OLM01.1) for organics and CLP RAS SOW ILM01.0 for inorganics. These Quality Control (QC) checks will apply to all TCL organic and TAL inorganic analyses. ~~including the TCLP extract.~~

For non-CLP analyses, the internal QC checks are summarized below and discussed in further detail in their respective SOPs (Appendix A).

| Analysis | QC Check Method |
|---|---|
| TCLP Extract Organics | Procedural and Solvent Blanks |
| | Surrogate Spikes |
| | Matrix Spike Duplicates |
| | Compound Identification Criteria |
| TCLP Extract Inorganics | Preparation Blanks |
| | Sample Spikes |
| | Sample Duplicates |
| | Laboratory Control Samples |
| | Detection Limits Report |
| TAL Inorganics (with low detection limits) | Same as TAL Inorganics |
| TCL Organics (with low detection limits) | Same as TCL Organics |

# 10.0 PERFORMANCE AND SYSTEM AUDITS

The description of performance and system audits provided in the approved QAPP, dated November 12, 1990, is applicable as presented with the following additions to the appropriate subsections.

## 10.1 Internal Audits

The description of internal audits provided in the approved QAPP, dated November 12, 1990, is applicable as presented with a minor addition to Section 10.1.2.

### 10.1.1 Field Activities

Section 10.1.1 of the approved QAPP, dated November 12, 1990, is applicable as presented.

### 10.1.2 Laboratory Activities

Subsection 10.1.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

> The performance and system audits performed by Skinner & Sherman, CompuChem, and RMAL are summarized on Table 10-1 and documented in more detail in Appendices B, D, and C respectively.

## 10.2 External Audits

Section 10.2 of the approved QAPP, dated November 12, 1990, is applicable as presented.

# 11.0 PREVENTATIVE MAINTENANCE

The description of preventative maintenance provided in the approved QAPP, dated November 12, 1990, is applicable as presented with minor additions to Section 11.2.

## 11.1 Field Equipment

Section 11.1 of the approved QAPP, dated November 12, 1990, is applicable as presented.

## 11.2 Laboratory Equipment

Section 11.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition:

> The preventative maintenance procedures for their laboratory equipment will be the responsibility of Skinner & Sherman and RMAL. Skinner & Sherman's, Compuchem's and RMAL's preventative maintenance programs are documented in Appendices B, D, and C, respectively.

## 12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The specific routine procedures used to assess data precision, accuracy, and completeness provided in the approved QAPP, dated November 12, 1990, are applicable as presented.

# 13.0 CORRECTIVE ACTION

The description of corrective action provided in the approved QAPP, dated November 12, 1990, is applicable as presented with an addition to Subsection 13.2.

## 13.1 Immediate Corrective Action in the Field

Section 13.1 of the approved QAPP, dated November 12, 1990, is applicable as presented.

## 13.2 Immediate Corrective Action in the Laboratory

Section 13.2 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition.

> Skinner & Sherman's, CompuChem's, and RMAL's procedures for immediate corrective action in the laboratory are presented in Appendices B, D, and C, respectively.

## 13.3 Other Corrective Action

Section 13.3 of the approved QAPP, dated November 12, 1990, is applicable as presented.

## 14.0 QUALITY ASSURANCE REPORTS

Section 14.0 of the approved QAPP, dated November 12, 1990, is applicable as presented with the following addition.

> ~~RMAL's and~~ Skinner & Sherman's, and CompuChem's Quality Assurance Officer (QAOs) will provide monthly quality assurance reports to the ERM-North Central's Project Manager. RMAL's Program Administrator will provide monthly QA reports to ERM. These reports will be reviewed by RMAL's QAO.

TABLE 1-1

TCLP TARGET PARAMETERS AND
PROJECT-REQUIRED DETECTION LIMITS
FOR NAPL ANALYSIS
LENZ OIL SERIVCE, SERVICE INC. SITE
(Page 1 of 2)

| Parameter | Project Required Limits mg/L** |
|---|---|
| Arsenic | 5.0 |
| Barium | 100.0 |
| Benzene | 0.5 |
| Cadmium | 1.0 |
| Carbon tetrachloride | 0.5 |
| Chlordane | 0.03 |
| Chlorobezene | 100.0 |
| Chloroform | 6.0 |
| Chromium | 5.0 |
| ~~o-Cresol~~ 2-methyl phenol | 200.0 |
| ~~m-Cresol~~ 3-methyl phenol* | 200.0 |
| ~~p-Cresol~~ 4-methyl phenol* | 200.0 |
| ~~Cresol~~ | ~~200.0~~ |
| 2,4-D | 10.0 |
| 1,4-Dichlorobenzene | 7.5 |
| 1,2-Dichloroethane | 0.5 |
| 1,1-Dichloroethene | 0.7 |
| 2,4-Dinitrotoluene | 0.13 |
| Endrin | 0.02 |
| Heptachlor (and its epoxide ~~hydroxide~~) | 0.008 |
| Hexachlorobenzene | 0.13 |
| Hexachlorobutadiene | 0.5 |

TABLE 1-1

**TCLP TARGET PARAMETERS AND PROJECT-REQUIRED DETECTION LIMITS FOR NAPL ANALYSIS LENZ OIL SERIVCE, SERVICE INC. SITE**



| Parameter | Project-Required Limits mg/L** |
|---|---|
| Hexachloroethane | 3.0 |
| Lead | 5.0 |
| Lindane | 0.4 |
| Mercury | 0.2 |
| Methoxychlor | 10.0 |
| ~~Methyl ethyl ketone~~ 2-Butanone (MEK) | 200.0 |
| Nitrobenzene | 2.0 |
| Pentachlorophenol | 100.0 |
| Pyridine | 5.0 |
| Selenium | 1.0 |
| Silver | 5.0 |
| ~~Tetrachyloroethylene~~ Tetrachloroethene | 0.7 |
| Toxaphene | 0.5 |
| ~~Trichloroethylene~~ Trichloroethene | 0.5 |
| 2,4,5-Trichlorophenol | 400.0 |
| 2,4,6-Trichlorophenol | 2.0 |
| 2,4,5-TP (Silvex) | 1.0 |
| Vinyl chloride | 0.2 |

* 3-methyl phenol and 4-methyl phenol cannot be differentiated and will be reported as 3/4-methyl phenol.

** RMAL reporting limits will be lower unless matrix or high levels of target compounds require dilution of sample.

**TABLE 1-2**
**TCL AND TAL TARGET PARAMETERS AND**
**PROJECT-REQUIRED DETECTION LIMITS**
**FOR RESIDENTIAL WELL SAMPLES**


| Volatiles | CAS Number | Quantitation Limits Water ug/L |
|---|---|---|
| 1. Chloromethane | 74-87-3 | 1 |
| 2. Bromomethane | 74-83-9 | 1 |
| 3. Vinyl Chloride | 75-01-4 | 1 |
| 4. Chloroethane | 75-00-3 | 1 |
| 5. Methylene Chloride | 75-09-2 | 2 |
| | | |
| 6. Acetone | 67-64-1 | 5 |
| 7. Carbon Disulfide | 75-15-0 | 1 |
| 8. 1,1-Dichloroethene | 75-35-4 | 1 |
| 9. 1,1-Dichloroethane | 75-34-3 | 1 |
| 10. cis-1,2-Dichloroethene | 156-59-4 | 1 |
| | | |
| 11. trans-1,2-Dichloroethene | 156-60-5 | 1 |
| 12. Chloroform | 67-66-3 | 1 |
| 13. 1,2-Dichloroethane | 107-06-2 | 1 |
| 14. 2-Butanone | 78-93-3 | 5 |
| 15. Bromochloromethane | 74-97-5 | 1 |
| | | |
| 16. 1,1,1-Trichloroethane | 71-55-6 | 1 |
| 17. Carbon Tetrachloride | 56-23-5 | 1 |
| 18. Bromodichloromethane | 75-27-4 | 1 |
| 19. 1,2-Dichloropropane | 78-87-5 | 1 |
| 20. cis-1,3-Dichloropropene | 10061-01-5 | 1 |
| 21. Trichloroethene | 79-01-6 | 1 |
| 22. Dibromochloromethane | 124-48-1 | 1 |
| 23. 1,1,2-Trichloroethane | 79-00-2 | 1 |

TABLE 1-2
TCL AND TAL TARGET PARAMETERS AND
PROJECT-REQUIRED DETECTION LIMITS
FOR RESIDENTIAL WELL SAMPLES


| | | |
|---|---|---|
| 24. Benzene | 71-43-2 | 1 |
| 25. trans-1,3-Dichloropropene | 10061-02-6 | 1 |
| 26. Bromoform | 75-25-2 | 1 |
| 27. 4-Methyl-2-pentanone | 108-10-1 | 5 |
| 28. 2-Hexanone | 591-78-6 | 5 |
| 29. Tetrachloroethene | 127-18-4 | 1 |
| 30. 1,1,2,2-Tetrachloroethane | 79-34-5 | 1 |
| 31. 1,2-Dibromoethane | 106-93-4 | 1 |
| 32. Toluene | 108-88-03 | 1 |
| 33. Chlorobenzene | 108-90-7 | 1 |
| 34. Ethylbenzene | 100-41-4 | 1 |
| | | |
| 35. Styrene | 100-42-5 | 1 |
| 36. Xylenes (total) | 1330-20-7 | 1 |
| 37. 1,3-Dichlorobenzene | 541-73-1 | 1 |
| 38. 1,4-Dichlorobenzene | 106-46-7 | 1 |
| 39. 1,2-Dichlorobenzene | 95-50-1 | 1 |
| 40. 1,2-Dibromo-3-chloropropane | 96-12-8 | 1 |

TABLE 1-2
TCL AND TAL TARGET PARAMETERS AND
PROJECT-REQUIRED DETECTION LIMITS
FOR RESIDENTIAL WELL SAMPLES


| Semivolatiles | CAS Number | Quantitation Limits Water ug/L |
|---|---|---|
| 1. Phenol | 108-95-2 | 5 |
| 2. bix-(2-Chloroethyl)ether | 111-44-4 | 5 |
| 3. 2-Chlorophenol | 95-57-8 | 5 |
| 4. 2-Methylphenol | 95-48-7 | 5 |
| | | |
| 5. 2,2'-oxybis(1-Chloropropane) | 108-60-1 | 5 |
| 6. 4-Methylphenol | 106-44-5 | 5 |
| 7. N-Nitroso-di-n-propylamine | 621-64-7 | 5 |
| 8. Hexachloroethane | 67-72-1 | 5 |
| 9. Nitrobenzene | 98-95-3 | 5 |
| | | |
| 10. Isophorone | 79-59-1 | 5 |
| 11. 2-Nitrophenol | 88-75-5 | 5 |
| 12. 2,4-Dimethylphenol | 105-67-9 | 5 |
| 13. bix-(2-Chloroethoxy)methane | 11-91-1 | 5 |
| | | |
| 14. 2,4-Dichlorophenol | 120-83-2 | 5 |
| 15. 1,2,4-Trichlorobenzene | 120-82-1 | 5 |
| 16. Naphthalene | 91-20-3 | 5 |
| 17. 4-Chloroaniline | 106-47-8 | 5 |
| 18. Hexachlorobutadiene | 87-68-3 | 5 |
| | | |
| 19. 4-Chloro-3-methylphenol | 59-50-7 | 5 |
| 20. 2-Methylnaphthalene | 91-57-6 | 5 |
| 21. Hexachlorocyclopentadiene | 77-47-4 | 5 |
| 22. 2,4,6-Trichlorophenol | 88-06-2 | 5 |
| 23. 2,4,5-Trichlorophenol | 95-95-4 | 20 |

TABLE 1-2
TCL AND TAL TARGET PARAMETERS AND
PROJECT-REQUIRED DETECTION LIMITS
FOR RESIDENTIAL WELL SAMPLES


| | | |
|---|---|---|
| 24. 2-Chloronaphthalene | 91-58-7 | 5 |
| 25. 2-Nitroaniline | 88-74-4 | 20 |
| 26. Dimethylphthalate | 131-11-3 | 5 |
| 27. Acenaphthylene | 208-96-8 | 5 |
| 28. 2,6-Dinitrotoluene | 606-20-2 | 5 |
| | | |
| 29. 3-Nitroaniline | 99-09-2 | 20 |
| 30. Acenaphthene | 83-32-9 | 5 |
| 31. 2,4-Dinitrophenol | 51-28-5 | 20 |
| 32. 4-Nitrophenol | 100-02-7 | 20 |
| 33. Dibenzofuran | 132-64-9 | 5 |
| 34. 2,4-Dinitrotoluene | 121-14-2 | 5 |
| 35. Diethylphthalate | 84-66-2 | 5 |
| 36. 4-Chlorophenyl-phenylether | 7005-72-3 | 5 |
| 37. Fluorene | 86-73-7 | 5 |
| 38. 4-Nitroaniline | 100-01-6 | 20 |
| | | |
| 39. 4,6-Dinitro-2-methylphenol | 534-52-1 | 20 |
| 40. N-Nitrosodiphenylamine | 86-30-6 | 5 |
| 41. 4-Bromopheynl-phenylether | 101-55-3 | 5 |
| 42. Hezachlorobenzene | 118-74-1 | 5 |
| 43. Pentachlorophenol | 87-86-5 | 20 |
| | | |
| 44. Phenanthrene | 85-01-8 | 5 |
| 45. Anthracene | 120-12-7 | 5 |
| 46. Di-n-butylphthalate | 84-74-2 | 5 |
| 47. Fluoranthene | 206-44-0 | 5 |
| 48. Pyrene | 129-00-0 | 5 |

**TABLE 1-2**
**TCL AND TAL TARGET PARAMETERS AND**
**PROJECT-REQUIRED DETECTION LIMITS**
**FOR RESIDENTIAL WELL SAMPLES**


| | | |
|---|---|---|
| 49. Butylbenzylphthalate | 85-68-7 | 5 |
| 50. 3,3'-Dichlorobenzidine | 91-94-1 | 5 |
| 51. Benzo(a)anthracene | 56-55-3 | 5 |
| 52. Chrysene | 218-01-9 | 5 |
| 53. bis-(2-Ethylhexyl)phthalate | 117-81-7 | 5 |
| | | |
| 54. Di-n-octylphthalate | 117-84-0 | 5 |
| 55. Benzo(b)fluoranthene | 205-99-2 | 5 |
| 56. Benzo(k)fluoranthene | 207-08-9 | 5 |
| 57. Benzo(a)pyrene | 50-32-8 | 5 |
| 58. Indeno(1,2,3-cd)pyrene | 193-39-5 | 5 |
| | | |
| 59. Dibenz(a,h)anthracene | 53-70-3 | 5 |
| 60. Benzo(g,h,i)perylene | 191-24-2 | 5 |

TABLE 1-2
TCL AND TAL TARGET PARAMETERS AND
PROJECT-REQUIRED DETECTION LIMITS
FOR RESIDENTIAL WELL SAMPLES


| Pesticides/PCBs | CAS Number | Quantitation Limits Water ug/L |
|---|---|---|
| 1. alpha-BHC | 319-84-6 | 0.01 |
| 2. beta-BHC | 319-85-7 | 0.01 |
| 3. delta-BHC | 319-36-8 | 0.01 |
| 4. gamma-BHC (Lindane) | 58-89-9 | 0.01 |
| 5. Heptachlor | 76-44-8 | 0.01 |
| | | |
| 6. Aldrin | 309-00-2 | 0.01 |
| 7. Heptachlor epoxide | 1024-57-3 | 0.01 |
| 8. Endosulfan I | 959-98-8 | 0.01 |
| 9. Dieldrin | 60-57-1 | 0.02 |
| 10. 4,4' - DDE | 72-55-9 | 0.02 |
| | | |
| 11. Endrin | 72-20-8 | 0.02 |
| 12. Endosulfan II | 33213-65-9 | 0.02 |
| 13. 4,4' - DDD | 72-54-8 | 0.02 |
| 14. Endosulfan sulfate | 1031-07-8 | 0.02 |
| 15. 4,4' - DDT | 50-29-3 | 0.02 |
| | | |
| 16. Methoxychlor | 72-43-5 | 0.10 |
| 17. Endrin ketone | 53494-70-5 | 0.02 |
| 18. Endrin aldehyde | 7421-36-3 | 0.02 |
| 19. alpha-Chlordane | 5103-71-9 | 0.01 |
| 20. gamma-Chlordan | 5103-74-2 | 0.01 |
| | | |
| 21. Toxaphene | 8001-35-2 | 1.0 |
| 22. Aroclor-1016 | 12674-11-2 | 0.20 |
| 23. Aroclor-1221 | 11104-11-2 | 0.20 |

**TABLE 1-2**
**TCL AND TAL TARGET PARAMETERS AND**
**PROJECT-REQUIRED DETECTION LIMITS**
**FOR RESIDENTIAL WELL SAMPLES**


| | | |
|---|---|---|
| 24. Aroclor-1232 | 11141-16-5 | 0.40 |
| 25. Aroclor-1242 | 53469-21-9 | 0.20 |
| | | |
| 26. Aroclor-1248 | 12672-29-6 | 0.20 |
| 27. Aroclor-1254 | 11097-69-1 | 0.20 |
| 28. Aroclor-1260 | 11096-82-5 | 0.20 |

**TABLE 1-2**
**TCL AND TAL TARGET PARAMETERS AND PROJECT-REQUIRED DETECTION LIMITS FOR RESIDENTIAL WELL SAMPLES**


| | Required Instrument Detection Limit[1] ug/L | | | Required Matrix Spike Concentrations ug/L | | |
|---|---|---|---|---|---|---|
| Compound | GFAA | ICP | Other | GFAA | ICP | Other |
| Aliminum | | 100 | | | 2000 | |
| Antimony[2] | 5 | | | 20 | 500 | |
| Arsenic | 5 | | | 20 | | |
| Barium | | 50 | | | 2000 | |
| Beryllium | | 5 | | | 50 | |
| Cadmium[2] | 0.5 | | | 2 | 50 | |
| Calcium[3] | | 1000 | | | 50,000 | |
| Chromium | | 10 | | | 200 | |
| Cobalt | | 10 | | | 500 | |
| Copper | | 10 | | | 250 | |
| Iron | | 100 | | | 1000 | |
| Lead[2] | 2 | | | 20 | 500 | |
| Magnesium[3] | | 1000 | | | 25,000 | |
| Manganese | | 10 | | | 200 | |
| Mercury | | | 0.2 | | | 1.0 |
| Nickel | | 20 | | | 400 | |
| Potassium[3] | | 2000 | | | 20,000 | |
| Selenium | 2 | | | 10 | | |
| Silver | | 5 | | | 50 | |
| Sodium[3] | | 1000 | | | 50,000 | |
| Thallium | 2 | | | 20 | | |
| Vanadium | | 10 | | | 500 | |
| Zinc | | 20 | | | 200 | |
| Cyanide | | | 10 | | | 100 |

**TABLE 1-2**
**TCL AND TAL TARGET PARAMETERS AND**
**PROJECT-REQUIRED DETECTION LIMITS**
**FOR RESIDENTIAL WELL SAMPLES**


[1] Instrument Detection Limits (IDL) must be met before any samples are analyzed. THe Lab may submit their quarterly Form XI with each case if all IDLs meet the detection limits. If limits cannot be met by using ICP, use of GFAA required.

[2] ICP analysis results may only be reported for Sb, Cd and Pb, if the concentration is $\geq$ 10 times the IDL of instrument used. If ICP results are reported, all ICP audits are required including matrix spike.

[3] Report Ca, Mg, Na, and K on separate Form V for Matrix Spike if a separate aliquot is used for this spike.

Table 1-3

Summary of Phase II Sampling and Analysis Program
Lenz Oil Service, Inc. RI/FS
(Page 1 of 2)

| | | | | | Investigative Samples | | | QA Samples: Duplicate | | | QA Samples: Field Blank | | | QA Samples: MS/MSD | | | Matrix |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Matrix | Field Parameters | Laboratory | Method | Laboratory Parameter | No. | Freq. | Total | No. | Freq. | Total | No. | Freq. | Total | No. | Freq. | Total | Total |
| Ground Water Samples From Monitoring Wells | -pH, Temperature | ARDL | *see Note (1)* | TCL Volatile Organics | 23 | 1 | 23 | 3 | 1 | 3 | 3 | 1 | 3 | 2 | 1 | 2 | 29 |
| | -Specific Conductivity | RMAL | *see Note (1)* | TCL Semivolatile Organics | 23 | 1 | 23 | 3 | 1 | 3 | 3 | 1 | 3 | 2 | 1 | 2 | 29 |
| | -Water Level | RMAL | *see Note (1)* | TCL Pest/PCBs | 23 | 1 | 23 | 3 | 1 | 3 | 3 | 1 | 3 | 2 | 1 | 2 | 29 |
| | -Qualitative Description | Skinner | *see Note (2)* | TAL Dissolved Metals | 23 | 1 | 23 | 3 | 1 | 3 | 3 | 1 | 3 | 0 | 0 | 0 | 29 |
| | of Water | Skinner | *see Note (2)* | TAL Cyanide | 23 | 1 | 23 | 3 | 1 | 3 | 3 | 1 | 3 | 0 | 0 | 0 | 29 |
| | -Qualitative Organic | Skinner | *see Note (2)* | TAL Total Metals | 23 | 1 | 23 | 3 | 1 | 3 | 3 | 1 | 3 | 0 | 0 | 0 | 29 |
| | Vapor Screening with HNu | | | | | | | | | | | | | | | | |
| NAPL Sample | -Qualitative Description | RMAL | *see Note (3)* | TCLP Organics & Inorganics | 1 | 1 | 1 | 1 | 0 | 1 | 0 | 0 | 0 | 1 | 1 | 1 | 2 |
| | of Oil | RMAL | *see Note (4)* | TCL Volatile Organics | 1 | 1 | 1 | 1 | 0 | 1 | 0 | 0 | 0 | 1 | 1 | 1 | 2 |
| | -Qualitative Organic | RMAL | *see Note (4)* | TCL Semivolatile Organics | 1 | 1 | 1 | 1 | 0 | 1 | 0 | 0 | 0 | 1 | 1 | 1 | 2 |
| | Vapor Screening with HNu | RMAL | *see Note (4)* | TCL Pest/PCBs | 1 | 1 | 1 | 1 | 0 | 1 | 0 | 0 | 0 | 1 | 1 | 1 | 2 |
| | | RMAL | *see Note (2)* | TAL Cyanide | 1 | 1 | 1 | 1 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 2 |
| | | RMAL | *see Note (2)* | TAL Total Metals | 1 | 1 | 1 | 1 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 2 |
| | | RMAL | ASTM | Specific Gravity | 1 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 |
| | | RMAL | ASTM | Viscosity | 1 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 |
| Residential Well Samples | -pH, Temperature | *CompuChem* | *see Note (5)* | TCL Volatile Organics | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 |
| | -Specific Conductivity | *CompuChem* | *see Note (5)* | TCL Semivolatile Organics | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 |
| | -Qualitative Description | *CompuChem* | *see Note (5)* | TCL Pest/PCBs | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 |
| | of Water | *CompuChem* | *see Note (5)* | TAL Cyanide | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 0 | 0 | 3 |
| | -Qualitative Organic | *CompuChem* | *see Note (5)* | TAL Total Metals | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 0 | 0 | 3 |
| | Vapor Screening with HNu | | | | | | | | | | | | | | | | |
| On-Site Soil Samples | -Qualitative Description | ARDL | *see Note (1)* | TCL Volatile Organics | 42 | 1 | 42 | 5 | 1 | 5 | 5 | 1 | 5 | 3 | 1 | 3 | 52 |
| | of Soil | ARDL | *see Note (1)* | TCL Semivolatile Organics | 42 | 1 | 42 | 5 | 1 | 5 | 5 | 1 | 5 | 3 | 1 | 3 | 52 |
| | -Qualitative Organic | ARDL | *see Note (1)* | TCL Pest/PCBs | 42 | 1 | 42 | 5 | 1 | 5 | 5 | 1 | 5 | 3 | 1 | 3 | 52 |
| | Vapor Screening with HNu | Skinner | *see Note (2)* | TAL Total Metals | 33 | 1 | 33 | 4 | 1 | 4 | 4 | 1 | 4 | 0 | 0 | 0 | 41 |
| | | ARDL | *see Note (3)* | TCLP Metals | 26 | 1 | 26 | 3 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 29 |
| | | Skinner | *see Note (2)* | TAL Cyanide | 33 | 1 | 33 | 4 | 1 | 4 | 4 | 1 | 4 | 0 | 0 | 0 | 41 |

Table 1-3

## Summary of Phase II Sampling and Analysis Program
## Lenz Oil Service, Inc. RI/FS
## (Page 2 of 2)

| Sample Matrix | Field Parameters | Laboratory | Method | Laboratory Parameter | Investigative Samples | | | QA Samples: Duplicate | | | QA Samples: Field Blank | | | QA Samples: MS/MSD | | | Matrix Total |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | No. | Freq. | Total | No. | Freq. | Total | No. | Freq. | Total | No. | Freq. | Total | |
| Background Soil Samples | -Qualitative Description of Soil | ARDL | *see Note (1)* | TCL Volatile Organics | 3 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 |
| | | ARDL | *see Note (1)* | TCL Semivolatile Organics | 3 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 |
| | -Qualitative Organic Vapor Screening with HNu | ARDL | *see Note (1)* | TCL Pest/PCBs | 3 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 |
| | | Skinner | *see Note (2)* | TAL Total Metals | 3 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 |
| | | ARDL | *see Note (3)* | TCLP Metals | 3 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 |
| | | Skinner | *see Note (2)* | TAL Cyanide | 3 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 |
| Surface Water Samples | -pH, Temperature<br>-Specific Conductivity<br>-Qualitative Description of Water | ARDL | *see Note (3)* | TAL Total Metals (see note below) | 6 | 1 | 6 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 0 | 0 | 8 |

KEY:
ARDL = Applied Research Development Laboratory
RMAL = Rocky Mountain Analytical Laboratory
CompuChem = CompuChem Laboratories, Inc.
Skinner = Skinner & Sherman Laboratory, Inc.
TCL = Target Compound List
TAL = Target Analyte List
TCLP = Toxicity Characteristics Leaching Procedures
Pest/PCBs = Pesticides/PCB
ASTM = American Society for Testing and Materials
MS/MSD = Matrix Spike/Matrix Spike Duplicate
SAS = Special Analytical Sample
NAPL = Nonaqueous Phased Liquids

NOTES:
*(1) Method CLP RAS SOW OLM01.0*
*(2) Method CLP RAS SOW ILM01.0*
*(3) SW846-1311 for sample preparation only. Analysis using method in Note (4).*
*(4) TCLP analysis using methods: 8240, 8270, 8080, and 8150 (RMAL SOP's in Appendix A)*
*(5) Low concentration samples using CLP SOW OLC01.0 for organics and CLP SOW ILC01.0 for inorganics*
-Water samples analyzed for TAL dissolved metals will be field filtered prior to sample preservation. Total metals samples will not be field filtered.
-Metals samples will be preserved with HNO3; cyanide samples with NaOH; volatile organic compound samples with HCl
-One trip blank sample consisting of two 40-ml glass vials filled with organic-free deionized water will be included with each shipment of aqueous samples targeted for volatile organic analysis.
-Surface water samples will only be analyzed for the TAL metals rejected during the Phase I sampling round
-MS/MSD samples are not included in the Matrix Total.

TABLE 3-1

**PROJECT-REQUIRED PRECISION AND ACCURACY REQUIREMENTS FOR LABORATORY ANALYSES**

| Parameter | Accuracy (percent) | Precision (percent) |
|---|---|---|
| TCL Volatiles | CLP | CLP |
| TCL Semivolatiles | CLP | CLP |
| TCL PCBs/Pesticides | CLP | CLP |
| TAL Cyanide | CLP | CLP |
| TCLP Extract Analysis (~~Organics and~~ Inorganics) | CLP | CLP |
| TCLP Extract Analysis (organic) | RMAL SOP's (Appendix A) | RMAL SOP's (Appendix A) |
| Low Concentration Analysis (organic and inorganic) | CLP | CLP |
| Viscosity | N/A | N/A |
| Specific Gravity | N/A | N/A |

Notes:

"CLP" means the accuracy and precision requirements will be those specified in the appropriate CLP SOW.

"N/A" means that precision and accuracy requirements are not applicable to the specified laboratory analysis.

**TABLE 4-1**
**SAMPLE VOLUME, CONTAINERS AND PRESERVATION TECHNIQUES FOR WATER AND SOIL SAMPLES**


| MATRIX | ANALYTICAL FRACTION | CONTAINER | PRESERVATION | FILLING PROCEDURES | MAXIMUM HOLDING TIMES |
|---|---|---|---|---|---|
| Water | TCL Volatile Organic Compounds | 3 - 40 ml glass vials with Teflon-lined septums | Cool - 4 degrees Celsius<br>HCl to pH < 2 | Zero headspace - no air bubbles | 14 days |
| Water | TCL Semivolatile Organic Compounds | 2 - 1 liter amber glass bottles with Teflon-lined caps | Cool - 4 degrees Celsius | Fill to neck of bottle | 7 days from collection or<br>5 days from VTSR,<br>40 additional days for analysis |
| Water | PCBs/Pesticides | 2 - 1 liter amber glass bottles with Teflon-lined caps | Cool - 4 degrees Celsius | Fill to neck of bottle | 7 days from collection or<br>5 days from VTSR,<br>40 additional days for analysis |
| Water | TAL Dissolved Metals | 1 - 1 liter polyethylene bottle | Nitric acid to pH<2<br>Cool - 4 degrees Celsius | Field filter and fill to neck of bottle | 6 months except for Hg which is 26 days |
| Water | TAL Total Metals | 1 - 1 liter polyethylene bottle | Nitric Acid to pH<2<br>Cool - 4 degrees Celsius | Fill to neck of bottle | 6 months except for Hg which is 26 days |
| Water | Cyanide | 1 - 1 liter polyethylene bottle | Sodium hydroxide to pH>12<br>Cool - 4 degrees Celsius | Fill to neck of bottle | 14 days |
| Soil | TCL Volatile Organic Compounds | 2 - 4 oz. wide-mouth glass bottles | Cool - 4 degrees Celsius | Fill completely, no head space | 10 days |
| Soil | TCL Semivolatile Organic Compounds<br>PCBs/Pesticides | 1 - 1 liter wide-mouth glass bottle | Cool - 4 degrees Celsius | Fill completely | 10 days |
| Soil | TAL Total Metals/Cyanide | 1 - 8 oz. wide-mouth glass bottle | Cool - 4 degrees Celsius | 3/4 Full | 6 months for metals,<br>14 days for cyanide |
| Soil | TCLP Metals | 2 - 1 liter amber, wide mouth glass jar with Teflon-Lined lid. | None | Fill to neck of bottle | Per SW846-131 protocol |
| NAPL | TCL Volatile Organic Compounds | 1 - 8 oz. wide-mouth glass bottle | None | Zero headspace-no air bubbles | 14 days |
| NAPL | TCL Semivolatile Organic Compounds<br>PCBs/Pesticides | *1 - 8 oz. wide-mouth glass bottle* | None | *Not applicable* | 7 days from collection or<br>5 days from VTSR,<br>40 additional days for analysis |
| NAPL | TAL Total Metals/Cyanide | *To be taken from the semivolatiles bottle* | None | Fill to neck of bottle | 6 months except for Hg which is 26 days |
| NAPL | Viscosity/Specific gravity | *To be taken from the 32-oz. TCLP bottle* | None | Not applicable | Not applicable |
| NAPL | TCLP | *1 - 32 oz. wide-mouth glass bottle*<br>*1 - 4 oz. wide-mouth glass bottle* | None | *Zero headspace-no air bubbles* | Per SW846-131 protocol |

KEY VTSR = Verified Time of Sample Receipt
NAPL = Non Aqueous Phased Liquid

**Table 10-1**

**Summary of Laboratory Performance and System Audits**
Lenz Oil Service, Inc. Site

| Laboratory | Audit Type | Audit Frequency | Auditor |
|---|---|---|---|
| Skinner & Sherman | WS/WP PES | Quarterly | USEPA |
| | NYDOH | Quarterly | N.Y. State |
| | Internal Lab Procedure | Semiannually | Lab QAO |
| | Blind PE samples (inorganics only) | Quarterly | USEPA |
| | System Audit | Annually | Corporate QA Program Manager |
| RMAL | WS/WP PES | Semiannually | USEPA |
| | Internal Lab Procedure | Quarterly | Lab QAO |
| | Blind PE samples | Quarterly | USEPA |
| | Site Audit | Annually | Corporate QA Program Manager |
| | Site Audit | Not Specified | USEPA |
| | Site Audit | Not Specified | Army Corp of Engineers |
| | Blind PE samples | Not Specified | Army Corp of Engineers |
| | Site Audit | Not Specified | U.S. Navy - NEESA |
| | Blind PE samples | Not Specified | U.S. Navy - NEESA |
| CompuChem | WS/WP PES | Semiannually | USEPA |
| | Internal Lab Procedure | Quarterly | Lab QAO |
| | Blind PE samples | Quarterly | USEPA |
| | Internal Lab Procedure | Quarterly | Lab QAO |

KEY: CompuChem = CompuChem Laboratories, Inc.
RMAL = Rocky Mountain Analytical Laboratory
NYDOH = New York Department of Health
PE = Performance Evaluation
QA = Quality Assurance
QAO = Quality Assurance Officer

# APPENDIX A

# STANDARD OPERATING PROCEDURES FOR NON-CLP ANALYTICAL METHODS

# GC/MS ANALYSIS OF VOLATILE ORGANICS

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| **LM-RMA-3001 VOA by GC/MS** | 3.0 | 4/24/90 |

Supersedes: 2.0

## 1. SCOPE AND APPLICATION

1.1 Analytes: Quantitative analysis is performed for the compounds listed in Appendix A. Qualitative analysis by mass spectral identification can be performed on other purgeable, chromatographable compounds.

1.2 Reporting Limits: Test Specific

1.3 Applicable matrices: Water, soil, solids, sludge and waste.

1.4 Dynamic range: Analytes can typically be quantitated between 5 ppb and 200 ppb for water samples and soils analyzed by the low level direct purge method. Analytes in soils analyzed by the methanol extraction method can be quantitated between 500 and 20000 ppb.

1.5 Analysis time: Approximate analytical time is 40 minutes per GC/MS run. The time required for data reduction will be dependent upon the complexity of the sample.

| | |
|---|---|
| Prepared by: | Date: |
| Management Approval: | Date: |
| QA Officer Approval: | Date: |

1.6 The applicability of this Standard Operating Procedure to particular matrices and/or analytical requests not specifically stated herein is determined by the GC/MS department manager and communicated to the client manager. The GC/MS department manager has the authority to make modifications to the SOP for certain matrices and/or analyses if his technical judgement deems it necessary and appropriate.

2. METHOD SUMMARY

2.1 Aqueous samples are analyzed by quantitative and/or qualitative purge and trap-GC/MS using an internal standard method.

2.2 Solid samples are extracted with methanol and the extract is analyzed by purge and trap-GC/MS for quantitative and/or qualitative identification using the same internal standard method.

2.3 Alternatively, lower detection limits can be achieved for analytes in soil samples by using the direct purge method.

3. COMMENTS

3.1 All deviations made from this SOP **MUST** be approved by the supervisor and well documented.

3.2 All reusable glassware should be washed with hot, soapy water and rinsed thoroughly. This is followed by a rinse with HPLC grade (or better) Methanol. Finally, the glassware is baked in a 105$^{o}$ oven.

3.3 Care should be taken not to introduce contamination into the samples. Particular attention should be paid to common laboratory solvents, such as acetone and methylene chloride.

3.4 Care should be taken when handling samples for volatiles analysis to avoid loss of analytes.

3.5 Do not overstock traps. Tenax has a lifetime of only a few months. Traps will also degrade with use due to the breakdown of tenax from rapid temperature changes.

4. SAFETY ISSUES

4.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely defined, however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible.

4.2 A Material Safety Data Sheet (MSDS) is available for all laboratory standard and reagent chemicals. The appropriate MSDS must be read before handling the chemical(s).

4.3 All laboratory personnel should be thoroughly familiar with the laboratory Safety Manual and Hazard Communication Standard before undertaking any laboratory work.

4.4 Samples should be considered as hazardous. Appropriate protective clothing, such as gloves, lab coat and safety glasses should be worn when handling samples.

5. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

5.1 Standard 40 mL glass screw-cap VOA vials with Teflon-faced silicone septa may be used for both liquid and solid matrices. Solid samples may also be collected in wide-mouth glass jars with Teflon-lined caps. Samples should be introduced into the containers with minimum agitation to avoid loss of volatile components. For liquid samples, each VOA vial should be filled without introduction of bubbles. Fill until there is a meniscus over the lip of the vial. The lid with septum (Teflon side toward the sample) should be tightened onto the vial. After tightening the lid, the vial should be inverted and

tapped to check for air bubbles. If there are any air bubbles present the sample must be retaken. Sample containers for solid samples should be filled as completely as possible with minimum air space.

5.2 Water samples may be preserved with HCl to extend the holding time of samples to be analyzed for aromatic compounds. Aromatic compounds are particularly susceptible to biodegradation at normal pH. The pH of the sample should be adjusted to less than 2 with HCl in the field at the time of sampling.

5.3 All samples must be iced or refrigerated at $4^{o}C$ from the time of collection until analysis.

5.4 All aqueous samples must be analyzed within 7 days of collection. If aqueous samples are preserved with HCl, the holding time is extended to fourteen days from date of collection. Medium level soil/waste samples must be extracted and analyzed within 14 days of collection. Soil samples which are analyzed by the low level method must be analyzed within 14 days from the sampling date.

6. APPARATUS

6.1 Gas-tight syringes: 10 uL, 25 uL, 50 uL, 100 uL, 250 uL, 500 uL, and 1 mL.

6.2 Syringe valve: two-way with Luer ends.

6.3 5 mL gas tight syringe, luer-lock type.

6.4 1 mL disposable pipet.

6.5 Top-loading balance capable of weighing 0.1 g.

6.6 Glass culture tubes with screw cap and teflon liner.

6.7 Volumetric flasks: 10 mL - 100 mL.

6.8 Screw top vials with teflon-lined septa for storage of methanol extracts and extract dilutions.

6.9 Disposable pasteur pipets.

6.10 Stainless steel spatula.

6.11 Gas Chromatograph for screening with all required accessories such as syringes, analytical columns, gases, flame ionization detector, recording device and optional autosampler or optional headspace analyzer. A data system is recommended for data acquisition, integration and output.

6.12 Heated bath or heater capable of maintaining the purging chamber to within +/- 1°C over the temperature range of ambient to 100°C.

6.13 Purge-and-trap device including the sample purger, the trap and the desorber. (Tekmar LSC-2 or equivalent) Device may include automatic sampler (Tekmar ALS or equivalent.)

6.14 Purging Chamber

6.14.1 Two piece glass purge cell with ground glass joint and airtight o-ring connection, single piece glass purge tube, and/or glass-fritted sparger or equivalent.

6.15 Traps Four phase: Supelco Volatile Purge trap, or equivalent.

6.15.1
1. 1 cm 3% SP-2100 on 60/80 Chromosorb WAW
2. 15 cm Tenax TA
3. 8 cm 35/60 Silica Gel Gd-15
4. 1.5 cm Carbosieve added to base

6.16 Chromatographic Columns

6.16.1 Screening column: JW P/N 125-1334 DB624. Mega-Bore 30m X .53mm I.D., or equivalent

6.16.2 Analytical Column:

A. JW P/N 125-1334. DB624 Mega-Bore 30m X .53mm I.D., or equivalent.

6.17 Gas Chromatograph/Mass Spectrometer

6.17.1 Gas chromatograph: An analytical system that includes a temperature-programmable gas chromatograph including all required accessories such as syringes, columns, and gases.

6.17.2 Mass spectrometer: Capable of scanning from 35 to 300 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

6.17.3 GC/MS interface: An interface that gives acceptable calibration points at 50 ng or less per injection for each of the analytes and achieves all acceptable performance criteria. An all glass interface is recommended.

6.17.4 Data system: A computer system must be interfaced to the mass spectrometer which will allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the analytical run. The software should be capable of producing extracted ion profiles and integrating these abundances. The EPA/NIH Mass Spectral Library should also be available on the system.

7. REAGENTS AND STANDARDS

7.1 Reagent water - Carbon filtered house distilled water, continuously sparged with an inert gas prior to use for volatiles analyses. Interferents should not be observed at or above the reporting limit of the parameters of interest.

7.2 GC/MS Calibration solution: 50 ng/uL bromofluorobenzene in methanol.

7.3 Internal standard and surrogate spiking solutions.
MS-VOA-IS (See SOP No. LS-RMA-010)
MS-VOA-SCS (See SOP No. LS-RMA-011)

7.4 Laboratory Control Sample standard spiking solution.
MS-VOA-LCS (see SOP No. LS-RMA-012)

7.5 GC screening standard. (See RMAL SOP "Volatiles Screening Analysis by GC/FID - SOP No. LM-RMA-3022.)

7.6 Calibration standard solutions:

A. MS-VOA-CS1 (See SOP No. LS-RMA-001)
B. MS-VOA-CS2 (See SOP No. LS-RMA-009)
C. MS-VOA-AP9-CSA (See SOP No. LS-RMA-015)
D. MS-VOA-API-CSA (See SOP No. LS-RMA-013)
E. MS-VOA-B&V-CSA (See SOP No. LS-RMA-039)

7.7 Standards containing other purgeable compounds can be prepared on special request by clients.

7.8 Methanol: purge-and-trap grade or equivalent. Store apart from other solvents.

8. PROCEDURE

8.1 Scheduling Samples for Analysis

8.1.1. Samples will be scheduled for analysis by experienced analysts and/or supervisors according to the following priorities:

a) Meet holding times
b) Meet promised due date

8.2 Methanol extraction of soil samples for analysis by the medium-level method. (minimum reporting limit: 500 ug/kg)

8.2.1 Carefully mix the contents of the sample container. Do not discard any supernatant liquids. Weigh 5 g of sample to the nearest 0.1 g. Note and record the actual weight.

8.2.2 Determine the percent moisture as in section 8.4 (if required).

8.2.3 Add 10 mL purge & trap methanol, (25 ug/mL). Cap and shake for 2 minutes.

8.2.4 Allow the soil to settle or centrifuge and transfer a portion of the extract to a labelled autosampler vial, leaving a minimum of headspace. Store these extracts at 4°C prior to analysis.

8.2.5 Prepare methanol blank for each day of extraction using same procedure as medium level.

8.2.6 Screen the methanol extracts as described in section 8.3

8.3 SCREENING. For complete information on the screening procedures for GC/MS Volatile compounds, refer to RMAL SOP No. LM-RMA-3022, Volatiles Screening Analysis by GC/FID.

8.4 Percent Moisture Determination (optional)

8.4.1 Weigh 5-10 g of sample into a tared weighing dish. Record the samples wet sight. Allow to dry overnight at 105°C. Re-weigh the sample. Record the samples dry weight. (Correct for weight of weighing dish) Calculate percent moisture using equation 10.2.7.

8.5 Instrument Set-Up for the GC/MS

8.5.1 Chromatographic Conditions

8.5.1.2 Mega-bore Capillary Column (See Section 6.17.3)

8.5.1.3 Temperature Program for Capillary Column:
Initial Temperature: -10°C
Temperature Ramp:
Program 1: 20°/min 1.5 minutes to 20°
Program 2: 5°/min 16 minutes to 100°
Program 3: 20°/min 3 minutes to 160°
Final Time: 7 minutes

These parameters are subject to change and are given as guidelines.

8.5.2 Mass Spectrometer parameters:

8.5.2.1 Mass range: 35-300 AMU

8.5.2.2 Scanning rate: 1 second/scan

8.5.3 Purge and Trap Parameters

8.5.3.1 Trap: As described in Section 6.16

8.5.3.2 Purge Flow: 30-50 mL/min (measured at vent in *purge mode*).

8.5.3.3 Purge pressure: 20 $lb/in^2$ (measured on the gauge)

8.5.3.4 Purge Time: 12 minutes
Desorb Time: 2-3 minutes
Bake Time: 8-10 minutes

8.6 Installation and conditioning of trap in the purge-and-trap device.

8.6.1 All traps should be installed with accompanying brass nut down and inlet marker to the top. A brass nut and ferrule should be used at the bottom connections. A teflon ferrule should be used at the top. The top nut should only be finger tightened.

8.6.2 Purge flow is adjusted by measuring the flow at the vent in purge mode.

The faster the purge flow the more efficient the purging process. The limit to increasing the flow is governed by the ability of the trap to adsorb and contain the purged analytes.

If the total purge volume is too large (flow rate too high) then the lightest compounds such as the gases will break through the end of the trap and their respective RFs will drop correspondingly. If the purge flow is slow the heavier and the more polar compounds will not purge efficiently. Thus, to improve the response of the CS2 compounds, the purge flow should be decreased and to improve bromoform response the purge flow should be increased.

In general, the purge flow should be between 30 and 50 mL/min.

If optimization of overall RFs by changing the purge flow cannot be attained, a new trap may need to be installed.

8.6.3 The trap is conditioned by placing an empty sparge cell on the purge & trap device and placing the unit into purge mode. After a few minutes, step into "bake" mode for 10-15 minutes.

8.7 Instrument Calibration

8.7.1 BFB

8.7.1.1 Each GC/MS MUST be hardware tuned to meet SW-846 3rd. Edition ion abundance criteria for BFB. The instrument tune MUST be verified at the start of every operation. When the tuning criteria are met, standards and samples may be run for a period of 12 hours depending on contract or method requirements. Ion abundance criteria are listed in SW-846 3rd Edition.

8.7.1.2 50 ng of BFB is directly injected on to GC column and peak data are acquired.

The spectrum of BFB is evaluated. Ion abundance criteria given in Table C-1 must be met. Choosing a scan that is one scan to the left or right of the peak apex and reasonable background subtraction or enhancement is acceptable.

Manipulations which will distort the spectrum, such as excessive background subtraction or multiple scan additions, are not acceptable.

8.7.1.3 If the ion abundance criteria are not met, the instrument is retuned and BFB reinjected.

8.7.1.4 If the ion abundance criteria are met, a hardcopy of the spectrum, mass abundance table, and ion abundance summary is generated and stored on file.

8.7.2 Initial calibration. An initial calibration must be performed before analysis of samples can begin. This consists of a five point calibration curve for the analytes of interest. Low-level soil analysis requires an initial calibration in which the standards in water are heated to 40° C during the purge step. Standard solutions are prepared in reagent water at concentrations of 20 ug/L, 50 ug/L, 100 ug/L, 150 ug/L and 200 ug/L, and purged, desorbed and analyzed on the GC/MS/DS. All 5-points must be injected within 12 hours of BFB injection which has met abundance criteria.

Certain compounds have been selected to monitor instrument performance. These compounds are identified as System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC).

The average response factor for each System Performance Check Compounds (SPCC) MUST be greater than or equal to 0.300, except for bromoform, which must be greater than or equal to 0.250.

The percent relative standard deviation (%RSD) for each Calibration Check Compound (CCC) MUST be less than 30%. The %RSD for all compounds should also be reviewed. Any values greater than 40% should be evaluated before analyses are continued.

If samples are NOT being analyzed for these specific compounds, the criteria for these compounds need not be met. The fact that these compounds are not in the analyte set should be documented.

See current CLP-SOW for a summary of CCC and SPCC criteria.

8.7.2.1 The five standard solutions are typically analyzed in the following order: 50 ppb, 20 ppb, 100 ppb, 150 ppb and 200 ppb.

Standards are prepared using a 5 mL calibrated gas-tight syringe which is rinsed with purged reagent water and then filled to a volume of 5 mL.

Internal standards, surrogates and analytes are then added.

8.7.2.2 Standards are analyzed using the ALS. Standards are introduced into a purge chamber on the ALS followed by purging & desorption into the GC/MS/DS as previously described.

8.7.2.3 The area counts of the internal standards in the 50 ppb standard are evaluated before calibration is continued. The first internal standard EICP (Bromochloromethane) should be between 15,000-50,000 counts depending on the instrument. This will minimize saturation in the higher level standards. If the peak area is outside these limits, the analyst should adjust the electron multiplier and re-inject the 50 ppb standard.

8.7.2.4 The SPCC and CCC limits MUST be verified and met. If any criteria is not met, the analyst and supervisor will choose appropriate corrective action. Sample analysis cannot take place until CCC and SPCC criteria are met for the initial calibration.

8.7.2.5 The quantitation list is reviewed to ensure that the internal standards are correctly identified and that the analytes are properly quantitated. Special attention is payed to the following compounds to ensure correct peak assignment:

dichlorodifluoromethane
chloroethane
vinyl chloride
acetone
cis-1,3-dichloropropene
trans-1,3-dichloropropene
2-butanone
2-hexanone
4-methyl-2-pentanone
xylene isomers
cis-1,2-Dichloroethene
trans-1,2-Dichloroethane
dichlorobenzene isomers
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene

8.7.2.6 If the standard evaluation criteria have been met, update with the response factors and retention times of the 50 ppb standard just analyzed. Standards and samples are processed.

8.7.2.7 The 20 ppb, 100 ppb, 150 ppb and 200 ppb standard are then analyzed and evaluated in the same manner as the 50 ppb standard, using programs on the data system designed for multipoint calibration data.

8.7.2.8 A table which lists each standards Response factors, the average Response factors and % RSD for each compound is generated. The % RSD for CCC and Response factors or SPCC are then evaluated. Compounds having % RSD greater than 30% are checked to verify accuracy of quantitation of each standard. Response factors which differ greatly from the compound average are also investigated.

8.7.2.9 If all CCC and SPCC criteria are met, the data are stored in an Initial Calibration file. This file is then referred to during continuing calibration.

8.7.3 Continuing calibration

The 50 ppb standard is used as the continuing calibration standard. It MUST be analyzed for each twelve hour period, immediately following a successful BFB analysis. The BFB injection time starts the twelve hour clock.

The response factor of each CCC compound should be compared to the initial calibration using multipoint calibration software. The percent difference for each CCC compound should be less than or equal to 25%. Analyses may continue if any one CCC or any one SPCC is not within limits but only if contractually allowable or approved by a supervisor. Response factors for all analytes should be reviewed. Any significant deviations from the initial calibration should be evaluated and quantities checked before analyses are continued. (e.g. deviations of greater than 30%)

8.7.3.1 The continuing calibration standard is run as described in section 8.7.2.1. The concentration of each of the surrogate compounds and the internal standard compounds is 50 ppb.

8.7.3.2 Process the data file as described in Section 8.7.2.2. (See Section 8.10.1).

8.7.3.3 The quantitation list is reviewed to ensure that the internal standards are correctly identified and that the analytes are properly quantitated. Check the compounds listed in 8.7.2.5 for proper integration and peak assignment.

8.7.3.4 A program on the data system is used to compare the continuing calibration standard to the initial calibration. The CCC and SPCC compounds are evaluated as outlined in Section 8.7.3. The RF and percent deviations from the initial calibration of all the other compounds are reviewed. (e.g. compounds with deviations > 30% are checked for proper peak assignment and integration.)

8.7.3.5 If the standard evaluation criteria are NOT met, corrective action must be taken. Corrective action may include replacement of the trap or the analytical column or replacement of calibration standards which may have degraded. Document corrective action taken. Repurge the continuing calibration standard and/or perform a new initial calibration. (See Section 8.7.2.)

8.7.3.6 If these criteria are not met and the decision is made to continue analysis, the rationale must be documented, approved by a supervisor, and kept on file with the continuing calibration record.

8.7.3.7 If the standard evaluation criteria have been met or the decision has been made to continue, update the data system with the Response factors and retention times of the continuing calibration standard just analyzed.

8.8 File Naming Conventions

The file names of BFB, standards, QC, and samples acquisitions are based on the instrument letter designation, and a continuing numeral integer progression from 0001 to 9999. When file number 9999 is reached, go to 0001. File names are recorded in the instrument log book on a daily basis as acquisitions are conducted.

8.9 Sample Analysis

8.9.1 Scheduling

8.9.1.1 Analyses are scheduled by the supervisor or a senior analyst generated from the Laboratory Information Management System (LIMS). Backlogs are available in the laboratory.

8.9.1.2 The analyst will determine the type of analysis requested. Modifications to the normal analytical protocol such as low detection limits, special compounds or special criteria will be indicated on the backlog or found in the LIMS database.

8.9.1.3 Sample ID's are tracked based on RMA project and sample numbers assigned by Sample Receiving. These ID's should be used when entering the "Sample ID" accounting parameter in data acquisition programs.

8.9.2 Analysis Sequence

8.9.2.1 A method blank (surrogate control sample) must always be analyzed after a calibration standard. See section 9.3 for method blank acceptance criteria. Duplicate control samples (DCS) are analyzed as required by SOP No. M-EQA-002. SCS and DCS sample results must be verified before samples can be analyzed. See Section 9.0 for QC criteria for SCS and DCS samples.

8.9.2.2 If the QC criteria are met, the sample analysis can begin. Consult the Enseco QAPP for specific requirements. Matrix spike and matrix spike duplicates and any other Project Specific QC when requested by the client, are analyzed in conjunction with the associated sample.

8.9.2.3 Samples must be injected within 12 hours of the BFB injection, unless a method or contract specifies otherwise.

8.9.3 Analysis of Water Samples

8.9.3.1 All samples must be allowed to warm to ambient temperature before analysis.

8.9.3.2 All purging cells are washed with soap and hot water, rinsed with water, then methanol and baked dry.

8.9.3.3 Screen data is first reviewed to determine if any samples require dilutions. When screens indicate samples are clean, or have low levels of volatile compounds, 5 mL will be used for analysis. Sample volume will be measured using a 5 mL gas-tight syringe. If screens indicate high levels of volatile compounds are present, sample volume will be adjusted so that the compound in highest concentration will quantitate at approximately 100 ppb. The final volume of sample is adjusted with reagent water so that 5 mL is always purged. Dilutions of up to 1:1000 (5 ul of sample or more) can be made directly in the 5 mL syringe. If necessary, serial dilutions of 1:10 are made so that 5 ul of sample will be the minimum volume injected into the 5 mL syringe.

8.9.3.4 The sample is carefully poured into the syringe barrel nearly filling the barrel. Vent any residual air while adjusting the samples volume to 5.0 mL. A second syringe may be filled at this time (if capped off), if there is only one vial for analysis. This will maintain the integrity of the sample. A sample may be stored, refrigerated, in a syringe for 24 hours.

8.9.3.5 Add 10 uL of the internal standard + surrogate (VOA IS/SCS) mix to the sample using 25 uL gas-tight syringe.

8.9.3.6 The syringe valve assembly is attached to the valve on the purging chamber. The valve on the chamber is opened and the sample is injected into the purging chamber.

8.9.3.7 The valve on the purging chamber is closed and the syringe is removed. The sample is then purged and analyzed using the same instrument parameters as those used to analyze the calibration standards.

8.9.3.8 Sample number and sample concentrations are recorded in the instrument log book.

8.9.3.9 Surrogate control samples and duplicate control samples are prepared using 5.0 mL of purged reagent water. Add 10 uL of internal standard and surrogate solution (VOA IS/SCS) to the water in the syringe. For DCS samples, add 10 uL of DCS spike solution in addition to the surrogates and internal standards. Place each aliquot into adjacent purge vessels. SCS must meet blank criteria described in Section 9.3.

8.9.3.10 Matrix spikes are prepared in the same manner. The additional aliquot(s) of the sample to be spiked is (are) placed in 5 mL gas-tight syringes. If the sample requires dilution, dilute the matrix spike aliquot as well. Add 10 uL of the internal standard + surrogate (VOA IS/SCS) mix and 10 uL of the matrix (VOA-DCS) spike solution to each aliquot prepared. Analyze the sample and matrix spike(s) consecutively, if possible.

8.9.4 Analysis of Methanol Extracts for the Medium Soil Method

8.9.4.1 The samples are extracted and screened as described in Section 8.2 and 8.3.3. A medium level blank consisting of 5 mL reagent water, 10 mL IS/SCS mix and 100 ul of methanol is run with each set of medium level samples.

8.9.4.2 A 5 mL gas-tight syringe is filled with purged reagent water. Adjust the volume to 5 mL. If screening data indicates sample requires between 5 uL and 100 uL of extract, then the appropriate volume of the methanol extract is added to the reagent water. If less than 5 uL of extract is indicated, a 1:10 dilution in Methanol is required. The extract is further diluted as necessary so that at least 5 uL of diluted extract is added to the reagent water purged. The maximum amount of Methanol extract that should be purged is 100 uL. It is acceptable to add the extract directly to the syringe, or directly to the reagent water after it has been added to the purge cell. Use Hamilton microliter syringes to deliver the 5 uL to 100 uL aliquot.

8.9.4.3 Add 10 uL of VOA IS/SCS mix to the syringe and place the sample in the purging chamber as described above.

8.9.4.4 Matrix spike samples are prepared at the instrument. The same volume of extract used in the sample analysis is used in the matrix spike. The matrix is prepared as described in sample analysis using 5 mL reagent water, 10 uL VOA IS/SCS mix, and 10 uL DCS mix.

8.9.5 Low Level Soil Analysis

8.9.5.1 QC SCS and DCS samples will be analyzed as described in Section 8.9.3.9, with one exception, soil DCS samples are to include 5 grams of Ottawa sand.

8.9.5.2 The screen data is required to determine the weight of samples to be purged (between 0.5g and 5.0g).

8.9.5.3 Mix the contents of the sample container. Do not discard any supernatant liquids. The amount of sample to be purged as determined by the screen is weighed into a tared purge vial. Note and record the actual weight to the nearest 0.1 g.

8.9.5.4 Prepare a 5.0 mL aliquot of reagent water containing 10 uL of VOA IS/SCS solution in a 5 mL syringe. Add the water to the vial, purge vial + sample by injecting the water through the valve on the purge and trap device as the purge cell is lifted into position. This will mix the soil and water and ensure the helium purge tube will not be blocked. Low level soils (and their corresponding calibrations) are purged at a temperature of 40$^{o}$C.

8.9.5.5 Repeat steps 8.9.5.3 and 8.9.5.4 for each sample.

8.9.5.6 Surrogate control samples and duplicate control samples are prepared as stated in section 8.9.3.9 with one exception. Each DCS sample also contains 5.0 grams of a solid, contaminant-free matrix, such as Ottowa Sand. No solid matrix is used. Add 10 uL of VOA IS/SCS solution to the water in the syringe. For DCS samples, also add 10 uL of matrix spike (VOA DCS) solution. Place each aliquot into a purge vial and affix to the unit as described above.

8.9.5.7 Matrix spikes are prepared using the same procedure as the samples. Weigh the same amount of sample for each aliquot required. In addition to the 10 uL of VOA IS/SCS added to the reagent water, 10 uL of DCS solution is added. Matrix spike samples should be analyzed along with the corresponding sample. Affix each aliquot to the unit as described above.

8.9.6 Data Acquisition

8.9.6.1 Samples are analyzed using the same instrument parameters as the calibration standards. Recommended conditions are listed in section 8.5. Blank, SCS and DCS data must be verified before sample analysis.

8.9.6.2 Sample data is acquired either as a single run, or as in an automated series of sample runs.

If a limited list of compounds is being used, the run time (number of scans desired) may be shortened, as appropriate to the compounds being analyzed. The actual number or scans collected may vary slightly from one instrument to another.

8.9.6.3 File names, the date, analyst initials, sample identification and appropriate dilution are recorded in the instrument log book.

8.10 Data Processing and Reduction

8.10.1 Following sample data acquisition, each sample data file is processed using the same software used to search for, identify and quantitate target compounds as was used for calibration data.

8.10.2 The data package generated contains many of the following printouts:

8.10.2.1 **Log Page:** Included on this page are the header information, GC conditions, scan parameters, and instrument tuning parameters that were used at the time of data acquisition.

8.10.2.2 **Chromatogram:** Included on this page is the header information and the reconstructed ion chromatogram displaying the range of scans used during data acquisition.

8.10.2.3 **Diagnostic Report:** Included in this page is a Table of Library entries and chromatographic and Library search information used in the computerized target compound evaluation.

8.10.2.4 **Quantitation Report:** Included in this section is the list of target compounds, internal standards and surrogate standards used for processing the data file, and compound library entry numbers. This section contains: **1)** Chromatographic information such as retention times (scan numbers) and relative retention times and, **2)** Quantitation information such as peak areas, updated library response factors, calculated response factors and quantitated amounts for all library entries.

8.10.2.5 **Quantitation Summary:** Included on this page is a list of internal standards, surrogates and those target compounds which were found in the AutoQuan target compound search routine. Listed along with each compound is the mass, scan, indication of internal reference standard, peak area, response factor and quantitation amounts.

8.10.2.6 **Spectra:** A mass spectra of the peak or each target compound is printed.

8.10.2.7 **Summary Cover Page:** Sample summary which lists surrogate recoveries + all target compounds. These target compounds which were found to have quantitation amounts, have dilution adjustments.

8.10.3 Internal Standard area counts must be monitored on all sample and QC runs. Areas or Percents are recorded in the log book. The areas should not vary by more than a factor of two (-50% to +100%). If the areas do vary by more than a factor of two, the analyst should first check to see if the peak has been correctly integrated. If the area is still outside the limits, the purge & trap/GC/MS system should be checked and corrections made if necessary. The sample will be reanalyzed if problems were detected and corrected. If, upon verification that the instrument is operating properly, the areas are still outside of the limits, submit both analyses with the data package and document the actions taken. If no problems were detected with the instrument, involve a supervisor to determine if sample reanalysis is necessary.

8.10.4 Surrogate recovery values are calculated and compared to recovery limits in Table D-2. Samples with surrogate recoveries outside QC limits should be checked for proper quantitation. Secondary ions may be used if there are interferences on primary quantitation ions. CLP contracted samples having surrogates outside the limits are reanalyzed. If surrogates are outside limits for the second analysis, both analyses are submitted with data package. Non-CLP samples, with one or more surrogates outside limits, are documented and evaluated on a case-by-case basis. Need for reanalysis will be determined by the analyst and supervisor.

8.10.5 Verify that the sample was analyzed at the proper dilution. If any sample contains target compounds that are greater than the highest point in the initial calibration plus 10% (over 220 ug/L on the QUAN report), the sample should be diluted by the appropriate factor and reanalyzed. Exceptions to this include acetone and 2-butanone for non-CLP work which have demonstrated linearity to 1200 ug/L and 500 ug/L respectively. Any sample that has been diluted should contain levels of compounds (either target or non-target compounds) that are high enough to justify the dilution. In the absence of target compounds, the peak height of non-target compounds should be approximately greater than or equal to the peak height of the nearest internal standard. If not, the sample should be reanalyzed at the correct dilution.

8.10.6 Spectra Verification

The data processing routines generate spectra for all target compounds > 1 ug/L for analyst interpretation. Each positive hit picked by the data system, must be examined by the analyst to either confirm or negate what the processing routine has chosen.

Prior to quantitative identification of target compounds in a sample "standard" mass spectra of known identity should be obtained on the instrument(s) used for sample analysis. Hardcopies of mass spectra from a calibration standard (e.g. 50 ug/mL continuing calibration standard) run on the instrument under similar tuning conditions would serve this purpose.

8.10.6.1 Each mass spectrum generated for a sample data file should be compared to the standard spectrum. EPA criteria for quantitative identification by comparison of mass spectra should be followed. The EPA criteria for comparing spectra are:

1) All ions present in the standard spectrum at a relative intensity greater than 10% of the base peak MUST be present in the sample spectrum.

2) The relative intensities of the ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard specrtum, the corresponding sample ion abundance must be between 30% and 70%.)

3) Ions greater than 10% in the sample spectrum, but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labelled with the contaminant.

8.10.6.2 The analyst should circle the name of target compound on the spectrum hard copy of spectra that meet the criteria listed in Section 8.10.6.1. All spectra that do not meet the EPA criteria should be labelled with the word "No" and the corresponding entries on the quantitation

summary should be crossed out (line out the amounts calculated for those compounds which are not "hits") by the analyst making the judgement.

8.10.6.3 If a compound cannot be verified by all of the criteria listed in 8.10.6.1, but in the technical judgement of the analyst the identification is correct, the compound should be reported.

8.10.7 Summary Sheets

A number of summary sheets are computer generated at the instrument following data file processing and pulling (hardcopying) target compound spectra. These summaries include:

8.10.7.1 Target Compound Summary (Also referred to as "Sum Sheets"): This one or two page summary tabulates pertinent information about a sample analysis including header information (e.g. sample number, dilution used for analysis, sample matrix), data analyzed, analyst's initials, run factor (refer to calculations in Section 10.1.1, final reporting units, surrogate % recoveries, target compounds, amounts >1.0 ug/L, scan number, amount listed on quantitation summary and concentration in sample (expressed in final reporting units, usually ug/L for waters and ug/kg for soils) are tabulated.

8.10.7.2 DCS Summary (Refer to section 9.0 for discussion of DCS QC samples): This one page summary tabulates pertinent information about the DCS samples representing a specific QC lot including QC Lot number, matrix (soil or water), date analyzed, analyst's initials, DCS data file

numbers, list of samples belonging to the QC lot and the QC limits. The program calculates the amount and average percent recovery for each spike compound and the RPD (Relative Percent Difference) between the two DCS recoveries for each spike component. Percent recoveries and RPD's that fall outside the acceptable QC limits are flagged with an asterisk (*) and require further action by the analyst (see Section 9.0).

When generating this summary sheet the analyst can input a complete list of samples belonging to the QC lot for QC tracking purposes. This information can be found in the QC summary notebook kept at each instrument.

8.10.7.3 SCS Summary (See Section 9.0 for discussion of QC samples): This one page summary tabulates pertinent information about the surrogate control sample representing a specific ongoing calibration. Included are QC lot number, matrix (soil or water), date analyzed, analyst's initials, data file number, list of samples belonging to the lot, names of surrogates and QC limits for surrogate % recoveries, and calculated amount and % recovery for each surrogate. Percent recoveries that fall outside the acceptable QC limits are flagged with an asterisk (*) and require further action by the analyst see Section 9.0). When generating this summary sheet, the analyst inputs the complete list of samples associated with the SCS sample for QC tracking and data reporting (Blank Subtraction) purposes. This information can be found in the QC summary note book kept at each instrument.

8.10.8 Library Searches (Optional)

Some data packages will include mass spectral library searches on non-target compounds (excluding internal standards, surrogates & spikes) as outlined in contract requirements or requested by the client. If library searches (also referred to as "TIC's", "TID's" or Tentatively Identified Compounds) are necessary to the data package, the 10 or 15 largest unidentified peaks that are greater than 10% of the peak (RIC) area of the nearest internal standard will be searched against the NBS library for possible matches.

8.10.8.1. The generation of TIC data is partially automated through the use of several RMA programs which perform the following functions:

1) Select non-target, unidentified peaks from the chromatogram for tentative identification,

2) Quantitate unknown peaks using RIC area of closest uninterfered standard as reference peak using a response factor of 1 and run factor adjustment. (See Section 10.2.8 for run factor calculations)

3) Perform and generate hardcopy mass spectral library searches on the unknown peaks (searching against the NBS library of mass spectra),

4) Select preliminary "Best Match" spectra from the library searches and tabulate them with quantitation information for all unknowns,

5) Allow editing of computer's final selections for compound names,

6) Allow manual quantitation of unusually shaped peaks,

7) Generate final summary with amounts calculated to reflect concentration in sample (i.e. run factor applied).

8.10.8.3 After reviewing each library search spectrum, the analyst should indicate the compound name selected by labelling the spectrum (or circling the correct name if it appears on the library search spectrum as one of the computer's selections). If the compound is to be reported as "Unknown" (See Section 8.11.6.5) the spectrum should be labelled as such.

8.10.8.4 When all of the library comparisons have been printed out, a summary sheet listing each peak with the best library fit will print out or be filled out by the analyst. The operator should go through each comparison, determine the probability that the match is correct, and assign a confidence level based on that probability. The confidence levels are:

3 = High, verfied by an authentic standard
2 = Moderate
1 = Low

In addition, two qualifiers,
I = Isomer
C = Chemical Class
can be assigned to the identification.

8.10.8.5 The EPA criteria to be used for making these decisions for tentative identifications are as follows:

1) Relative intensities of major ions present in the library spectrum at a relative intensity greater than 10% of the base peak should be present in the sample spectrum.

2) The relative intensities of the ions specified in (1) must agree within +/- 20% between the library and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

3) Molecular ions present in the library spectrum should be present in the sample spectrum.

4) Ions present in the sample spectrum, but not present in the standard spectrum should be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labelled with the contaminant.

5) Ions present in the library spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

8.10.8.6 If in the technical judgement of the analyst, no valid tentative identification can be made, the compound should be reported as unknown. The analyst should consult with a more experienced analyst or a supervisor if there are questions concerning compound identification.

8.10.9 Label the RIC to identify the internal standards surrogate compounds, target compounds, and TIC if requested. If the sample is particularly dirty and the peaks are buried in other peaks, estimate the location of the peak.

For industrial, non-CLP work the abbreviations IS-internal standard, SS-surrogate standard, SP or SC-spike compound, TC-target compound and TIC-tentatively identified compound are sufficient. In CLP work the internal standards and surrogates are usually stamped with the compound names; the abbreviations SC, TC and TIC are normally sufficient for contract work for spike compounds, target compounds and tentatively identified compounds provided a legend is clearly indicated on each chromatogram page.

8.10.9.1 Each data package submitted by the analyst for secondary review must include an anomaly sheet summarizing any problems or unusual circumstances incurred during sample analysis. If no problems were encountered during analysis, the analyst must still submit an anomaly sheet to that effect. The supervisor must sign the anomally sheet before the package progresses to the data review group.

8.10.9.2 QC Summary Sheets (SCS & DCS)

Each data package submitted for secondary review must include copies of DCS and SCS summary sheets for all QC related to the sample set in the data package.

8.10.10.3 Instrument and sample data are organized by the analyst at each instrument. Instrument tuning records and BFB data are filed at the instrument. STD, SCS, and DCS Raw data are filed separately by date at the instrument. If the above data is required as part of the client deliverable package, copies are made and added to the data package assembly. The data package assembly will

include hardcopies of printouts described in Section 8.10.11 for each sample. TIC library comparison spectra and summaries are included when required.

8.10.11 Summary of Data Package Requirements

When initial data review has been completed by the analyst the data package should be assembled to include the following:

Anomaly Sheet
DCS Summary Sheet
SCS Summary Sheet
Blank Target Compound Summary Sheet
Calibration Data
Out of Control Forms (if necessary) see Section 9.0
Sample Raw Data
Sample Target Compound Summaries (attached to sample raw data)

TIC data and summary (when requested) in some cases contract requirements or client requests will necessitate including tuning and calibration information (i.e. BFB), daily standard raw data and initial and continuing calibration response factor tables.

8.10.12 Upon completion of the data package assembly and review, the data package is passed on to the data review specialist for Level 2 review.

9. QA/QC Requirements (See SOP no.: M-EQA-002)

9.1 DCS. Duplicate laboratory control samples are analyzed for every 20 samples. DCS components are spiked into organic-free water. A solid matrix (Ottawa sand) is incorporated as a representative solid matrix. Aqueous and Solid DCS Q.C. lots are maintained separately.

9.2 SCS. A single control sample is analyzed with every analytical lot. This sample serves as the method blank. An analytical lot for volatile organics is defined as samples analyzed within a twelve hour period.

9.3 Blank. A Method Blank is run with each daily calibration. For samples run under Enseco QC guidelines the SCS will serve as the Method Blank. Medium level soils will also require a medium level blank. All target compounds should be below reporting limits. Consult with a supervisor if any target compound is present at or above rep. limit. Surrogate recoveries must fall between limits set for SCS acceptance. Reanalyze blank or SCS if surrogate recoveries are outside of limits. If recoveries still out, troubleshoot system and recalibrate if necessary.

9.4 Surrogates Spiked into Samples. Surrogate compounds are required to be spiked into all samples and QC samples for this method.

9.5 Matrix specific QC. Matrix spike and matrix spike duplicates are performed at the request of the client. Matrix spike compounds will be the same as those used for the DCS. These compounds will be spiked into aliquots of the sample specified by the client at the same concentration level as the DCS.

9.6 Data acceptability is based upon the results of the Duplicate Control Samples unless otherwise negotiated with the client. QC data must fall within established control limits in order for the laboratory to be considered "in control" when samples from that QC lot were analyzed. An out-of-control form must be filled out if any of the DCS or SCS criteria is not met.

9.6.1 At least 80% of the DCS average recovery data, and at least 80% of the DCS precision data and at least 80% of the SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Blank values must be acceptable. For this method the maximum number of values permitted outside control limits are:

| | average recovery | precision |
|---|---|---|
| DCS: | 1 | 1 |
| SCS: | 0 | |

9.6.2 Control Limits: Control limits can be found in the current CLP-SOW. Data will be accumulated and limits statistically adjusted over time.

9.6.3 Holding blanks will be run at a frequency determined by the Enseco QC Department. A holding blank consists of a VOA vial properly filled with purged reagent water. These will be analyzed at a frequency that reflects the maximum allowable holding time of a sample.

9.6.4 When trip blanks are generated by the laboratory to be shipped to a sampling site, a separate blank will be prepared on a weekly basis. This blank is analyzed for volatile organics and the data evaluated for contaminants, to ensure that contaminant-free trip blanks are sent into the field.

10. Calculations

10.1 Calculations required to reduce data

10.1.1 Concentration factor for sample (CF):

CF = NOMINAL SAMPLE SIZE/ACTUAL SAMPLE AMOUNT USED

The nominal sample size is 5 mL for water and 5 g for soil.

10.1.2 VOA SURROGATE CONCENTRATION FACTOR (SCF):

SCF = (Vol purged X 100) / amt spiked (total ng)

10.2 Calculations required to verify automated data reduction

10.2.1 Response factors

$$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

$A_x$ = Area of the characteristic ion for the compound to be measured.

$A_{is}$ = Area of the characteristic ion for the specific internal standard.

$C_{is}$ = Concentration of the internal standard (ng/uL).

$C_x$ = Concentration of the compound to be measured (ng/uL).

10.2.2 Average response factor

$$RF_{ave} = [(RF_{20})(2.5) + RF_{50} + (RF_{100}/2) + (RF_{150}/3) + (RF_{200}/4)]/5$$

Where $RF_x$ = Response factor of a compound in each of the calibration solutions.

10.2.3 Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

Where:

RSD = Relative Standard Deviation

SD = Standard Deviation of initial response factors (per compound)

Where: $\bar{S}D = \sum_{i=1}^{N} \frac{(x_i - x)^2}{N-1}$

$RF_{ave}$ = mean of initial response factors (per compound)

10.2.4 Percent Deviation from average response factor

$$\% \text{ Difference} = \frac{RF_I - RF_C}{RF_I} \times 100$$

Where:

$RF_I$ = average response factor from initial calibration.

$RF_C$ = response factor from current calibration check standard.

10.2.5 Percent Recovery

$$\text{Matrix Spike Percent Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from spiking mix

10.2.6 Relative percent difference

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where:

RPD = Relative Percent Difference
$D_1$ = First Sample Value
$D_2$ = Second Sample Value (duplicate)

10.2.7 Percent moisture

$$\frac{\text{wet (sample+dish)} - \text{dry (sample+dish)}}{\text{wet (sample+dish)}} \times 100 = \%\ \text{moisture}$$

10.2.8 Run factors: CF are multiplied by the actual T.C. concentration found in the quan list to get the concentration found in sample.

10.2.8.1 Aqueous:

$$CF = \frac{.005\ l}{XL}$$ where X = Volume of sample used.

10.2.8.2 Soil (Low Level):

$$CF = \frac{.005\ kg}{Xkg}$$ X = Weight of sample used.

Projects which require % moisture corrections

$$CF = \frac{0.005\ kg}{Xkg}\ \left(\frac{1-\%\ moisture}{100}\right)$$

10.2.8.3 Medium Level:

$$CF = \frac{.005\ l\ (10\ ml)}{(Xkg)(YmL)}$$ X=amount of sample used in extraction

Y=volume of MEOH from extr act used

Projects which require % moisture

$$CF = \frac{005\ l\ (10\ ml)}{Xkg(YmL)\left(1-\frac{\%moisture}{100}\right)}$$

11. Reporting requirements

11.1 Units: Aqueous samples will be reported in ug/L
Solid and sludge samples will be reported in ug/Kg or mg/kg on a wet weight basis, unless requested otherwise by the client.

11.2 Reporting limits: Test Specific

Reporting limits will be adjusted according to sample dilution.

11.3 Significant figures: All data <10 ug/L will be reported with one significant figure. All other data will be reported with two significant figures.

11.4 LDMS data entry

12. References

12.1 Method Source: Method 8240, SW-846 Third Edition, USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, September 1986.

12.2 Deviations from Source Methods

12.2.1 Deviations from the specified criteria for continuing calibration are allowed, provided the rationale is documented. Additionally, non-routine compounds may be analyzed for using a single, midpoint calibration.

12.2.2 An internal QA/QC program has been adopted by Enseco, Inc. This is described in Enseco's "Quality Assurance Program Plan for Environmental Chemical Monitoring." Specific elements of the QC program are described in SOP NO. M-EQA-002: "Internal QC Checks--Laboratory Performance QC." The laboratory control samples generated in this program are used to monitor method performance. Matrix spikes are performed at the request of the client.

12.2.3 The medium level method for soil referred to in this SOP is equivalent to the High-level method described in method 8240. The only exception is that this SOP uses a 5 g sample compared to the 4 g sample recommended in Method 8240.

Appendix A Tables of Analyte Lists to Include Analyte Name, Reporting Limit and Quantitation Ions

Table A-1 HSL List/Priority Pollutant List
Table A-2 Appendix IX List

## APPENDIX A

A-1. HSL/Priority Pollutant List

| Compound | QUAN Ion | Reporting Limits Low Level | Reporting Limits Medium Level |
|---|---|---|---|
| Chloromethane | 50 | 10 | 1000 |
| Bromomethane | 94 | 10 | 1000 |
| Dichlorodifluoromethane | 85 | 10 | 1000 |
| Vinyl Chloride | 62 | 10 | 1000 |
| Chloroethane | 64 | 10 | 1000 |
| Methylene Chloride | 84 | 5 (25)* | 500 |
| Acetone | 58 | 10 (25)* | 1000 |
| Carbon Disulfide | 76 | 5 | 500 |
| Trichlorofluoromethane | 101 | 5 | 500 |
| 1,1-Dichloroethene | 96 | 5 | 500 |
| 1,1-Dichloroethane | 63 | 5 | 500 |
| Trans-1,2-Dichloroethene | 96 | 5 | 500 |
| Chloroform | 83 | 5 | 500 |
| Trichloro-Trifluoro-Etha | 151 | 5 | 500 |
| 1,2-Dichloroethane | 62 | 5 | 500 |
| 1,4-Dioxane | 88 | 100 | 10,000 |
| 2-Butanone | 72 | 10 (25)* | 1000 |
| 1,1,1-Trichloroetha | 97 | 5 | 500 |
| Carbontetrachloride | 117 | 5 | 500 |
| Vinyl Acetate | 43 | 10 | 1000 |
| Bromodichloromethan | 83 | 5 | 500 |
| 1,2-Dichloropropane | 63 | 5 | 500 |
| Cis-1,3-Dichlorop | 75 | 5 | 500 |
| Trichloroethene | 130 | 5 | 500 |
| Dibromochloromethan | 129 | 5 | 500 |
| 1,1,2-Trichloroetha | 97 | 5 | 500 |
| Benzene | 78 | 5 | 500 |
| Trans-1,3-Dichlorop | 75 | 5 | 500 |
| 1,2-Dibromoethane | 107 | 5 | 500 |
| Bromoform | 173 | 10 | 1000 |
| 4-Methyl-2-Pentanon | 58 | 10 | 1000 |
| 2-Hexanone | 58 | 5 | 500 |
| Tetrachloroethene | 164 | 5 | 500 |
| 1,1,2,2-Tetrachloro | 83 | 5 | 500 |
| Toluene | 92 | 5 | 500 |
| Chlorobenzene | 112 | 5 | 500 |
| Ethylbenzene | 106 | 5 | 500 |
| Styrene | 104 | 5 | 500 |

| | | | |
|---|---|---|---|
| O-Xylene | 106 | 5 | 500 |
| M+P Xylenes | 106 | 5 | 500 |
| M-Dichlorobenzene | 146 | 5 | 500 |
| O-Dichlorobenzene | 146 | 5 | 500 |
| P-Dichlorobenzene | 146 | 5 | 500 |

* = Blank Correction Limit

## A-2. APPENDIX IX COMPOUNDS

| Compound | QUAN Ion | Reporting Limits Low Level | Reporting Limits Medium Level |
|---|---|---|---|
| Acetonitrile | 41 | 100 | 10,000 |
| Iodomethane | 142 | 5 | 500 |
| Acrolein | 56 | 100 | 10,000 |
| Acrylonitrile | 53 | 100 | 10,000 |
| Ethylcyanide | 54 | 5 * | 500 |
| 3-Chloropropene | 41 | 5 | 500 |
| Dibromomethane | 174 | 5 | 500 |
| Methacrylonitrile | 41 | 5 | 500 |
| Isobutyl Alcohol | 41 | 100 | 10,000 |
| 2-Chloro-1,3-Butadiene | 53 | 5 * | 500 |
| 1,1,1,2-Tetrachloroethan | 131 | 5 | 500 |
| 1,2,3-Trichloropropane | 75 | 5 | 500 |
| T-1,4-Dichloro-2-Butene | 53 | 5 | 500 |
| 1,2-Dibromo-3-Chloroprop | 157 | 5 | 500 |

* = Blank Correction Limit

Appendix B Tables of Solutions Used in this Method - To Include Analyte List, Concentration of Solution, and Amount to Use to Spike Samples or Inject

Table B-1 VOA-IS/SCS
Table B-2 VOA-DCS
Table B-3 Calibration Standard Solutions
Table B-4 Preparation of Standard for 5-Point Calibration

**Table B-1 VOA IS/SCS**

| Compounds | Standard Concentration ug/mL | Volume Added |
|---|---|---|
| Bromochloromethane | 50 | 5 ul in 5 mL |
| 1,4-Difluorobenzene | 50 | |
| Chlorobenzene-D5 | 50 | |
| Tuluene-D8 | 50 | |
| Bromofluorobenzene | 50 | |
| 1,2-Dichloroethane- | 50 | |

**Table B-2 VOA-DCS**

| Compounds | Units ug/mL | Volume Added |
|---|---|---|
| 1,1-Dichloroethene | 50 | 5 ul in 5 mL |
| Tichloroethene | 50 | |
| Chlorobenzene | 50 | |
| Toluene | 50 | |
| Benzene | 50 | |

**Table B-3 Calibration Standard Solutions**

**A. CS1**

| Compounds | Units ug/mL | Volume Added |
|---|---|---|
| Chloromethane | 50 | 5 ul in 5 mL |
| Bromomethane | 50 | |
| Vinyl Chloride | 50 | |
| Chloroethane | 50 | |
| Dichlorodifluoromethane | 50 | |
| Trichlorodrifluoromethane | 50 | |

**Table B-3 Calibration Standard Solutions**

**B. CS2**

| Compounds | Units ug/mL | Volume Added |
|---|---|---|
| Methylene Chloride | 50 | 5 ul in 5 mL |
| Acetone | 50 | |
| Carbon Disulfide | 50 | |
| Trichlorofluoromethane | 50 | |
| 1,1-Dichloroethene | 50 | |
| 1,1-Dichloroethane | 50 | |
| Trans-1,2-Dichloroethene | 50 | |
| Chloroform | 50 | |
| 1,2-Dichloroethane | 50 | |
| 2-Butanone | 50 | |
| 1,1,1-Trichloroethane | 50 | |
| Carbontetrachloride | 50 | |
| Vinyl Acetate | 50 | |
| Bromodichloromethane | 50 | |
| 1,2-Dichloropropane | 50 | |
| Cis-1,3-Dichloropropene | 70 * | |
| Trichloroethene | 50 | |
| Dibromochloromethane | 50 | |
| 1,1,2-Trichloroethane | 50 | |
| Benzene | 50 | |
| Trans-1,3-Dichloropropene | 29 * | |
| Bromoform | 50 | |
| 4-Methyl-2-Pentanone | 50 | |
| 2-Hexanone | 50 | |
| Tetrachloroethene | 50 | |
| 1,1,2,2-Tetrachloroethane | 50 | |
| Toluene | 50 | |
| Chlorobenzene | 50 | |
| Ethylbenzene | 50 | |
| Styrene | 50 | |
| O-Xylene | 50 | |
| M+P Xylenes | 50 | |
| M-Dichlorobenzene | 50 | |
| O-Dichlorobenzene | 50 | |
| P-Dichlorobenzene | 50 | |

* = Cis and Trans Isomers may vary over time requiring adjustments of Library Update amounts.

**Table B-3 Calibration Standard Solutions**

**C. API**

| Compounds | Units ug/mL | Volume Added |
|---|---|---|
| 1,4-Dioxane | 1000 | 5 ul in 5 mL |
| 1,2-Dibromoethane | 100 | |
| Isobutanol | 100 | |
| 1,1,1,2-Tetrachloroethane | 50 | |

**Table B-3 Calibration Standard Solutions**

**D. Appendix IX**

| Compounds | Units ug/mL | Volume Added |
|---|---|---|
| Acetonitrile | 50 | 5 ul in 5 mL |
| Iodomethane | 100 | |
| Acrolein | 50 | |
| Acrylonitrile | 50 | |
| Ethylcyanide | 50 | |
| 3-Chloropropene | 50 | |
| Dibromomethane | 50 | |
| Metharcrylonitrile | 50 | |
| Isobutyl Alcohol | 100 | |
| 2-Chloro-1,3-Butadiene | 50 | |
| 1,1,1,2-Tetrachloroethan | 50 | |
| 1,2,3-Trichloropropane | 50 | |
| T-1,4-Dichloro-2-Butene | 50 | |
| 1,2-Dibromo-3-Chloroprop | 100 | |

**Table B-4 Preparation of Standard for 5-Point Calibration Curve**

**A. Water and Low-Level Soil**

The amount of standard solutions added to the reagent water for each standard level will be identical for the water and low-level soil. The low-level soil standards will be heated to 40°C when purged.

| Standard Level | Vol of Purge Water | Vol of IS + Surrogate | Vol of Std | Vol of Gases |
|---|---|---|---|---|
| 50 ppb | 5 mL | 10 uL | 5 uL | 5 uL |
| 20 ppb | 5 mL | 10 uL | 2 uL | 2 uL |
| 100 ppb | 5 mL | 10 uL | 10 uL | 10 uL |
| 150 ppb | 5 mL | 10 uL | 15 uL | 15 uL |
| 200 ppb | 5 mL | 10 uL | 20 uL | 20 uL |

**B. Medium-Level Soil**

The standards for the medium level soil are prepared as above for water. The same curve is used for water and medium level soil. The volume of standard is the same. The standard levels are identical when the appropriate sample equivalent is taken into account. That is, for a medium-level soil analysis 0.05 g or 50 mg of soil is purged while for water 5 mL or .005 L is purged.

Appendix C

Table C-1

BFB KEY IONS AND ION ABUNDANCE CRITERIA

| Mass | Ion Abundance Criteria |
|---|---|
| 50 | 15.0 - 40.0 percent of the base peak |
| 75 | 30.0 - 60.0 percent of the base peak |
| 95 | base peak, 100 percent relative abundance |
| 96 | 5.0 - 9.0 percent of the base peak |
| 173 | less than 1.00 percent of the base peak |
| 174 | greater than 50.0 percent of the base peak |
| 175 | 5.0 - 9.0 percent of mass 174 |
| 176 | greater than 95.0 percent but less than 101.0 percent of mass 174 |
| 177 | 5.0 - 9.0 percent of mass 176 |

Table C-2

## CALIBRATION CHECK COMPOUNDS

**Initial Calibration:** The percent relative standard deviation for the each compound listed below should be <30%.

**Continuing Calibration:** The percent deviation from the average response factor determined in the initial calibration should be <30% for each compound listed below.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

## SYSTEM PERFORMANCE CHECK COMPOUNDS

**Initial Calibration:** The average response factor for each compound listed below should be >0.300, 0.250 for Bromoform.

**Continuing Calibration:** The response factor for each compound listed below should be >0.300, 0.250 for Bromoform.

Chloromethane
1,1-Dichloroethane
Bromoform
1,1,2,2-Tetrachloroethane
Chlorobenzene

**Appendix D**

TABLE D-1
SPIKE LEVELS FOR LABORATORY CONTROL SAMPLES

| DCS | Spike Level: Aqueous or Low-level Solid (ppb) | Spike Level: Medium-Level Solid* (ppb) |
|---|---|---|
| 1,1-Dichloroethene | 50 | 5,000 |
| Trichloroethene | 50 | 5,000 |
| Chlorobenzene | 50 | 5,000 |
| Toluene | 50 | 5,000 |
| Benzene | 50 | 5,000 |
| **SCS** | | |
| 1,2-Dichloroethane-$d_4$ | 50 | 5,000 |
| 4-Bromofluorobenze (BFB) | 50 | 5,000 |
| Toluene-$d_8$ | 50 | 5,000 |

Surrogates added to samples

Same as SCS

Blank components in LDMS (AUX lot)--minimum

Methylene chloride
Acetone
2-Butanone
4-Methyl-2-pentanone
2-Hexanone
Toluene

* Assumes 5g/10 mL method; 100 uL purged

TABLE D-2

CLP CONTROL LIMITS

**DCS**

| | AQUEOUS | | SOLID | |
|---|---|---|---|---|
| | DCS | RPD | DCS | RPD |
| 1,1-Dichloroethene | 61-145 | 14 | 59-172 | 22 |
| Trichloroethene | 71-120 | 14 | 62-137 | 24 |
| Chlorobenzene | 75-130 | 13 | 60-133 | 21 |
| Toluene | 76-125 | 13 | 59-139 | 21 |
| Benzene | 76-127 | 11 | 66-142 | 21 |

**SCS**

| | | |
|---|---|---|
| 1,2-Dichloroethane-$d_4$ | 76-114 | 70-121 |
| 4-Bromofluorobenze (BFB) | 86-115 | 74-121 |
| Toluene-$d_8$ | 88-110 | 81-117 |

## APPENDIX E ANALYTICAL TEST REFERENCES

Reference 1. HSL CLP - Contract Lab Program

Reference 2. HSL 624

Reference 3. Refinery

Reference 4. Appendix IX

Reference 5. Appendix VIII

Reference 6. USGS - 624 United States Geological Survey

Reference 7. USGS - 524 United States Geological Survey

Reference 8. AFIR

Reference 9. TCLP - Toxicity Characteristic Leaching Procedure

Reference 10. LRR - Land Restriction Rule

CLP HSL VOA

SOP No.: LM-RMA-3001 VOA by GC/MS ORIGINAL Effective Date: 12/9/87
Reference 1

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

## CLP HSL VOA

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         OWA In GC load parameter 2

      2. STD, QC, Samples

         All instruments automatically loaded by ACQR program except OWA
         OWA - In GC load parameter 3

   C. Standard: in 5 mL $H_2O$

      10 ul IS/SCS 25 ug/mL
      5 ul MS-VOA-CS1
      5 ul MS-VOA-CS2
      5 ul MS-VOA-API

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples: *use CLP Header Format
      3. AUTOSP - Auto Run

2. QC

A. QC to be Run: BFB/STD (50 ppb), A MATRIX SPIKE AND MATRIX SPIKE DUP must be performed every 10 samples. Look for sample designation on backlog. (Spikes are required for each matrix type. i.e., Waters, low level soil, medium level soil.)

B. Acceptance Criteria: EPA CRITERIA for all STDS, BLANKS AND surrogate recoveries (See EPA CLP criteria)

C. Project Specific QC:

* EPA = 12 HOUR CURVE/ALL compounds must be below detection LIMIT in blank except common lab solvent ($MECl_2$,2-butamone and Acetone can be 5 X DL)

3. Data

A. Processing Procedures:

1. Library V1,V2,V3

2. TC Filename,, VOACLP for STD, Blanks and samples

B. Data Package:

1. Data is captured on floppy disc using:

a. Initial Calibration uses ICAP
b. Ongoing Calibration, Blanks, Samples uses SCAP

2. Cover Page (see cover sheet that is attached)

a. Run factor calculation must use % moistures

b. Use SUM1 program

3. Chromatogram must be stamped.

4. DUAL Spectra and STD spectra must be included for all HSl compounds in BLANKS, samples and matrix spikes excluding the spike compounds themselves.

5. No more than 10 TIC's will be pulled for any one sample, if there are more, pull only the 10 largest. These must be QUANTITATED and captured using TIC programs.

6. A BFB summary sheet must be filled out and BFB spectra, mass list and instrument summary included.

7. Anomaly sheet

8. Complete copy of standard, updated QUAN summary, Initial and Ongoing Calibration XCALIB summary

9. Complete copy of blank data

VOA HSL/624

SOP No.: LM-RMA-3001 VOA by GC/MS ORIGINAL Effective Date: 12/9/87
Reference 2

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

VOA HSL/624

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         OWA - In GC load parameter 2

      2. STD, QC, Samples All instruments automatically loaded by ACQR program except OWA
         OWA - In GC load parameter 3

   C. Standard: in 5 mL $H_2O$

      10 ul IS/SCS 25 ug/mL
      5 ul MS-VOA-CS1
      5 ul MS-VOA-CS2
      5 ul MS-VOA-API

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples
      3. AUTOSP - Auto Run

2. QC

   A. Required DCS and SCS analysis. SCS must meet EPA Blank target compound limits.
   B. Acceptance Criteria: ENSECO QC Criteria for DCS and SCS

C. Project Specific QC: Check Backlog (For project specific QC). Do not rerun if surrogates are out.
D. 24 hr Calibration unless specified other wise by client.

3. Data

A. Processing Procedures:
(libraries, library lists, compound list)
Note: please assoc. compound list with its libraries and processing routines.

1. Library V1,V2,V3
2. TC Filename,, VOASTD

B. Data Package

1. Cover Page

a. Run factor calculation: Based on Wet Weight (no % moisture required)
b. Specific cover page using SUM1

2. DUAL spectra

3. Chromatogram just marked in pen

4. Summary SCS and DCS sheets

5. Anomaly sheet

6. Blank summary: Use SCS SUM1 summary

**VOA Refinery**

SOP No.: **LM-RMA-3001 VOA by GC/MS** ORIGINAL Effective Date: 12/9/87
**Reference 3**

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

**VOA REFINERY**

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         OWA - In GC load parameter 2

      2. STD, QC, Samples All instruments automatically loaded by ACQR program except OWA
         OWA - In GC load parameter 3

   C. Standard: in 5 mL $H_2O$

      10 ul IS/SCS 25 ug/mL
      5 ul MS-VOA-CS1
      5 ul MS-VOA-CS2
      5 ul MS-VOA-API

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples
      3. AUTOSP - Auto Run

2. QC

   A. Samples can be run with CCC or SPCC out. 24 hr BFB calibration
   B. QC to be Run: "Enseco" Laboratory QC - appropriate DCS & SCS
   C. Acceptance Criteria: lab criteria
   D. Project Specific QC: Check Backlog

3. Data

A. Processing Procedures:

(libraries, library lists, compound list)
Note: please assoc. compound list with its libraries and processing routines.

Process under
1. Library V1,V2,V3
2. STD: TC File,, VOASTD
3. Samples: TC File,, VOAREF

Compound list:

| | |
|---|---|
| Carbon Disulfide | Benzene |
| O & P Xylene | Chloroform |
| 1,2-Dibromoethane | M. Xylene |
| 1,2-Dichloroethane | Toluene |
| 2-Butanone | Chlorobenzene |
| 1,4-Dioxane | ethylbenzane |
| styrene | |

B. Data Package:

1. Cover Page

   a. Run factor calculation based on wet weight (NO % moisture required)
   b. Specific cover page: use SUM1 program

2. Chromatogram does not need stamping

3. Dual Spectra

4. Anomaly sheets

5. QC sheets - SCS and DCS summary

6. Blank summary: Use SCS SUM1 summary

VOA Appendix IX

SOP No.: LM-RMA-3001 VOA by GC/MS ORIGINAL Effective Date: 12/9/87
Reference 4

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

## VOA APPENDIX IX

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         OWA - In GC load parameter 2

      2. STD, QC, Samples All instruments automatically loaded by ACQR program except OWA
         OWA - In GC load parameter 3

   C. Standard: in 5 mL $H_2O$

      1. 10 ul IS/SCS 25 ug/mL
         5 ul MS-VOA-CS1
         5 ul MS-VOA-CS2
         5 ul MS-VOA-API

      2. 10 ul IS/SCS 25 ug/mL
         5 ul MS-AP9-CS
         * Second standard must be a separate run. Both standards must be updated in QUAN

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples
      3. AUTOSP - Auto Run

2. QC

   A. QC to be Run: ENSECO SCS or DCS's
   B. Acceptance Criteria: ENSECO QC criteria
   C. Project Specific QC: See Backlog for additional QC runs
   D. 24 hr daily calibration

3. Data

   A. Processing Procedures:
      (libraries, library lists, compound list)

      1. Library V1,V2,V3

      2. a. To process AP9 Standard use: TC filename,, VOAAP9STD
         b. To process regular STD use: TC filename,, VOASTD

      3. To process SCS and samples: TC filename,, VOAAP9

   B. Data Package:

      1. Cover Page

         a. Run factor calculation: use wet weight (NO% moistures)
         b. Use SUM1

      2. Chromatogram - marked in Pen

      3. Dual spectra

      4. SCS and DCS summary sheets

      5. Anomaly sheet

      6. Blank Summary Sheet: Use SCS SUM1 summary

VOA Appendix VIII

SOP No.: LM-RMA-3001 VOA by GC/MS ORIGINAL Effective Date: 12/9/87
Reference 5

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

## VOA APPENDIX VIII

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         a. OWA In GC Load parameter 2

      2. STD, QC, Samples All instruments automatically loaded by ACQR program except OWA

   C. Standard: in 5 mL $H_2O$

      1. 10 ul IS/SCS 25 ug/mL
         5 ul MS-VOA-CS1
         5 ul MS-VOA-CS2
         5 ul MS-VOA-API

      2. 10 ul IS/SCS 25 ug/mL
         5 ul MS-AP9-CS
         * Second standard must be a separate run. Both standards need to be updated in QUAN.

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples
      3. AUTOSP - Auto Run

2. QC

   A. QC to be Run: Normal SCS or DCS's as necessary
   B. Acceptance Criteria: Normal QC criteria
   C. Project Specific QC: See Backlog for possible additional QC requested by client
   D. 24 hr daily calibration

3. Data

A. Processing Procedures:
(libraries, library lists, compound list)

1. Library V1,V2,V3

2. a. HSL standard use: TC Filename,, VOASTD
b. AP9 Standard: TC Filename,, VOAAP9STD

3. Samples and SCS: TC Filename,, VOAAP9 and search for the following compounds: Ethylene oxide and Malononitrile. See attached program "eight" for automated search

B. Data Package:

1. Cover Page

a. Run factor calculation: use wet weight (NO % moisture)
b. Use SUM1 and add compounds searched for even if they are not found.

2. Dual spectra

3. Chromatogram - marked in Pen

4. SCS and DCS summary

5. Blank summary: Use SCS SUM1 summary

6. Hard copies of additional searches. Include dual spectra for any searches that have matching mass chromatogram peaks.

7. Anomaly sheet

```
Trace of procedure EIGHT
    * SETN EIGHT.NL
    * EIGHT1
    *
    SETN EIGHT.NL
    EIGHT1
       * GETN
       * CHRO(I;D1,680;42;43;44;H;E)-ethylene Oxide
       * CHRO(I;D1,680;66;H;E)-Malononitrile
       * LOOP
       *
       GETN
       CHRO (I;D1,680;42;43;44;H;E)
       CHRO (I;D1,680,66;H;E)
       LOOP
```

1. Edit EIGHT.NL namelist to contain files to be searched.

2. In IDOS edit CHRO statements in EIGHT1 program to search desired chromatogram scan range.

3. Type EIGHT for search hardcopy.

4. Check possible matches with enclosed spectra.

USGS - 624

SOP No.: LM-RMA-3001 VOA by GC/MS ORIGINAL Effective Date: 12/9/87
Reference 6

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

USGS - 624

1. Instrumentation

A. FC43 * * use instrument letter, follow commands

B. GC Descriptors

1. BFB All instruments automatically loaded by ROAD program except OWA
OWA In GC load parameter 2

2. STD, QC, Samples

All instruments automatically loaded by ACQR program except OWA
OWA - In GC load parameter 3

C. Standard: in 5 mL $H_2O$

* 10 ul USGS IS/SCS 25 ug/mL
8 ul MS-VOA-CS1
8 ul MS-VOA-CS2
8 ul MS-VOA-API
* May need to make dilution from stock

** USGS requires separate 3 pt. Initial Calibration. The 3 pt. standard levels are 20,80,200 ppb. Use XCALIB program with USGSCALIB.

D. Instrument set up.

1. ROAD - BFB
2. ACQR - STD, QC, Samples
3. AUTOSP - Auto Run

2. QC

A. QC to be Run: Blank & Blank Spike (100 ppb spike compound level) every 12 hours
B. Acceptance Criteria: Surrogates between 70-130% Recovery; Spike compounds 70-130% Recovery
C. Project Specific QC: Check backlog for any additional QC. No ENSECO QC required.
D. 12 hr daily calibration

3. Data

A. Processing Procedures:
(libraries, library lists, compound list)
Note: please assoc. compound list with its libraries and processing routines

1. Library V6

2. Process STD and samples: TC filename,, VOAUSGS

B. Data Package:

1. Cover Page (see cover sheet that is attached)

a. Run factor calculation same as industrial
b. Specific cover page(s) to use supplied by USGS. See USGS forms file.

2. Dual spectra

3. Chromatogram stamped and complete

4. Calibration information

   a. Initial Calibration raw data for 3 pt.
   b. Daily calibration raw data including new QSORT
   c. Complete USGS calibration forms

5. BFB: Fill out USGS BFB Summary Sheet and include BFB mass list, spectra and instrument check sheet hardcopy.

6. Blank summary sheet and blank raw data

7. Blank pike summary sheet and spike raw data.

* NOTE: USGS forms can be found in USGS forms file.

USGS - 524

SOP No.: LM-RMA-3001 VOA by GC/MS ORIGINAL Effective Date: 12/9/87
Reference 7

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

USGS - 524

1. Instrumentation

A. FC43 * * use instrument letter, follow commands

B. GC Descriptors

1. BFB All instruments automatically loaded by ROAD program except OWA
OWA In GC load parameter 2

2. STD, QC, Samples

All instruments automatically loaded by ACQR program except OWA
OWA - In GC load parameter 3

C. Standard: in 25 mL $H_2O$

* 10 ul USGS IS/SCS 25 ug/mL
8 ul MS-VOA-CS1
8 ul MS-VOA-CS2
8 ul MS-VOA-API
* May need to make dilution from stock

USGS low level detection limit test requires separate 3 pt. initial calibration. STD levels for 3 pt. are 2, 10 and 30 ppb. Use XCALIB program and USGSCALIB input file.

D. Instrument set up.

1. ROAD - BFB
2. ACQR - STD, QC, Samples
3. AUTOSP - Auto Run

2. QC

A. QC to be Run: Blank & Blank Spike every 12 hours
B. Acceptance Criteria: Surrogates between 70-130% Recovery; Spike compounds 70-130% Recovery
C. Project Specific QC: Check backlog for any additional QC. No ENSECO QC required.
D. 12 hr daily calibration

3. Data

A. Processing Procedures:
(libraries, library lists, compound list)
Note: please assoc. compound list with its libraries and processing routines

1. Library V6

2. Process STD and samples: TC filename,, VOAUSGS

B. Data Package:

1. Cover Page (see cover sheet that is attached)

a. Run factor calculation uses wet weight
b. Specific cover page(s) to use supplied by USGS. See USGS forms file.

2. Dual spectra

3. Chromatogram stamped and complete

4. BFB: Fill out USGS BFB Summary Sheet and include BFB mass list, spectra and instrument check sheet hardcopy.

5. Blank summary sheet

6. Calibration information

   a. Initial Calibration raw data for 3 pt.
   b. Daily calibration raw data including new QSORT
   c. Complete USGS calibration forms

7. Blank pike summary form

* NOTE: USGS forms can be found in USGS forms file.

**AFIR**

SOP No.: **LM-RMA-3001 VOA by GC/MS** ORIGINAL Effective Date: 12/9/87
**Reference 8**

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

**AFIR**

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         OWA - In GC load parameter 2

      2. STD, QC, Samples All instruments automatically loaded by ACQR program except OWA
         OWA - In GC load parameter 3

   C. Standard: in 5 mL $H_2O$

      10 ul IS/SCS 25 ug/mL
      5 ul MS-VOA-CS1
      5 ul MS-VOA-CS2
      5 ul MS-VOA-API
      * 5 ul BV-STD
      * Projects for Black and Veatch require additional compounds. The BV-STD can be run along with the HSL standard or separately as a second run.

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples
      3. AUTOSP - Auto Run
         NOTE: For AFIR soils 1.0 gm is maximum amount for low level analysis.

2. QC

   A. Must hit CLP/EPA Continuing Calibration criteria

   B. Run normal ENSECO SCS and DCS QC

   C. 12 hr calibration curve for all AFIR except USGS which can be 24 hr.

   D. Check Backlog to see if additional QC has been requested by client

3. Data

   A. Processing Procedures:

      1. Library V1,V2,V3

      2. Standards, SCS and samples: TC filename,, VOASTD

      3. Black and Veatch:

         a. Combined Standard, SCS and samples: TC filename,, BLACK
         b. Separate BV-STD use: TC filename,, BLACKSTD

   B. Data Package:

      1. Cover Page:

         a. Run factor calculations based on wet weight
         b. Use SUM1 program

      2. Chromatogram stamped and completed

      3. Dual Spectra

      4. SCS and DCS summary

      5. Blank summary: Use SCS SUM1 summary

      6. BFB - copies of mass list, spectra and hard copy of summary

7. Ongoing XCALIB page

8. Anomaly sheet

VOA TCLP

SOP No.: LM-RMA-3001 VOA by GC/MS ORIGINAL Effective Date: 12/9/87
Reference 9

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

## VOA TCLP

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         OWA In GC load parameter 2

      2. STD, QC, Samples All instruments automatically loaded by ACQR program except OWA
         OWA - In GC load parameter 3

   C. Standard: in 5 mL $H_2O$

      1. 10 ul IS/SCS 25 ug/mL
         5 ul MS-VOA-CS1
         5 ul MS-VOA-CS2
         5 ul MS-VOA-API

      2. 10 ul IS/SCS 25 ug/mL
         5 ul MS-AP9-CS
         * Second standard must be a separate run. Both STD must be updated in QUAN.

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples
      3. AUTOSP - Auto Run

E. 24 hr daily calibration

NOTE: 1mL/5mL is the most sample that needs to be run. If screen says 100% clean use 1mL.

2. QC

A. QC to be Run: DCS/SCS
B. Acceptance Criteria: Enseco QC Program
C. Project Specific QC: usually requires a TCLP Blank. Check for possible blank spike.

3. Data

A. Processing Procedures:
(libraries, library lists, compound list)
Note: please assoc. compound list with its libraries and processing routines. (will depend on Elution Times varying for each instrument)

1. Library V1,V2,V3

2. Process STDS

a. For the HSL STD use: TC Filename,, VOASTD
b. For the AP9 STD use: TC Filename,, VOAAP9STD

3. Process samples and blank

a. Tc Filename,, VOATCLP

B. Data Package:

1. Cover Page:
a. Run factor calculation: Standard Industrial $H_2O$
b. Specific cover page use SUM1: Add above compounds

2. Dual Spectra

3. Chromatogram: marked in pen

4. SCS and DCS water QC summary forms

5. Anomaly sheet

6. Blank summary

**VOA LRR**

SOP No.: **LM-RMA-3001 VOA by GC/MS** ORIGINAL Effective Date: 12/9/87
**Reference 10**

Supersedes:

The following analysis of SOP contains information specific to particular compound lists and LIMS tests. For complete laboratory analytical methodology, please refer to VOLATILE ORGANICS ANALYSIS BY GC/MS.

**VOA LRR**

1. Instrumentation

   A. FC43 * * use instrument letter, follow commands

   B. GC Descriptors

      1. BFB All instruments automatically loaded by ROAD program except OWA
         OWA In GC load parameter 2

      2. STD, QC, Samples All instruments automatically loaded by ACQR program except OWA
         OWA - In GC load parameter 3

   C. Standard: in 5 mL $H_2O$

      10 ul IS/SCS 25 ug/mL
      5 ul MS-VOA-CS1
      5 ul MS-VOA-CS2
      5 ul MS-VOA-API

   D. Instrument set up.

      1. ROAD - BFB
      2. ACQR - STD, QC, Samples
      3. AUTOSP - Auto Run

   E. 24 hr daily calibration

      NOTE: 1mL/5mL is the most sample that needs to be run. If screen says 100% clean use 1mL.

2. QC

   A. QC to be Run: Normal SCS or DCS's as necessary
   B. Acceptance Criteria: Normal limits
   C. Project Specific QC: check backlog for additional QC if needed
   D. 24 hr daily calibration

3. Data

   A. Processing Procedures:
      (libraries, library lists, compound list)
      Note: please assoc. compound list with its libraries and processing routines.

      1. Library V1,V2,V3

      2. STD and Samples use VOASTD. Note not all compounds in VOASTD Library are required. See attached compound list.

      3. In addition, search for the following compounds:

         Ethylether
         Ethylacetate
         Isobutanol
         Cyclohexanone
         n-Butylalcohol

         See attached program "LR" and mass spectra for above compounds

   B. Data Package:

      1. Cover Page:
         a. Run factor calculation: wet weight (NO % moistures)
         b. Use HSL cover sheet from SUM1 & add names to it for compounds searched

      2. Chromatogram: marked in pen

      3. Dual spectra

4. SCS and DCS summary forms

5. Blank summary

6. Hardcopies of additional searches. Include dual spectra for any searches which have matching mass CHRO peaks

7. Anomaly sheet

STANDARD
OPERATING
PROCEDURE

| Subject or Title: | | Page 1 of 17 |
|---|---|---|
| **Halogenated Volatile Organics - Purgeable Halocarbons (EPA Method 601/8010)** | | |
| SOP No.: LM-RMA-4008 | Revision No.: 1.0 | Effective Date: January 31, 1989 |
| Supersedes: Original | | |

1. Scope and Application.

   1.1 Analyte - The analytes that can be determined by this method are listed in Table 1.

   1.2 Detection limits - The instrument and method detection limits are listed in Table 2.

   1.3 Applicable matrices - Water, solid/waste

   1.4 Dynamic range - The dynamic range under normal operating conditions is approximately $10^3$.

   1.5 Approximate analytical time - 1 hr/sample.

2. Summary of Method.

   2.1 This is a purge and trap gas chromatographic method applicable to the determination of the halocarbons listed in Table 1. Helium gas is bubbled through a 5 mL water sample, contained in a specially-designed purging chamber, at ambient temperature. For solid/waste samples, a methanol extract is purged in 5 mL of reagent water. the halocarbons are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with helium gas to desorb the analytes onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the analytes which are then detected with a halide specific detector.

| Prepared by: Michael Hoffman | Date: 1/31/89 |
|---|---|
| Management Approval: Michael Hoffman | Date: 2/1/89 |
| QA Officer Approval: Gary Cal | Date: 2/9/89 |

3. Comments

3.1 Interferences

3.1.1 Interferences may result from impurities in the purge gas.

3.1.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage.

3.1.3 Contamination by carry-over can occur when high level and low level samples are sequentially analyzed.

3.1.4 The analytical system must be demonstrated to be free from contamination by running laboratory reagent blanks under the conditions of the analysis.

3.1.5 The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.1.6 To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

4. Safety Issues.

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Sample Collection, Preservation, Containers, and Holding Times.

5.1 All samples must be iced or refrigerated from the time of collection until extraction. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm $Cl_2$) to the empty sample bottle just prior to shipping to the sampling site. USEPA methods 330.4 and 330.5 may be used for measurement of residual chlorine. Field test kits are available for this purpose.

5.2 Sample vials distributed by RMAL contain 200 uL of 50% HCL as a preservative for aromatic analytes.

5.3 Grab samples are collected in glass containers having total volume of at least 25 mL. The sample bottle is filled just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for one minute. maintain the hermetic seal on the sample bottle until time of analysis.

5.4 All samples must be analyzed within 14 days of collection.

6. Apparatus.

6.1 Sampling equipment for discrete sampling.

6.2 Vial - 25 mL capacity or larger, equipped with a screw cap with hole in center (Pierce #13075 or equivalent). Detergent wash, rinse cap with tap and distilled water, and dry at 105°C before use.

6.3 Septum - Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105°C for one hour before use.

6.4 Purge and trap device - Tekmar Model ALS 4000 Automatic Lab Sampler equipped with ten sample purge cells designed to accept 5 mL samples, or OI Corporation Model 4460A Purge and Trap concentrator equipped with autosampler and standard injection module.

6.4.1 Trap - Supelco Volatile Purge Trap (#2-0294) or equivalent.
1 cm 3% SP-2100 on 60/80 mesh Chromosorb WAW.
7.7 cm 60/80 Tenax TA (Diphenylene oxide polymer).
7.7 cm 35/60 Silica Gel GD-15.
7.7 cm 20/50 Charcoal.

6.4.2 Purge at 30°C for 11 min, 30 cc/min; desorb at 180°C for 4 min, 40 cc/min; bake at 225°C for 12 min; transfer line 125°C; 6-port valve 125°C.

6.5 Gas Chromatograph - Gas chromatograph equipped with an electrolytic conductivity detector interfaced with a Nelson data system.

6.5.1 Column - 8 ft x 1/3 in od x 2 mm in glass column packed with 1% SP-1000 on Carbopack B, 60/80 mesh.

6.5.2 Column temperature and gas flow - Initial 45°C, 3 min; program rate 8°C/min; final 225°C, 25 min; carrier flow rate 40 cc/min.

6.5.3 Alternate Column - 105 m RTx-Volatiles Column made by Restek Corporation.
Conditions: Initial 35°C, 10 min.: program rate 4°C/min.: final 190°C, 15 min., carrier flow 10 cc/min.

6.5.4 Detector - OI Corporation electrolytic conductivity detector.

7. Reagents and Standards

7.1 Reagent water - Carbon-filtered water purged with helium prior to use.

7.2 Methyl alcohol - Purge and Trap quality or equivalent.

7.3 Stock standard solutions - Prepared by QA/QC standards preparation staff at a concentration of 50 ug/mL. Prepared from pure standard materials or purchased as certified solutions.

7.3.1 The standards are freshly prepared every two weeks and are stored with minimal headspace in a freezer when not in use. The stock standards are diluted with water to appropriate concentrations by analyst when preparing calibration standards.

7.4 Laboratory control sample (LCS) standard - Prepared by QC/QA standards preparation staff. Prepared in methanol from EPA stock standards at the following concentrations:

| | |
|---|---|
| 1,1-Dichloroethane | 250 ug/mL |
| Chloroform | 250 ug/mL |
| Dichlorobromomethane | 500 ug/mL |
| Trichloroethene | 250 ug/mL |
| Chlorobenzene | 250 ug/mL |

Diluted by analyst to 5 ug/mL in methanol for analysis as LCS check standard. A 5.0 uL aliquot of the standard is added to 5.0 mL of reagent water for a 5.0 ug/L working standard.

7.5 Surrogate control (SCS) standard - Prepared by QC/QA standards preparation staff. Standard contains bromochloromethane at 30 ug/mL in methanol, and is used as a surrogate. This is diluted 1:1000 in all samples and standards analyzed.

7.6 Internal Standard - Prepared by QC/QA standard preparation staff. Standard contains 1-chloro-4-fluorobenzene at 30 ug/mL in methanol. This is diluted 1:1000 in all standards and samples analyzed.

8. Procedure.

8.1 Preparation - No sample preparation is required for water samples. A 5.0 mL sample is purged directly. For solid/waste samples, a 5 g sample is extracted with 10 mL methanol. A 100 ul aliquot of the methanol extract is added to 5.0 mL reagent water and purged.

8.2 Calibration for Instruments With Packed Columns - Prepare a secondary standard at 2.0 ug/mL by adding 72 mL of the CS1 and 72 mL of the CS2 to 1656 mL of methanol. Prepare calibration standards at concentration levels of 60, 20, 5.0, 1.0 and 0.50 ug/L by adding the following aliquots of the 50 ug/mL and 2.0 ug/mL standards to 5.0 mL of reagent water in the purge cell:

60 ug/L - 6.0 uL of 50 ug/mL standard
20 ug/L - 2.0 uL of 50 ug/mL standard
5.0 ug/L - 12.5 uL of 2.0 ug/mL standard
1.0 ug/L - 2.5 uL of 2.0 ug/mL standard
0.5 ug/L - 1.25 uL of 2.0 ug/mL standard

8.2.1 Add 5.0 uL of the 30 ug/mL SCS and internal standard to each calibration standard and attach purge cell to autosampler for analysis.

8.3 Calibration for Megabore Instruments - Prepare a secondary standard at 20 ug/mL by adding 720 uL of CS1 and 720 uL of CS2 to 360 uL of methanol. Prepare calibration standards at concentrations of 20, 5, 2, 1, 0.5 ug/L by adding the following aliquots of the 20 ug/mL standard to 50 mL of reagent water in a 50 mL volumetric flask:

20 ug/L - 50 uL
5 ug/L - 12.5 uL
2 ug/L - 5 uL
1 ug/L - 2.5 uL
0.5 ug/L - 1.25 uL

8.3.1 SCS and I.S. solutions are added automatically by the OI Standard Injection Module.

8.4 Prepare a calibration curve using a point-to-point type curve.

8.5 Analysis

8.5.1 Analysis on Packed Column Instrument - Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5 mL syringe and attach

a closed syringe valve. Open the sample bottle and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Inject the sample into the purge cell, add 5.0 uL of the 30 ug/mL SCS standard and internal standard (place the needle of the microliter syringe below the surface of the sample when adding standard), and connect to purge line on the first position of the Tekmar autosampler. Repeat procedure with next sample and connect to the purge line on the next position of the autosampler. When all ten positions are filled, start autosampler.

8.5.2 Analysis on Megabore Instrument - For clean water samples place the 40 mL VOA vial directly in the autosampler. For soil extracts 1 mL is diluted in a 50 mL volumetric flask with reagent $H_2O$ and transferred to a clean, baked 40 mL VOA vial. For water samples with high levels of contamination, appropriate dilutions can be made in 50 mL volumetric flasks and transferred to clean, baked 40 mL VOA vials. The I.S./SCS standard is added by the Standard Injection Module.

9. QA/QC Requirements.

9.1 QC samples - The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control.

Prior to the analysis of samples each day, analyze a 20 ug/L calibration check standard, an SCS standard, and an LCS standard in duplicate.

An additional 20 ug/L calibration check standard must be analyzed at the end of each run to verify the analytical system stability. If this standard is unacceptable, all samples analyzed after the last acceptable standard must be reanalyzed.

9.2 Acceptance criteria (precision and accuracy)

9.2.1 The acceptance criteria for each of the analytes in the LCS and the SCS are listed in Table 3.

9.2.2 The acceptance criteria for the 20 ug/L calibration check standard is listed in Table 4.

9.2.3 The acceptance criteria for the internal standard area in the samples is 70-160% of the I.S. area in the 20 ug/L calibration check standard. Reanalysis is required if it is out of this range. If reanalysis produces similar results the data is reported with an anomaly stating this.

9.3 Corrective action required (reference current QC manual).

10. Calculations

Tabulate peak area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using the following equation:

$$RF = \frac{(A_s) \times (C_{is})}{(A_{is}) \times (C_s)}$$

where:

$A_s$ = Response for the parameter to be measured
$A_{is}$ = Response for the internal standard.
$C_{is}$ = Concentration of the internal standard
$C_s$ = Concentration of the parameter to be measured.

If the RF over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$ vs RF.

To determine the concentration of individual compounds in a sample, use the following equation.

$$\text{Concentration (ug/L)} = \frac{(A_s) \times (C_s)}{(A_{is}) \times (RF)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$C_{is}$ = Concentration of the internal standard.

11. Reporting

11.1 Reporting units - Water samples ug/L; solid samples ug/kg; waste samples mg/kg.

11.2 Reporting limits - Reporting limits are listed in Table 5 for water samples, and Table 6 for solid/waste samples. See Appendix for reporting limits for specific contract work.

11.3 Significant figures - Report sample results to two significant figures and QC resuLts to three significant figures.

11.4 Data entry - After analysis and data reduction have been completed, all sample and QC/QA analytical results are entered into LIMS for subsequent review.

12. References

12.1 Method source -

"EPA Method 601 - Purgeable Halocarbons, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, U.S. Environmental Protection Agency; Federal Register Vol. 49, No. 209, October 26, 1984.

"Methods 330.4 (Titrimetric, DPA-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45266, March 1979.

12.2 Deviations from source method and rationale - Methods used is essentially EPA Method 601.

**TABLE 1. HALOGENATED VOLATILE ORGANICS**

| Compound | CAS Number |
|---|---|
| Chloromethane | 74-87-3 |
| Bromomethane | 74-83-9 |
| Vinyl Chloride | 75-01-4 |
| Dichlorodifluoromethane | 75-71-8 |
| Chloroethane | 75-00-3 |
| Methylene Chloride | 75-09-2 |
| Trichlorofluoromethane | 75-69-4 |
| 1,1-Dichloroethene | 75-35-4 |
| 1,1-Dichloroethane | 75-34-3 |
| t-1,2-Dichloroethene | 156-60-5 |
| Chloroform | 67-66-3 |
| 1,1,2-Trichloro-trifluoroethane | 76-13-1 |
| 1,2-Dichloroethane | 107-06-2 |
| 1,1,1-Trichloroethane | 71-55-6 |
| Carbon Tetrachloride | 56-23-5 |
| Bromodichloromethane | 75-27-4 |
| 1,2-Dichloropropane | 78-87-5 |
| t-1,3-Dichloropropene | 10061-02-6 |
| Trichloroethylene | 79-1-6 |
| 1,1,2-Trichloroethane | 79-00-5 |
| Dibromochloromethane | 124-48-1 |
| cis-1,3-Dichloropropene | 10061-01-5 |
| 1,2-Dibromoethane (EDB) | 106-93-4 |
| 2-Chloroethylvinyl ether | 131-11-3 |
| Bromoform | 75-25-2 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 |
| Tetrachloroethene | 127-18-4 |
| Chlorobenzene | 108-90-7 |

**TABLE 2. HALOGENATED VOLATILE ORGANICS DETECTION LIMITS**

| Compound | Detection Limits, ug/L* |
|---|---|
| Chloromethane | 0.17 |
| Bromomethane | 0.49 |
| Vinyl Chloride | 0.50 |
| Freon 12 | 0.50 |
| Chloroethane | 0.25 |
| Methylene Chloride | 0.36 |
| Trichlorofluoromethane | 0.36 |
| 1,1-Dichloroethene | 0.45 |
| 1,1-Dichloroethane | 0.30 |
| t-1,2-Dichloroethene | 0.58 |
| Chloroform | 0.51 |
| 1,1,2-Trichloro-trifluoroethane | ** |
| 1,2-Dichloroethane | 0.51 |
| 1,1,1-Trichloroethane | 0.63 |
| Carbon Tetrachloride | 0.38 |
| Bromodichloromethane | 0.24 |
| 1,2-Dichloropropane | 0.41 |
| t-1,3-Dichloropropene | 0.35 |
| Trichloroethylene | 0.45 |
| 1,1,2-Trichloroethane | 1.0 |
| Dibromochloromethane | 1.0 |
| cis-1,3-Dichloropropene | 1.0 |
| 1,2-Dibromoethane (EDB) | ** |
| 2-Chloroethylvinyl ether | 0.26 |
| Bromoform | 0.52 |
| 1,1,2,2-Tetrachloroethane | 0.95 |
| Tetrachloroethene | 0.95 |
| Chlorobenzene | 0.35 |

* Calculated detection limits - Three times the standard deviation of 7-8 replicate analyses. Probably not achievable under normal operating conditions. Method and instrument detection limits are estimated to be similar because no sample preparation is involved.

** Not included in EPA Method 601.

**TABLE 3. VOLATILE HALOGENATED ORGANICS ACCEPTANCE CRITERIA FOR LCS AND SCS**

| Analytes | Spike Concentration | | Control Limits | |
|---|---|---|---|---|
| LCS | Water, ug/L | Soil, mg/kg | % Recovery | Rel. % Dif.* |
| 1,1-Dichloroethane | 5.0 | 500 | 80-130 | 20 |
| Chloroform | 5.0 | 500 | 80-120 | 20 |
| Dichlorobromomethane | 10 | 1000 | 80-120 | 20 |
| Trichloroethene | 5.0 | 500 | 70-120 | 20 |
| Chlorobenzene | 5.0 | 500 | 80-120 | 20 |
| SCS | | | | |
| Bromochloromethane | 30 | 3000 | 70-160 | - |

* Relative % difference of duplicate runs.

NOTE: 80% of the spike analytes accuracy values must be within control limits, and 80% of the precision values must be within control limits.

**TABLE 4. VOLATILE HALOGENATED ORGANICS ACCEPTANCE CRITERIA**

| Analyte | Spike Concentration | Control Limits (mg/L)[1] |
|---|---|---|
| LCS | | |
| Chloromethane | 20 | 11.9-28.1 |
| Bromomethane | 20 | 11.7-28.3 |
| Vinyl Chloride | 20 | 13.7-26.3 |
| Chloroethane | 20 | 15.4-24.6 |
| Methylene Chloride | 20 | 15.5-24.5 |
| Trichlorofluoromethane | 20 | 13.3-26.7 |
| 1,1-Dichloroethene | 20 | 12.6-27.4 |
| 1,1-Dichloroethane | 20 | 16.8-23.2 |
| trans-1,2-Dichloroethene | 20 | 12.8-27.2 |
| Chloroform | 20 | 15.0-25.0 |
| 1,1,2-Trichloro-2,2,1-trifluoroethane | 20 | 13.3-26.7 |
| 1,2-Dichloroethane | 20 | 14.3-25.7 |
| 1,1,1-Trichloroethane | 20 | 14.2-25.8 |
| Carbon Tetrachloride | 20 | 13.7-26.3 |
| Bromodichloromethane | 20 | 15.2-24.8 |
| 1,2-Dichloropropane | 20 | 14.8-25.2 |
| cis-1,3-Dichloropropene | 14 | 9.00-19.0 |
| Trichloroethylene | 20 | 15.4-24.6 |
| 1,1,2-Trichloroethane | 20 | 15.7-24.3 |
| Dibromochloromethane | 20 | 13.1-26.9 |
| trans-1,3-Dichloropropene | 6.0 | 4.20-7.80 |
| 1,2-Dibromoethane (EDB) | 20 | 14.7-25.3 |
| Bromoform | 20 | 14.7-25.3 |
| 1,1,2,2-Tetrachloroethane | 20 | 9.8-30.2 |
| Tetrachloroethene | 20 | 14.0-26.0 |
| Chlorobenzene | 20 | 14.4-25.6 |
| 1,3-Dichlorobenzene | 20 | 9.9-30.1 |
| 1,2-Dichlorobenzene | 20 | 14.0-26.0 |
| 1,4-Dichlorobenzene | 20 | 13.9-26.1 |

[1] 3rd Edition of SW-846, Rev. 0, p. 8010-11

TABLE 6. HALOGENATED VOLATILE ORGANICS REPORTING LIMITS (SOLID/WASTE)

| Parameter | Solid Reporting Limit ug/kg | Waste Reporting Limit mg/kg |
|---|---|---|
| Chloromethane | 500 | 0.50 |
| Bromomethane | 500 | 0.50 |
| Vinyl Chloride | 100 | 0.10 |
| Chloroethane | 500 | 0.50 |
| Methylene Chloride | 500 | 0.50 |
| 1,1-Dichloroethene | 50 | 0.050 |
| 1,1-Dichloroethane | 50 | 0.050 |
| trans-1,2-Dichloroethene | 50 | 0.050 |
| Chloroform | 50 | 0.050 |
| 1,2,2-Trichloro-2,2,1-trifluoroethane | 100 | 0.10 |
| 1,2-Dichloroethane | 100 | 0.10 |
| 1,1,1-Trichloroethane | 50 | 0.050 |
| Carbon Tetrachloride | 50 | 0.050 |
| Bromodichloromethane | 100 | 0.10 |
| 1,2-Dichloropropane | 100 | 0.10 |
| trans-1,3-Dichloropropene | 100 | 0.10 |
| Trichloroethene | 50 | 0.050 |
| Dibromochloromethane | 100 | 0.10 |
| cis-1,3-Dichloropropene | 200 | 0.20 |
| 1,1,2-Trichloroethane | 100 | 0.10 |
| 1,2-Dibromoethane | 200 | 0.20 |
| Bromoform | 500 | 0.50 |
| 1,1,2,2-Tetrachloroethane | 100 | 0.10 |
| Tetrachloroethene | 50 | 0.050 |
| Chlorobenzene | 200 | 0.20 |
| Additional prompts (not in Std. Product): | | |
| 2-Chloroethyl vinyl ether | 100 | 0.10 |
| 1,2-Dichlorobenzene | 100 | 0.10 |
| 1,3-Dichlorobenzene | 100 | 0.10 |
| 1,4-Dichlorobenzene | 100 | 0.10 |
| Dichlorofluoromethane | 200 | 0.20 |
| Trichlorofluoromethane | 100 | 0.10 |

TABLE 5. HALOGENATED VOLATILE ORGANICS REPORTING LIMITS (WATER)

| Parameter | Reporting Limit ug/L |
|---|---|
| Chloromethane | 5.0 |
| Bromomethane | 5.0 |
| Vinyl Chloride | 1.0 |
| Chloroethane | 5.0 |
| Methylene Chloride | 5.0 |
| 1,1-Dichloroethene | 0.50 |
| 1,1-Dichloroethane | 0.50 |
| trans-1,2-Dichloroethene | 0.50 |
| Chloroform | 0.50 |
| 1,2,2-Trichloro-2,2,1-trifluoroethane | 1.0 |
| 1,2-Dichloroethane | 1.0 |
| 1,1,1-Trichloroethane | 0.50 |
| Carbon Tetrachloride | 0.50 |
| Bromodichloromethane | 1.0 |
| 1,2-Dichloropropane | 1.0 |
| trans-1,3-Dichloropropene | 0.50 |
| Trichloroethene | 1.0 |
| Dibromochloromethane | 1.0 |
| cis-1,3-Dichloropropene | 2.0 |
| 1,1,2-Trichloroethane | 1.0 |
| 1,2-Dibromoethane (EDB) | 2.0 |
| Bromoform | 5.0 |
| 1,1,2,2-Tetrachloroethane | 1.0 |
| Tetrachloroethene | 0.50 |
| Chlorobenzene | 2.0 |

Additional prompts (not in Std. Product):

| | |
|---|---|
| 2-Chloroethylvinyl ether | 1.0 |
| 1,2-Dichlorobenzene | 1.0 |
| 1,3-Dichlorobenzene | 1.0 |
| 1,4-Dichlorobenzene | 1.0 |
| Dichlorodifluoromethane | 2.0 |
| Trichlorofluoromethane | 1.0 |

## APPENDIX A:

FOR AFIRP SAMPLES THE FOLLOWING ADDITIONAL STEPS MUST BE TAKEN AND CHANGES TAKEN INTO ACCOUNT.

A.1 Change in reporting limits: See Table A-1

A.2 Secondary Analysis: Any sample which has an analyte(s) above reporting limit must be reanalyzed using an analytical column which is different than the one used for the primary analysis.

A.3 Data Interpretation: Both values are reported. The preferable value is footnoted in the report. The preferable value will be the lesser of the two values if both are above reporting limits and do not co-elute on either column. If coelution occurs on either column, the other value is preferable. If the secondary analysis result is below reporting limit, an ND is reported and footnoted as the preferable result. If interferences are visibly present on a chromatogram the other value is preferable.

TABLE A-1. REPORTING LIMITS FOR AFIRP ANALYSIS

| Parameter | Water (ug/L) | Soil/Sediment (mg/kg) |
|---|---|---|
| Bromodichloromethane | 0.5 | 0.1 |
| Bromoform | 1 | 0.1 |
| Bromomethane | 6 | 0.1 |
| Carbon Tetrachloride | 0.6 | 0.1 |
| Chlorobenzene | 1.2 | 0.1 |
| Chloroethane | 3 | 0.1 |
| 2-Chloroethyl vinyl ether | 5.5 | 0.5 |
| Chloroform | 0.3 | 0.1 |
| Chloromethane | 1.1 | 0.1 |
| Dibromochloromethane | 0.6 | 0.1 |
| 1,2-Dichlorobenzene | 0.5 | 0.1 |
| 1,3-Dichlorobenzene | 1 | 0.1 |
| 1,4-Dichlorobenzene | 0.5 | 0.1 |
| Dichlorodifluoromethane | 9 | 0.3 |
| 1,1-Dichloroethane | 0.4 | 0.1 |
| 1,2-Dichloroethane | 0.5 | 0.1 |
| 1,1-Dichloroethene | 0.7 | 0.1 |
| trans-1,2-Dichloroethene | 0.5 | 0.1 |
| 1,2-Dichloropropane | 0.5 | 0.1 |
| trans-1,3-Dichloropropene | 2 | 0.2 |
| Methylene Chloride | 2 | 0.1 |
| 1,1,2,2-Tetrachloroethane | 1.4 | 0.1 |
| Tetrachloroethene | 0.4 | 0.1 |
| 1,1,1-Trichloroethane | 0.2 | 0.1 |
| 1,1,2-Trichloroethane | 0.2 | 0.1 |
| Trichloroethene | 0.6 | 0.1 |
| Trichlorofluoromethane | 5 | 0.2 |
| Vinyl Chloride | 0.6 | 0.1 |
| Benzyl Chloride | 6.8 | 0.7 |
| Bromobenzene | 5.0 | 0.1 |
| Bis(2-chloroethoxy)methane | Not Recovered | - |
| Bis(2-chloroisopropyl)ether | 10 | 1.0 |
| Chloroacetaldehyde | Not Recovered | - |
| 1-Chlorohexane | 5.0 | 0.1 |
| Chloromethyl methyl ether | Not Recovered | - |
| 4-Chlorotoluene | 23 | 2.3 |
| Dibromomethane | 5.0 | 0.1 |
| 1,1,1,2-Tetrachloroethane | 5.0 | 0.1 |
| 1,2,3-Trichloropropane | 5.0 | 0.5 |

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1. Scope and Application

1.1 Analytes

The four metals most commonly analyzed by Graphite Furnace Atomic Absorption Spectrophotometry (GFAA) are Arsenic, Lead, Selenium, and Thallium.

Other elements that may be analyzed include (but are not limited to) Antimony, Cadmium, Chromium, and Silver.

1.2 Reporting Limits

Reporting limits range from 0.5 to 10 ug/L and ug/kg according to the analyte. Instrument detection limits (IDL) are also available. Consult the specific analyte information at the end of this SOP for details.

| Prepared by: | Date: |
|---|---|
| David B. Roberts, Jr. | March 3, 1991 |
| Management Approval: David B Roberts Jr | Date: 6-27-91 |
| QA Officer Approval: Willie J Thomas | Date: 06/27/91 |

1.3 Applicable Matrices

This method is applicable to the analysis of metals in dissolved, recoverable and total water samples, as well as digests of soils, wastes and leachates.

1.4 Dynamic Range

The dynamic range is element specific. For the four most common analytes, it extends to 100 ug/L. Higher concentrations may be analyzed by dilution of the sample. Consult the specific analyte information at the end of this SOP for details.

1.5 Analysis Time

Approximately 20 samples per instrument-shift can be analyzed. It is common practice to run two instruments simultaneously. Sample preparation time is not included.

2. Method Summary

2.1 The sample is first digested according to the appropriate method. Dissolved water samples are analyzed without digestion unless specifically requested by the client.

2.2 An aliquot of the prepared samples is automatically injected into an electrothermally heated graphite furnace which is programmed through a series of drying and charring stages. The element of interest is then atomized by a rapid increase in the temperature of the furnace.

2.3 Radiation from a hollow cathode lamp (HCL) or an electrodeless discharge lamp (EDL) is focused through the graphite furnace. The lamp radiates light at the wavelengths specific to the element being analyzed; atoms of the element in the furnace absorb light at the characteristic wavelength. The absorbance is measured electronically.

2.4 The concentration of the element is calculated by comparing the sample absorbance to the absorbances measured for standards containing known concentrations of the element. This calculation is performed by the instrument.

3. Comments

3.1 Interferences

Any substance which alters the atomization or absorption characteristics of the sample or absorbs light at the same wavelength as the metal being analyzed is a potential interference. Specific interferences are discussed in the individual element procedures.

3.1.1 Chemical and physical interferences are prevalent when analyzing samples using these methods.

3.1.2 Many substances produce non-specific background absorption which must be compensated for by the use of a background correction system, either Deuterium Arc or Zeeman.

3.1.3 Samples are highly subject to contamination at the trace levels of interest in this method. Deionized water and high-purity acids are to be used unless otherwise specified.

3.1.4 Autosampler trays are to be kept covered during analysis.

3.1.5 All work areas used to prepare standards and spike samples are to be cleaned before and after each use.

3.2 Helpful Hints

A summary of the operating conditions used is given in a table at the end of this SOP. For more detail, consult the procedures for the individual elements. Instrument-specific information can be found in the instructions for the instrument being used.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

4.3 The graphite furnace reaches **EXTREMELY** high temperatures. Be sure that it has cooled sufficiently before removing the tube.

4.4 Radiation emitted by the tube during the atomization step is harmful to the eyes. **DO NOT** look at the tube during atomization.

4.5 The furnace should be properly vented with an exhaust hood directly over the furnace chamber.

4.6 Samples and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.

4.7 Refer to the RMAL safety manual for additional precautions and instructions for working in the metals lab.

5. Sample Collection and Preservation

5.1 Dissolved water samples should be filtered at the time of collection, preserved with nitric acid to a pH of less than 2 and stored in clean glass or plastic bottles.

5.2 For collection and preservation information of other matrices, consult the appropriate prep SOPs. See section 8.1.

5.3 The holding time for all metals described by this SOP is 6 months.

6. Apparatus

6.1 Atomic absorption spectrophotometer equipped with graphite furnace, background correction system, autosampler and printer. Examples include:

6.1.1 Perkin Elmer Model 2380 with HGA 400 Graphite Furnace and AS 40 Autosampler.

6.1.2 Perkin Elmer Model 5000 Zeeman with HGA 500 Graphite Furnace and AS 40 Autosampler.

6.1.3 Perkin Elmer 5100 Zeeman with HGA 600 Graphite Furnace and AS 60 Autosampler and Epson computer.

6.2 Electrodeless discharge lamps and power supply, or hollow cathode lamps, specific to the element(s) being analyzed.

6.3 Oxford and Eppendorf pipettors, various sizes, with disposable tips.

6.4 Autosampler cups.

6.5 Graphite tubes, platforms, and other supplies.

6.6 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 Standards

7.1.1 Preparation of standard solutions is element specific. Consult the individual element SOPs for details. (See Attachment 1)

7.2 Matrix modifiers

All elements require the addition of a matrix modifier to the sample in order to control interferences. (See Attachment 1 and 3)

7.3 EPA ICV Solution

This solution is used for the Initial Calibration Verification (ICV). It is analyzed immediately following standardization. This solution is obtained from the EPA through EMSL (Las Vegas) by request.

7.4 CCV Solution

This solution is used for Continuing Calibration Verification (CCV). It is analyzed at a frequency of at least 10% (one every 10 samples) during an analytical run. This solution is usually the mid-range standard and prepared daily.

7.5 ICB/CCB Solution

This solution is used for the Initial Calibration Blank (ICB) and the Continuing Calibration Blank (CCB). It is analyzed after the ICV at a frequency of at least 10% (one every 10 samples) during an analytical run. This solution consists of 1% nitric acid plus any matrix modifier used and is prepared daily.

8. Procedure

8.1 Preparation

8.1.1 Total waters, Total Recoverable waters, EP Tox extracts and TCLP extracts are digested using SOP: LM-RMA-2002.

8.1.2 Soils and Wastes are digested using SOP: LM-RMA-2029.

8.2 Instrument Set-Up and Calibration

Instructions vary according to instrument and element. General instructions are given here; consult the appropriate procedures and manuals for the specific analyte being measured and instrument used.

8.2.1 Turn on the main power, cooling water, and argon.

8.2.2 Install the proper lamp and adjust to the proper power setting.

8.2.3 Set the correct wavelength and slit for the element being analyzed.

8.2.4 **CAREFULLY** inspect and clean the quartz windows of the graphite furnace.

8.2.5 After warming up for 10 minutes, optimize the instrument energy throughput.

8.2.6 Make sure that the energy can be turned up past the autogain energy. If problems are encountered achieving the proper energy level, re-optimize the instrument. If this fails to help, then the lamp probably needs to be replaced or the furnace realigned.

8.2.7 Observe the baseline and re-optimize the instrument if it is unstable. If the baseline is still unstable, the lamp probably needs to be replaced.

8.2.8 Turn on the background corrector if using a D2 background correction system. Readjust the lamp power setting, and allow to warm up for 10 minutes.

8.2.9 Observe the baseline again. If it is unstable, re-optimize the instrument. If this fails, check the furnace alignment and readjust if necessary. If the problem persists, turn up the EDL power supply 1 to 2 watts. The deuterium lamp may also need to be replaced.

8.2.10 Inspect the graphite tube and platform, replace if necessary. Clean the contact rings, injection tip and outside of the graphite furnace.

8.2.11 Program the autosampler, furnace, and AA according to the procedures for the instrument being used and the metal being analyzed.

8.2.12 Blanks and calibrations standards must contain any matrix modifier in the same concentration as is in the samples. This is usually added by the instrument using the "Alternate Volume" function or equivalent.

8.2.13 A Calibration Blank and three standards are analyzed.

8.2.2.1 Run blanks successively until the baseline is stable.

8.2.2.2 Calibrate the instrument using the standards indicated for in the procedure for the given instrument and element.

8.2.14 Analyze the EPA ICV, CCV and ICB solutions. If these are not within 10% of the expected value, corrective action must be taken before proceeding.

8.3 Analysis

8.3.1 Add the matrix modifier to all samples

8.3.2 Before analyzing any samples, run the Prep Blank (if applicable) and DCS's. If these are not within acceptable limits, corrective action must be taken before proceeding.

8.3.3 An analytical spike is to be performed on EACH sample. See section 9.2 for acceptance criteria.

8.3.4 Samples exceeding the highest standard are to be diluted and reanalyzed or the results taken from the ICP. Dilutions must be made in such a way that the diluted sample contains the same concentration of acid and any matrix modifier as the undiluted sample.

8.3.5 A Blank (CCB) and standard check (CCV) must be performed after every 10 or less samples. If these are not within 10% of the true value, corrective action must be taken and all samples analyzed since the previous "in control" CCV must be reanalyzed.

8.3.6 A final CCB and CCV should be run.

8.4 Conclusion

8.4.1 After completing the analytical run, follow the shut-down procedure for the specific instrument used. This will include removing and storing the lamp, placing the autosampler, graphite furnace and spectrophotometer on stand-by and turning off the gas and water.

8.4.2 Complete ALL paperwork neatly and in a timely manner. Data packages should include: a cover sheet with all samples and QC information listed, a review checklist filled out by the analyst, all special instructions, an anomaly form describing the analysis, and all benchsheets with samples and results to be entered into LIMS highlighted and all instrument printouts.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A Prep Blank is required with every batch of 20 or less samples. Dissolved samples will not have a prep blank.

9.1.2 Two DCS samples are required with each batch of 20 or less samples.

9.1.3 A blank (CCB) and standard check (CCV) are required after every 10 or less samples.

9.1.4 Duplicates may be required for project specific QC.

9.1.5 Matrix Spikes may be required for project specific QC.

9.1.6 Analytical spikes are to be performed on **ALL** samples.

9.2 Acceptance Criteria

9.2.1 Method blanks should be less than the reporting limit. If the method blank is more than two times the reporting limit the supervisor should be notified for corrective action.

9.2.2 Average DCS recovery must be 75 - 125%.

9.2.3 The RPD for the DCS must be less than or equal to 20%.

9.2.4 The EPA ICV and all standard checks must be within 10% of the expected value.

9.2.5 There are no acceptance criteria for project specific QC.

9.2.6 Analytical Spike Recovery

(See Flow diagram)

9.2.6.1 Sample reads **below** the reporting limit (RL)

| Recovery | Action |
|---|---|
| < 40% | Dilute, reanalyze |
| >=40% and <80% | Report ND at 2x RL |
| >=80% and <=120% | Report ND at RL |
| Recovery >120 | Report ND at 2x RL |

9.2.6.2 Sample reads **at or above** the reporting limit (RL)

| Recovery | Action |
|---|---|
| < 40% | Dilute, reanalyze |
| >=40% and <80% | Dilute, reanalyze |
| >=80% and <=120% | Report result at RL |
| > 120% | Dilute, reanalyze |

NOTE: MSA = Method of standard addition - i.e. be applied under supervisor discretion.

(See Attachment 2)

9.2.7 Dilutions

When a sample must be diluted the dilution chosen will be determined by the analyst's best judgement and should place the analyte concentration in the optimum range of the calibration curve. The sample should not be over-diluted.

9.3 Corrective Action Required

9.3.1 Corrective action taken when the acceptance criteria are not met may include: reanalysis of samples not bracketed by acceptable QC, restandardization of the instrument and reanalysis, or repreparation of the samples.

9.3.2 Follow the corrective actions outlined in the current Enseco QAPP Manual.

10. Calculations

10.1 The instrument automatically calculates sample concentrations from the absorbances.

10.2 Multiply the concentration from the instrument by any dilution factors made during sample preparation and analysis.

10.3 To convert from ug/L (ppb) to mg/L, divide by 1000.

11. Reporting Requirements

11.1 Units

Reporting units are mg/L for aqueous samples and mg/kg for non-aqueous samples. As a convenience, units of ug/L (ppb) may used during analysis.

11.2 Limits

Samples less than the reporting limit are reported as N.D.

11.3 Significant Figures

| Element | Rep. Limit | 0.0001-0.0009 | 0.001-0.009 | 0.01-0.09 | > 0.10 |
|---|---|---|---|---|---|
| Arsenic | 0.005 mg/L | -- | 1 | 2 | 2 |
| Lead | 0.005 mg/L | -- | 1 | 2 | 2 |
| Selenium | 0.005 mg/L | -- | 1 | 2 | 2 |
| Thallium | 0.005 mg/L | -- | 1 | 2 | 2 |
| Antimony | 0.01 mg/L | -- | -- | 1 | 2 |
| Cadmium | 0.0005 mg/L | 1 | 2 | 2 | 2 |
| Chromium | 0.005 mg/L | -- | 1 | 2 | 2 |
| Silver | 0.0005 mg/L | 1 | 2 | 2 | 2 |

11.4 LIMS Data Entry

The usual standards for data entry apply.

11.5 Anomalies

11.5.1 Samples that contain high levels of soluble salts may form crystals upon drying in the furnace and require dilution to obtain acceptable spike recoveries.

11.5.2 High concentrations of non-target compounds may cause poor spike recovery. These samples must be diluted until the spike recovery is acceptable and the reporting limit raised accordingly.

11.5.3 Samples containing significant concentrations of organic material may not pipette smoothly into the furnace or be deposited on the platform properly. These samples must also be diluted or digested before analysis.

11.5.4 Samples requiring large dilutions due to the presence of high concentrations of analyte may be reported using alternate techniques, ie. Flame AA or ICP.

12. Review Requirements

12.1 All data packages must be complete, signed and dated by both the analyst and the peer reviewer. Both the coversheet and the benchsheet should state the analyst's name, the date and type of analysis performed and the instrument used.

12.2 Benchsheets must contain all calibration information, true values for all QC solutions and recoveries for all QC and analytical spikes.

12.3 Verify that calculations were done correctly by randomly checking a few of them.

13. References

13.1 Source Method: USEPA SW-846, "Test Methods for Evaluating Solid Waste", Chapter 3, 7000-series methods, 3rd Edition, 1986.

13.2 Related Documents

13.2.1 Methods for Chemical Analysis of Water and Wastes, EPA 600 Methods, revised March 1983.

13.2.2 Instruction manuals for Perkin Elmer Model 2380, Zeeman 5000 and Zeeman 5100

13.3 Deviations from Source Method and Rationale

Consult individual element procedures.

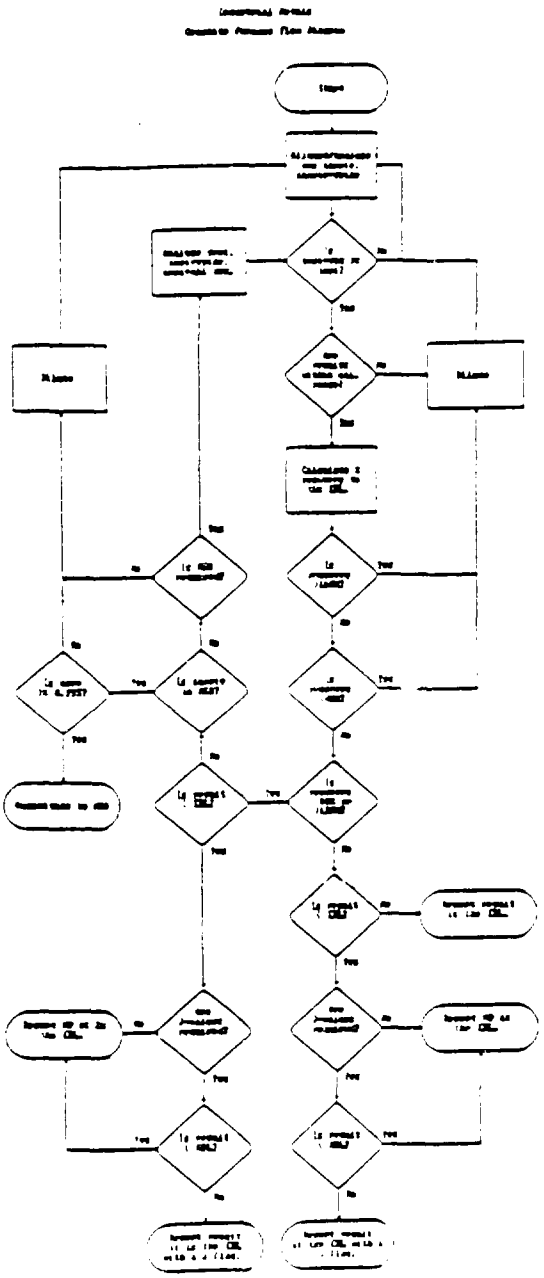
## ATTACHMENT 1

## STANDARD OPERATING CONDITIONS FOR METALS (GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROPHOTOMETRY)

| Element | EPA Ref. | SOP Ref. | Wavelength | Slit | Matrix Modifier |
|---|---|---|---|---|---|
| Arsenic | 7060 | LM-RMA-2007 | 193.7 nm | 0.7 | $Ni(NO_3)_2$ |
| Lead | 7421 | LM-RMA-2013 | 283.3 nm | 0.7 | $(NH_4)_2HPO_4$ |
| Selenium | 7740 | LM-RMA-2019 | 196.0 nm | 0.7 | $Ni(NO_3)_2$ |
| Thallium | 7841 | LM-RMA-2026 | 276.8 nm | 0.7 | $H_2SO_4$ |
| Antimony | 7041 | LM-RMA-2005 | 217.6 nm | 0.2 | $Ni(NO_3)_2$ |
| Cadmium | 7131 | LM-RMA-2011 | 228.8 nm | 0.7 | $(NH_4)_2HPO_4$ |
| Chromium | 7191 | LM-RMA-2036 | 357.9 nm | 0.7 | $Mg(NO_3)_2$ |
| Silver | 7761 | LM-RMA-2023 | 328.1 nm | 0.7 | $PO_3$ |

| Element | Rep. Limit | Standard Concentrations | Analytical Spike(ug/L) |
|---|---|---|---|
| Arsenic | 0.005 mg/L | 0, 20, 50, 100 | 20 |
| Lead | 0.005 mg/L | 0, 20, 50, 100 | 20 |
| Selenium | 0.005 mg/L | 0, 20, 50, 100 | 20 |
| Thallium | 0.005 mg/L | 0, 20, 50, 100 | 20 |
| Antimony | 0.01 mg/L | 0, 20, 50, 100 | 20 |
| Cadmium | 0.0005 mg/L | 0, 1, 2.5, 5 | 1 |
| Chromium | 0.005 mg/L | 0, 20, 50, 100 | 20 |
| Silver | 0.0005 mg/L | 0, 1, 2.5, 5 | 1 |

## ATTACHMENT 2



ATTACHMENT 3

MATRIX MODIFIERS
MARCH 1991

| Element | SW846 | RMAL | Concentrations |
|---|---|---|---|
| Sb Z | (7041) 0.1% $NiNO_3$ | 0.1% Ni | 20 uL of 7.5% $NiNO_3$ to 1.5 mL sample (cup) |
| As Z | (7060) 0.1% $NiNO_3$ | 0.1% Ni | 20 uL of 7.5% $NiNO_3$ to 1.5 mL sample (cup) |
| Cd D2 | (7131) ?x40% $(NH_4)_2$ $HPO_4$ | 1% $PO_4$ | 20 uL of 75% $(NH_4)_2$ to 1.5 mL sample (cup) |
| Cr D2 | (7191) ?x1.18% $Ca(NO_3)_2$ 4 $H_2O$ | 0.025% Mg $(NO_3)_2$ | 20 uL of 1.87% $Mg(NO_3)_2$ in 1.5 mL sample (cup) |
| Pb D2 | (7421) 0.85% $HPO_4$ | 1% $PO_4$ | 20 uL of 75% $(NH_4)_2$ $HPO_4$ to 1.5 mL sample (cup) |
| Ag D2 | (7761) Uses a different prep | 1% $PO_4$ | 20 uL of 75% $(NH_4)_2$ $HPO_4$ to 1.5 mL sample (cup) |
| Se Z | (7740) 0.1% Ni | 0.1% Ni and 0.125% $Mg(NO_3)_2$ | 20 uL of 7.5% $NiNO_3$ and 9.4% $Mg(NO_3)_2$ to 1.5 mL sample (cup) |
| Tl D2 | (7841) ?x0.025% $PdCl_2$ and ?x1% $HNO_3$ | 0.01% $H_2SO_4$ | 20 uL of 0.75% $H_2SO_4$ to 1.5 mL sample (cup) |

STANDARD
OPERATING
PROCEDURE

Subject or Title: Page 1 of 12

**Mercury Preparation and Analysis**
**(Aqueous, Soil, Waste and Leachate Matrices)**

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| **LM-RMA-2005** | **1.0** | **June 25, 1991** |

Supersedes: **Original**

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## 1. Scope and Application

### 1.1 Analytes

This method is the preparation and cold vapor analysis of aqueous samples (dissolved & total), Cam Wet leachates, EP Toxicity leachates (EPI & EPII), Toxicity Characteristic Leaching Procedure leachates (TCLP), soil samples, industrial waste samples, and oily TCLP extracts for total mercury (organic and inorganic) by cold vapor atomic absorption spectrophotometry (CVAAS).

| Prepared By: | Date: |
|---|---|
| Richard M. Persichitte | June 25, 1991 |
| Management Approval: | Date: |
| David B Roberts Jr | 7-10-91 |
| QA Officer Approval: | |
| [illegible] | 07-11-91 |

1.2 Reporting Limits

The reporting limit for mercury (Hg) in both total and dissolved aqueous samples is 0.00020 mg/L. EP Tox, TCLP and Cam Wet leachates are reported at 0.0020 mg/L. Soil or waste samples and oily TCLP extracts are reported at 0.10 mg/kg.

1.3 Applicable Matrices

This method is applicable to total and dissolved aqueous samples, leachates, soil samples and industrial waste matrices.

1.4 Dynamic Range

The dynamic range of samples reported for Hg by CVAA extends upward from a 0.0002 ppm Hg initial concentration.

1.5 Analysis Time

The preparation and analysis time for one batch of 20 samples and required QC is approximately 8 hours (not including instrument warm up time).

## 2. Method Summary

2.1 A representative aliquot of sample is weighed into a 300 mL biochemical oxygen demand (BOD) bottle. Concentrated sulfuric acid ($H_2SO_4$), concentrated nitric acid ($HNO_3$), potassium permanganate ($KMnO_4$), and potassium persulfate ($K_2S_2O_8$) is added. The sample is placed into a water bath and heated to 95°C.

2.1.1 Dissolved and total aqueous samples require a 100 g aliquot (1x).

2.1.2 Leachates require a 10 g aliquot that is diluted to a 100 mL volume with Milli Q water (10x).

2.1.3 Soil or industrial waste samples and oily extracts require a 0.2 g aliquot that is diluted to a 100 mL volume with Milli Q water (500x).

NOTE: If a sample reading for Hg extends beyond the linear range of the highest instrument calibration standard during the first analysis, it must be re-prepped, diluted accordingly and re-analyzed.

2.2 The sample is cooled and sodium chloride-hydroxylamine hydrochloride is added.

2.3 The Hg is then reduced to the elemental state with stannous chloride and aerated from the solution into a closed system. The Hg vapor is passed through a cell positioned in the light path of an atomic absorption spectrometer. The Hg concentration is determined as a function of absorbance at 253.7 nm by CVAAS.

3. Comments

3.1 Interferences

3.1.1 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transferring, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

3.1.2 Failure to homogenize the sample well prior to removal of the desired aliquot may yield erroneous results.

3.1.3 Sea waters, brines, and industrial effluents high in chlorides require additional permanganate. The chlorides are converted to free chlorine, which also absorbs at 253.7 nm and may cause a positive interference. When this interference is suspected use up to 25 mL of additional potassium permanganate during preparation of the sample. During analysis the head space of the BOD must be purged before the addition of the stannous sulfate.

3.1.4 The recovery of Hg from spiked a sample may be affected if the sample contains concentrations of copper > 10 mg/L.

3.1.5 Concentrations of sodium sulfide > 20 mg/L may may interfere with the recovery of added inorganic Hg from ASTM Type II water.

3.2 Helpful Hints

3.2.1 Bring soil samples within 0.01 g of the desired mass and aqueous samples within 0.2 g of the desired mass.

3.2.2 Visual inspection of the Hg vapor cell and pump tubing before starting the instrument warm up procedure is advised.

3.2.3 DO NOT leave the aerator soaking in 1% $HNO_3$ after instrument shutdown.

3.2.4 Leave the peristaltic pump on for 15 minutes after instrument shutdown.

## 4. Safety Issues

4.1 Because mercury vapor is toxic, precaution MUST be taken to avoid its inhalation.

4.2 Proper protective gear (i.e., safety glasses, face shield, gloves, and rubber apron) should be worn when using acids.

4.3 Sample transfers, digestions, and analysis should take place in a properly vented hood, and spills should be cleaned up immediately.

4.4 Sulfuric acid, nitric acid, and stannous chloride are corrosive and will cause skin irritation. Avoid contact with eyes, skin and clothing. Wash thoroughly with copious amounts of water.

4.5 All waste generated from the test will be placed into an approved, correctly labelled waste container.

4.6 After analysis, allow high level samples to aerate in the hood until the analyzer indicates the Hg level has returned to the base line.

4.7 The RMAL safety manual should be referred to for more detailed cautions and instructions.

## 5. Sample Collection, Preservation and Holding Times

5.1 Samples should be collected in glass or plastic containers.

5.2 Dissolved and total aqueous samples should be acidified with nitric acid to pH <2 at the time of collection.

5.3 For dissolved metals analyses, the samples should be filtered through 0.45 micron filter paper before acid preservation. Filtration must be done in the field or within 24 hr of collection.

5.4 Mercury has a 28 day holding time. All other metals analyses have 180-day holding times.

## 6. Apparatus

6.1 Hot plate, capable of 95°C.

6.2 Miscellaneous pipettes.

6.3 Miscellaneous benchsheets.

6.4 Calculator.

6.5 300 mL Wheaton biochemical oxygen demand (BOD) bottles.

6.6 Top-loading balance, 0.01 g.

6.7 Water bath with temperature controlled at 95°C.

6.8 Spectro-Products HG-3 cold vapor mercury analyzer.

6.9 Milli volt (mV) meter capable of digital voltage display.

6.10 Peristaltic air pump with variable speed control set at a flow rate of 1 liter/min.

6.11 Aeration tube (a glass frit with coarse porosity attached to air pump).

6.12 250 mL beaker.

## 7. Reagents and Standards

7.1 ASTM Type II water, (Milli Q or equivalent).

7.2 Hg standard stock solution "A": See RMAL Hg LCS + Calibration Stock SOP LS-RMA-4501.

7.3 Hg DCS stock solution "B": See RMAL Hg LCS + Calibration Stock SOP LS-RMA-4501.

7.4 Sulfuric acid, ($H_2SO_4$), concentrated, trace metal grade.

7.5 Sulfuric acid, 0.5 N: Carefully add 14 mL of concentrated sulfuric acid to 1 liter of Milli Q water.

7.6 Nitric acid, ($HNO_3$), concentrated, trace metal grade.

7.7 Nitric acid, ($HNO_3$), 1% solution (v/v): Carefully add 20 mL concentrated nitric acid to 1980 mL water.

7.8 Potassium permanganate, mercury-free, 5% solution (w/v): dissolve 100 g of potassium permanganate in 2 liters of Milli Q water.

7.9 Potassium persulfate, 5% solution (w/v): dissolve 100 g of potassium persulfate in 2 liters of Milli Q water.

7.10 Sodium chloride-hydroxylamine hydrochloride solution: dissolve a 240 g portion of sodium chloride and 240 g of hydroxylamine hydrochloride in 2 liters of Milli Q water.

7.11 Stannous chloride: add 100 g stannous chloride to 1 liter of 0.5 N $H_2SO_4$. This mixture is a suspension (should appear cloudy) and should be stirred continuously during use.

## 8. Procedure

8.1 Instrument Set-up and Calibration

8.1.1 Turn on hot plate, set for 95°C.

8.1.2 Verify the calibration of the balance and pipettes.

8.1.3 Turn on the Hg analyzer and warm up instrument as follows:

8.1.3.1 Turn on the mV meter (0.000 mV will display) and depress the power switch (the button will glow orange) of the Hg analyzer.

8.1.3.2 Rotate the "B" lamp control clockwise until the mV reading is 0.950 mV and stop to allow the Fe reference lamp a 15 minute warm up period.

8.1.3.3 Continue to stage the the mV reading of the reference lamp to 1.000 mV by increasing the previous mV reading by 50% and a 5 minute warm up period.

8.1.3.4 With the reference lamp at 1.000 mV, depress the AGC switch (the button will glow green and a temporary fluctuation in the mV reading will occur, if the mV reading does NOT return to 1.000 mV within 1 minute consult your supervisor for corrective action). After the mV reading stabilizes at 1.000 mV, rotate the "A" control clockwise until the mV reading of the Hg base lamp is 0.500 mV and allow a 15 minute warm up period.

8.1.3.5 Continue to stage the mV reading of the base lamp to 0.000 mV by decreasing the previous mV reading by 50% and a 5 minute warm up period.

NOTE: Reverse the above steps (with exception to the warm up periods) to shut the instrument down.

8.2 General Preparation

8.2.1 Identify the test to be preformed by using the backlogs.

8.2.2 Check for special instructions concerning each sample.

8.2.3 Sign out samples from sample receiving.

8.2.4 Complete bench sheet.

NOTE: Aqueous samples reported in mg/L may NOT be placed on the same bench sheet with soil, waste or oily TCLP extract samples that are reported in mg/kg.

8.2.5 Update the backlog to reflect which samples are in progress.

NOTE: Consult your supervisor if a sample can NOT be located.

8.2.6 Transfer a homogenized (well-mixed) sample aliquot into a clean numbered 300 mL BOD bottle. Cap the BOD.

8.2.6.1 Total and dissolved water samples are analyzed at a 1x dilution and require a 100 g aliquot.

8.2.6.2 EP Tox, TCLP, and Cam Wet leachates are analyzed at a 10x dilution and require a 10 g sample aliquot brought to a 100 g mass with Milli Q water.

NOTE: A sample spike must be analyzed on 1 sample for each batch of EP Tox leachates prepared for Hg.

8.2.6.3 Soil or waste samples and oily TCLP extracts are analyzed at a 500x dilution and require a 0.2 g sample aliquot brought to a 100 g mass with Milli Q water.

8.2.7 Add 5 mL of concentrated $H_2SO_4$ and 2.5 mL of concentrated $HNO_3$ and mix after each addition. Recap the BOD and allow to stand for 5 minutes or until the acid fumes subside.

8.2.8 Add 15 mL potassium permanganate solution. Additional permanganate may be required. Recap BOD. Shake and add additional portions of permanganate as required until the purple color persists for at least 15 min. Recap BOD.

8.2.9 Add 8 mL of potassium persulfate to each bottle and heat for 2 hours in a water bath at 95°C. Remove samples from water bath and allow to cool.

8.3 Analysis

8.3.1 Add 6 mL of sodium chloride-hydroxylamine hydrochloride to the cooled samples in the BOD bottles. This reduces the excess

permanganate and causes the solution to decolorize. Allow the samples to stand with the caps ajar for 5-10 minutes to allow any chlorine gas to dissipate. Treat the samples separately at this point.

8.3.2 Samples are to be analyzed according to the bench sheet in the following order: instrument calibration standards, initial calibration verification (ICV), initial calibration blank (ICB), PB, 2 duplicate control samples (DCS), 10 or less samples, followed by a continuing calibration verification (CCV) and a continuing calibration blank (CCB). For every twenty (or less) samples analyzed, 2 DCS samples will be analyzed along with a CCV and CCB for every 10 samples (or less) to be analyzed.

8.3.3 Insert the aerator into the sample BOD (above the fluid level) and purge the head space for 30 seconds.

8.3.4 Add 5 mL of stannous chloride and immediately attach the aeration apparatus to the BOD bottle. As the Hg is bubbled out of the sample, the absorbance (peak height measured in mV) will rise to a maximum value and level off within approximately 30 seconds. Record the mV reading of each sample on the bench sheet. For all samples reading above 0.05 mV, allow the apparatus to aerate with the frit tube ajar until the reading has returned to the base line (this dissipates the Hg vapors into the hood).

NOTE: Samples reading above the highest instrument calibration standard must be flagged during analysis. Upon conclusion of the analysis, these BODs will be flushed with hot tap water, rinsed and filled with 275 mL of DI water, and topped off with $H_2SO_4$ then allowed to stand for 1 hour. The BOD will then be ready for routine washing procedures.

8.3.5 Place the aerator into 100 mLs of 1% $HNO_3$ and allow to bubble rinse until the next sample analysis.

8.3.6 Analyze the initial calibration standards. Construct a calibration curve by plotting the peak height in mV versus Hg concentration. The linear regression of these data should produce a correlation coefficient for the curve of >0.995.

The initial calibration standards must then be re-analyzed if the calibration curve does not meet the above criterion.

8.3.7 Analyze each sample (including all QC samples) and determine the Hg concentration in ppm from the linear regression fit of the calibration curve.

## 9. QA/QC Requirements

9.1 Instrument Calibration Standards Preparation

9.1.1 Prepare and analyze a 100 g Milli Q water sample per the procedure as described in section 8.2-8.3, for the instrument calibration blank.

9.1.2 Add, 50 ug, 100 ug, 200 ug, and 400 ug of the Hg standard stock solution "A" to 4 separate BOD bottles, each containing 100 g of Milli Q water, and prepare and analyze the instrument calibration standards as described in section 8.2-8.3.

NOTE: These 5 instrument calibration standards will be used to determine the linear regression of the standard curve. One set of instrument calibration standards will be prepared for each batch of samples prepped.

9.2 ICBs and CCBs must be prepared and analyzed.

9.2.1 ICB: Prepare and analyze a 100 g Milli Q water sample per the procedure described in section 8.2-8.3. One ICB is prepared for each sample batch. It must be analyzed after the ICV sample.

9.2.2 CCB: Prepare and analyze a 100 g Milli Q water sample as per the procedure described in section 8.2-8.3. One CCB shall be prepared for each 10 samples digested or for each batch, whichever is more frequent. It must be analyzed after each CCV sample.

9.3 ICVs and CCVs must be prepared and analyzed.

9.3.1 ICV: Add 200 ug Hg EPA ICV 5 concentrate standard to 100 g of Milli Q water. Prepare and analyze the ICV per the procedure described in section 8.2-8.3. One shall be prepared for each each batch of samples prepped. Recovery must be within 90% to 110% of the true value following QA/QC requirements.

9.3.2 CCV: Add 100 ug Hg standard stock solution "A" to 100 g of Milli Q water. Prepare and analyze the CCV per the procedure described in section 8.2-8.3. One CCV shall be prepared for each 10 (or less) samples digested. Recovery must be within 90% to 110% of the true value following QA/QC requirements.

9.4 DCS samples shall be prepared and analyzed in duplicate for each 20 (or less) samples digested. These 2 samples are prepared by adding 100 ug of the Hg DCS stock solution "B" to 100 g of Milli Q water and digesting them as described in section 8.2-8.3. The recovery for each DCS must be 75% to 125% and their precision must be a relative percent difference of <20% per QA/QC requirements. If this is not the case, terminate the analysis and prep all the associated samples again.

9.5 Client-specific sample duplicates and sample spikes (i.e., matrix spikes and spike duplicates) will be prepared only upon client request and will then appear on the prep scheduling sheet. The MS and SD samples will be spiked with 100 ug of Hg standard stock solution "A".

## 10. Calculations

Use a scientific calculator with linear regression capabilities to calculate the standard calibration curve. Calculate from mV to concentration of Hg in the sample as ug/L or mg/L.

## 11. Reporting Requirements

11.1 Total and recoverable aqueous sample results are reported as mg/L units at 0.00020 ppm Hg.

11.2 Leachate sample results are reported in mg/L units at 0.0020 ppm Hg.

11.3 Soil, waste or oily TCLP extract sample results are reported in mg/kg units at 0.10 ppm Hg.

NOTE: Report all results with 2 significant figures if the Hg value is less than 10 units. When the Hg value exceeds 10 units, report all results with 3 significant figures.

## 12. Review Requirements

12.1 All data packages must be complete, signed, and dated by both the analyst and the peer reviewer. Both the coversheet and the benchsheet should state the analyst's name, the date and type of analysis performed, and the instrument used.

12.2 Benchsheets must contain all calibration information, true values for all QC solutions and recoveries for all QC and analytical spikes.

12.3 Verify that calculations were done correctly by randomly checking a few of them.

## 13. References

13.1 Methods 7470 and 7471. SW-846 - Test methods for evaluating solid waste. 3rd edition.

13.2 U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work 787 (Rev 12/87) Exhibit D - Analytical Methods.

## 14. Deviations From Source Methods

14.1 Soils and wastes are digested according to section 7.2 of Method 7471 and are heated in a water bath rather than an autoclave.

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| LM-RMA-4003 | 3.0 | September 21, 1989 |

Supersedes:
2.0

## Analysis of Organochlorine Pesticides and PCBs

1. Scope and Application

1.1 Analytes: organochlorine pesticides and polychlorinated biphenyls (see accompanying analyte lists)

1.2 Detection Limits: see analyte list in Section 2.2

1.3 Applicable matrices: water,soil, solids, oils, leachates, vegetation.

1.4 Linear Range of Instrument

1.4.1 Five point calibration curves are generated for all analytes except the multi-peak components (PCBs, chlordane, toxaphene), which have single point calibrations.

1.5 Analysis time

1.5.1 Sample prep requires at least 2 days for completion unless cleanup is required.

1.5.2 Analysis time is approximately 35 min per sample or standard.

| Prepared by: | Date: |
|---|---|
| Debra Henderer | February 23, 1989 |
| Management Approval: | Date: |
| Susan M. Brillante | 9/22/89 |
| QA Officer Approval: | Date: |
| Ina M Ligon | 9/22/89 |

2. Summary of Method

2.1 Samples are extracted with methylene chloride ($CH_2Cl_2$). Extracts are dried, exchanged to hexane, and concentrated to a final volume of 10.0 mL. Analysis is performed on a gas chromatograph equipped with an electron capture detector. Samples are simultaneouly analyzed on two dissimilar columns. Data from one column are reported.

2.2 Reporting limits for the target parameters are presented in the following tables:

| Parameter | 608-PP-A Std. Prod. Reporting Limit |
|---|---|
| alpha-BHC | 0.0030 ug/l |
| beta-BHC | 0.0060 |
| delta-BHC | 0.0090 |
| gamma-BHC (Lindane) | 0.0040 |
| Heptachlor | 0.0030 |
| Aldrin | 0.0040 |
| Heptachlor Epoxide | 0.083 |
| Endosulfan I | 0.014 |
| Dieldrin | 0.0080 |
| 4,4'-DDE | 0.0080 |
| Endrin | 0.0060 |
| Endosulfan II | 0.0080 |
| 4,4'-DDD | 0.011 |
| Endosulfan Sulfate | 0.066 |
| 4,4'-DDT | 0.012 |
| Endrin Aldehyde | 0.023 |
| Methoxychlor | 0.25 |
| Chlordane | 0.040 |
| Toxaphene | 0.60 |
| Aroclor-1016 | 0.065 |
| Aroclor-1221 | 0.065 |
| Aroclor-1232 | 0.065 |
| Aroclor-1242 | 0.065 |
| Aroclor-1248 | 0.065 |
| Aroclor-1254 | 0.50 |
| Aroclor-1260 | 0.50 |

608-HSL-A & 608-AP9-A
Std. Prod.

| Parameter | Reporting Limit |
|---|---|
| alpha-BHC | 0.050 ug/L |
| beta-BHC | 0.050 |
| delta-BHC | 0.050 |
| gamma-BHC (Lindane) | 0.050 |
| Heptachlor | 0.050 |
| Aldrin | 0.050 |
| Heptachlor Epoxide | 0.050 |
| Endosulfan I | 0.050 |
| Dieldrin | 0.10 |
| 4,4'-DDE | 0.10 |
| Endrin | 0.10 |
| Endosulfan II | 0.10 |
| 4,4'-DDD | 0.10 |
| Endosulfan Sulfate | 0.10 |
| 4,4'-DDT | 0.10 |
| Endrin Ketone | 0.10 |
| Methoxychlor | 0.50 |
| Chlordane | 0.50 |
| Toxaphene | 1.0 |
| Aroclor-1016 | 0.50 |
| Aroclor-1221 | 0.50 |
| Aroclor-1232 | 0.50 |
| Aroclor-1242 | 0.50 |
| Aroclor-1248 | 0.50 |
| Aroclor-1254 | 1.0 |
| Aroclor-1260 | 1.0 |
| **Appendix IX** | |
| Chlorobenzilate | 0.50 ug/L |
| Diallate | 1.5 |
| Isodrin | .050 |
| Kepone | .10 |

| Parameter | 608-RCRA-A Reporting Limit | 8080-RCRA-S Reporting Limit |
|---|---|---|
| Lindane | 0.20 ug/L | 1.0 ug/kg |
| Endrin | 0.20 | 1.0 |
| Methoxychlor | 1.0 | 5.0 |
| Toxaphene | 2.0 | 10 |

| Parameter | 608-SDWAP-A Reporting Limit |
|---|---|
| Lindane | 0.10 ug/L |
| Endrin | 0.10 |
| Methoxychlor | 0.50 |
| Toxaphene | 1.0 |

| Parameter | 8080-HSL-S Reporting Limit (ug/kg) | 8080-HSL-L-S Reporting Limit (ug/kg) |
|---|---|---|
| alpha-BHC | 8.0 | 1.0 |
| beta-BHC | 8.0 | 1.0 |
| delta-BHC | 8.0 | 1.0 |
| gamma-BHC (Lindane) | 8.0 | 1.0 |
| Heptachlor | 8.0 | 1.0 |
| Aldrin | 8.0 | 1.0 |
| Heptachlor Epoxide | 8.0 | 1.0 |
| Endosulfan I | 8.0 | 1.0 |
| Dieldrin | 16 | 2.0 |
| 4,4'-DDE | 16 | 2.0 |
| Endrin | 16 | 2.0 |
| Endosulfan II | 16 | 2.0 |
| 4,4'-DDD | 16 | 2.0 |
| Endosulfan Sulfate | 16 | 2.0 |
| 4,4'-DDT | 16 | 2.0 |
| Endrin Ketone | 16 | 2.0 |
| Methoxychlor | 80 | 10 |
| Chlordane | 80 | 10 |
| Toxaphene | 160 | 20 |
| Aroclor-1016 | 80 | 10 |
| Aroclor-1221 | 80 | 10 |
| Aroclor-1232 | 80 | 10 |
| Aroclor-1242 | 80 | 10 |
| Aroclor-1248 | 80 | 10 |
| Aroclor-1254 | 160 | 20 |
| Aroclor-1260 | 160 | 20 |

8080-AP9-S & 8080-PP-S

| Parameter | Reporting Limit (ug/kg) |
|---|---|
| alpha-BHC | 8.0 |
| beta-BHC | 8.0 |
| delta-BHC | 8.0 |
| gamma-BHC (Lindane) | 8.0 |
| Heptachlor | 8.0 |
| Aldrin | 8.0 |
| Heptachlor Epoxide | 8.0 |
| Endosulfan I | 8.0 |
| Dieldrin | 16 |
| 4,4'-DDE | 16 |
| Endrin | 16 |
| Endosulfan II | 16 |
| 4,4'-DDD | 16 |
| Endosulfan Sulfate | 16 |
| 4,4'-DDT | 16 |
| Endrin Aldehyde | 16 |
| Methoxychlor | 80 |
| Chlordane | 80 |
| Toxaphene | 160 |
| Aroclor-1016 | 80 |
| Aroclor-1221 | 80 |
| Aroclor-1232 | 80 |
| Aroclor-1242 | 80 |
| Aroclor-1248 | 80 |
| Aroclor-1254 | 160 |
| Aroclor-1260 | 160 |

Appendix IX

| | |
|---|---|
| Chlorobenzilate | 80 ug/kg |
| Diallate | 240 |
| Isodrin | 8.0 |
| Kepone | 16 |

3. Comments

3.1 Interferences are myriad. A florisil or alumina column cleanup is often required for soil samples (see Sections 8.5 and 8.6 respectively).

3.2 Helpful hints:

3.2.1 If a dirty-looking sample must be analyzed, insert a hexane blank in the following autosample position.

3.2.2 Use microvials for small sample volumes.

3.2.3 Always rinse autosampler vials with methanol (first) and hexane (last) and dry before use.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Sample Collection, Preservation and Handling

5.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed, except that the bottle must not be pre-rinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance

with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

5.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction.

5.3 All aqueous samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction. Soil/waste samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

6. Apparatus

6.1 Separatory funnel - 2 L, with Teflon stopcock.

6.2 Filtering Funnel - 75 mm top diameter.

6.3 Chromatographic column - 400 mm long X 22 mm ID.

6.4 Concentrator tube, Kuderna-Danish - 10 mL graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

6.5 Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

6.6 Snyder column, Kuderna/Danish - 3-ball macro (Kontes K-503000-0121 or equivalent).

6.7 Vials - 10-15 mL with Teflon-lined screw cap.

6.8 Boiling chips - Approximately 10/40 mesh.

6.9 Water bath - Heated, with concentric ring over cover, capable of temperature control (± 2°C). The bath should be used in a hood.

6.10 Balance - Analytical capable of accurately weighing 0.0001g.

6.11 Sonicator - A horn-type sonicator equipped with a titanium tip should be used. The following sonicator, or an equivalent brand and model is recommended:

Ultrasonic cell disrupter: Heat Systems - Ultrasonics, Inc. Model W-385 (475 W) sonicator or equivalent (Power wattage must be a minimum of 375 W with pulsing capability and No. 200 1.2" Tapped Disrupter Horn) plus No. 207 3/4" Tapped Disrupter Horn, and No. 419 1/8" Standard Tapered microtip probe.

6.12 Sonabox - Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

6.13 Gas Chromatography - An analytical system complete with a gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-cart recorder. A data system is recommended for measuring peak areas.

6.14 Detector - Electron capture detector.

7. Reagents and Standards

7.1 Reagent water - Reagent water is defined as a water in which an interferent is not observed at or above the method detection limit of each parameter of interest.

7.2 Sodium hydroxide solution (10 N)

7.3 Sulfuric Acid (1:1) ACS sp. gr. 1.84

7.4 Acetone, hexane, methylene chloride - pesticide quality or equivalent

7.5 Diethyl ether - nanograde, redistilled in glass if necessary. Must be free of peroxides as indicated by EM Quant test strips. After opening a bottle of diethyl ether, one must test it daily for peroxides. (Available from Scientific Products Co., Cat. NO. P1126-8, and other suppliers.) Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL ethyl alcohol preservative must be added to each liter of ether.

7.6 Sodium sulfate - (ACS) granular, anhydrous. Dry in a shallow tray at 400°C for a minimum of 4 hr to remove phthalates and other interfering organic substances. Alternatively, heat 16 hr at 450-500°C in a shallow tray or Soxhlet extract with $CH_2Cl_2$ for 48 hr.

7.7 Florisil - PR grade (60/100 mesh). Purchase activated at 1250°F and store in dark in glass container with ground glass stopper or foil-lined screw cap. Before use activate each batch at least 16 hr at 130°C in a foil covered glass container.

7.8 TBA-sulfite (Tetrabutylammonium sulfite)

7.9 Isopropyl alcohol

7.10 Primary standard solutions may be prepared from pure standard materials or purchased as certified solutions.

8. Extraction of Liquid Samples

8.1 Method Summary

This method is applicable to the determination of organochlorine pesticides and PCBs in aqueous samples. A 1 L water sample is extracted with $CH_2Cl_2$. The extract is dried, exchanged to hexane, and concentrated to a volume of 10.0 mL. Analysis is performed on a gas chromatograph equipped with an electron capture detector.

8.2 Extraction by Shakeout

8.2.1 If the sample contains particulate matter, transfer 3-4 mL of sample to a 12 mL screw top glass vial with a Teflon liner. Add approximately 1 mL of $CH_2Cl_2$. Shake vigorously for about 30 s and allow the layers to separate. If no emulsion forms, proceed with the shakeout technique. If an emulsion forms, use the continuous extractor method. In general, pesticide recoveries may be poor if there is an extreme amount of sediment or an extreme emulsion forms during the emulsion test.

NOTE: All glassware must be solvent rinsed with $CH_2Cl_2$ before use.

8.2.2 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle. All others are mixed by shaking the bottle.

8.2.3 For samples in 1 L or smaller bottles, mark the meniscus on the side of the sample bottle. After emptying the

sample into a separatory funnel, fill the bottle with tap water and measure that amount in a graduated cylinder. For samples in larger bottles, measure 1.0 L into a graduated cylinder. Pour the sample into a 2 L separatory funnel. For blanks and quality control samples, pour 1.0 L of carbon filtered water into the separatory funnel. Record the volume of sample extracted on the prep sheet.

8.2.4 Add the appropriate surrogate standard to the sample using a 1 mL disposable pipet. Make sure the pipet tip is below the surface of the sample while the standard is being added. Mix the sample immediately by stirring with the disposable pipet. Record the name of the standard, verification number, amount added to sample, standard preparation date, initials of person who prepared standard, standard concentration, date added, initials of person adding standard, and initials of a witness on the prep sheet.

8.2.5 Add the spiking standard, if appropriate, using the procedure described in the preceding step.

8.2.6 Check the pH and note the initial pH on the prep sheet. Adjust the pH to 5-8, if necessary, with 10 N NaOH or 1:1 $H_2SO_4$. Note the adjusted pH on the prep sheet.

8.2.7 For samples that were mixed before pouring, add 60 mL $CH_2Cl_2$ to the sample bottle to rinse the inner walls. Do **NOT** cap and shake the bottle. Rinse the glass only; transfer the solvent to the separatory funnel. For samples that were decanted, add the first aliquot of $CH_2Cl_2$ directly to the separatory funnel. Extract the sample by shaking it for 2 min with frequent ventilation.

8.2.8 Allow the layers to separate. If there is an emulsion, break it mechanically or by centrifuging. Note any emulsions and corrective actions on the prep sheet.

8.2.9 Drain the bottom organic layer ($CH_2Cl_2$) into a 250 mL beaker.

8.2.10 Repeat the extraction 2 more times using a 60 mL aliquot of $CH_2Cl_2$ each time. Collect the solvent in the same beaker. Record the total volume of solvent recovered on the prep sheet.

8.2.11 Put a plug of glass wool in a funnel and fill about 2/3 full with $Na_2SO_4$. Rinse the funnel and $Na_2SO_4$ with 30-40 mL of $CH_2Cl_2$. Discard this rinsate in an appropriate waste container. Pour the extract through the $Na_2SO_4$ into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the beaker then the $Na_2SO_4$ with small amounts of $CH_2Cl_2$. Add these rinses to the K-D.

8.2.12 Add a boiling chip to the K-D and attach a 3 ball Snyder column to the top. Prewet the column by adding approximately 1 mL of $CH_2Cl_2$ through the top.

NOTE: The concentration step is critical; losses of target compounds can occur if the bath temperature is too high or too low or if extracts are allowed to boil too long.

8.2.13 Place the K-D in an 85°C hot water bath so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, remove the K-D from the bath and allow it to cool completely.

8.2.14 When the K-D has cooled, pour 50 mL of hexane through the top of the Snyder column. Add a fresh boiling chip and shake the Snyder column to make sure that the balls are not stuck. When the water bath has fully reheated to 95°C return the K-D to the water as before.

NOTE: Losses of target compounds can occur at this step. Make sure solvent loss is rapid (5-10 min). Do not allow the extract to evaporate for more than 10 min.

8.2.15 If the extract is highly colored or a precipitate forms during concentration, consult a supervisor.

8.2.16 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of hexane. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of hexane. Be sure to rinse all of the ground glass joints well, as compounds collect on the ground glass.

8.2.17 Carefully concentrate the extract to 10.0 mL under a gentle stream of nitrogen using the N-evap apparatus. If the extract is highly colored, forms a precipitate, or stops evaporating, consult a supervisor. Transfer the extract to a 12.0 mL vial with a teflon lined cap and mark the meniscus. Write your initials, date of final concentration, volume, parameter, solvent, project, and sample number on the vial.

8.2.18 If there is **ANY** color to the extract or precipitate, the organochlorine pesticide fraction will need a florisil column cleanup. Refer to Section 8.5.

8.2.19 Complete the prep sheet for the extraction and concentration steps.

8.3 Extraction by Continuous Extractor

8.3.1 See Section 8.2.1 for guidance on when to use the continuous extractor.

8.3.2 Put 250 mL $CH_2Cl_2$ in a round bottom flask and add a few boiling chips. Add 300 mL of $CH_2Cl_2$ to the extractor flask.

8.3.3 Carefully pour 500 mL of carbon filtered water into the extractor. Minimize the disturbance of the solvent layer and avoid getting water into either sidearm by pouring the water down the back of the extractor.

8.3.4 Open the sample container and remove a small aliquot of the sample with a pasteur pipet. Place enough of the sample on a pH paper to thoroughly soak the paper. Compare the pH paper with the chart on the container and record the initial pH on the prep sheet.

8.3.5 Aliquoting different kinds of samples

8.3.5.1 Water samples: Decant ground water samples into the extractor. Mix all other water samples by shaking the sample bottle. For samples in 1 L or smaller bottles, mark the meniscus on the side of the sample bottle. After emptying the sample into the extractor, fill the bottle with

tap water and measure that amount in a graduated cylinder. For samples in larger bottles, measure 1.0 L into a graduated cylinder. Pour the sample into the extractor. For blanks and quality control samples, pour 1.0 L of carbon filtered water into the extractor. Record the volume of sample extracted on the prep sheet.

8.3.5.2 Sludge samples: Remove an aliquot of approximately 10 g, place it in a pre-weighed aluminum boat and air-dry it overnight to determine the percent moisture. Weigh the bottle containing the sample and carefully transfer the sample to the extractor flask. Re-weigh the bottle, calculate the difference, and record it as the amount extracted on the prep sheet.

8.3.6 Add enough carbon filtered water to the extractor to bring the level of the solvent in the removable sidearm to about 4 cm from its highest point. Make sure that the extractor stays in a strictly vertical position.

8.3.7 Add the appropriate surrogate standard to the extractor using a disposable pipet. Make sure that the tip of the pipet is below the surface of the liquid in the extractor as the standard is being added. Mix the sample immediately using the disposable pipet. Record the name of the standard, verification number, amount added to sample, standard preparation date, initials of person who prepared standard, standard concentration, date added, initials of person adding standard, and initials of a witness on the prep sheet.

8.3.8 Add the spike mix, if appropriate, using the procedure described in the preceding step.

8.3.9 Adjust the pH to 5-8 with 10 N NaOH or 1:1 $H_2SO_4$ as appropriate.

8.3.10 Add enough carbon filtered water to the extractor flask to allow the solvent in the removable sidearm to just begin to drip into the round bottom flask. Record the total volume of carbon filtered water that was added on the prep sheet.

8.3.11 Remove the condenser from the rack and rinse the lower joint and lip with a squirt bottle ($CH_2Cl_2$). Place the condenser on top of the extractor. Turn on the water chiller (set at 10°C) and check the flow indicators.

8.3.12 Turn on the heating mantle so that the indicator light is barely lit. Record the starting time on the prep sheet. Check after 15 min to be sure that the solvent in the round bottom flask is boiling, that solvent is dripping from the lip of the condenser, and that the volume of the solvent in the round bottom flask is still approximately 250 mL. Adjust the rheostat so that the light is just bright after refluxing has started. Check all extractor joints for leaks with the corner of a Kimwipe.

8.3.13 Allow the extraction to proceed for a minimum of 6 hr.

8.3.14 Turn off the heating mantle and allow the apparatus to cool (30-60 min) with water flowing through the condenser.

8.3.15 The solvent ($CH_2Cl_2$) contained in the round bottom flask is the extract. Remove the flask from the extractor and transfer the extract to a rinsed 400 mL beaker. Rinse the flask with a small aliquot of $CH_2Cl_2$ and add this rinsate to the 400 mL beaker. Record the volume of extract recovered on the prep sheet.

8.3.16 Go to Section 8.2.11 and proceed with the prep.

8.4 Extraction of Soil Samples by Sonication

NOTE: All glassware must be solvent rinsed with $CH_2Cl_2$ before use.

8.4.1 Weigh 4 g of the sample into a 250 mL centrifuge bottle. Add 10 g of dried $Na_2SO_4$ and stir the mixture well with a steel spatula. The sample should have a grainy texture. If a clump forms add more $Na_2SO_4$ and note it on the prep sheet.

8.4.2 Add 50 mL of $CH_2Cl_2$ to all samples.

8.4.3 Add the appropriate surrogate standard using a disposable pipet.

NOTE: For samples being analyzed for PCBs only, the surrogate is added only to the method blank (SCS). Drizzle the standard over the surface of the sample and immediately swirl the flask to mix the standard with the sample. On the prep sheet, record all of the required information regarding the standard.

8.4.4 Add the spike, if appropriate, using the procedure described in the previous step.

8.4.5 Sonicate the samples for 1.5 min at an output setting of 10 with the 3/4 inch sonicator horn 1/2 inch below the surface of the solvent. The sonicator should be in the 1 s pulse mode, with the duty cycle set at 50%. Centrifuge the samples for 3-5 min at 35 K RPM.

8.4.6 Decant the solvent layer into a rinsed 250 mL beaker.

8.4.7 Repeat the extraction 2 more times using 50 mL aliquots of $CH_2Cl_2$ each time. Collect these extracts in the same beaker.

8.4.8 Record the total volume of solvent recovered on the prep sheet.

8.4.9 Put a plug of glass wool in a funnel and fill 2/3 full with $Na_2SO_4$. Rinse the funnel and $Na_2SO_4$ with 30-40 mL of $CH_2Cl_2$, discard. Pour the extract through the $Na_2SO_4$ into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the beaker then the $Na_2SO_4$ with small amounts of $CH_2Cl_2$ into the K-D flask.

8.4.10 Add a boiling chip to the K-D and attach a 3 ball Snyder to the top. Pre-wet the column by adding about 1 mL of $CH_2Cl_2$ through the top.

NOTE: The concentration step is critical; losses of target compounds can occur if the temperature of the bath is too high or low or the extract is exposed to heat too long.

8.4.11 Place the K-D in a heated water bath set at 85°C so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 2-5 mL, remove the K-D from the bath and allow it to completely cool.

8.4.12 When the K-D has cooled, pour 50 mL of hexane through the top of the Snyder column. Add a fresh boiling chip and shake the Snyder column to make sure that the balls have not stuck. When the water bath has re-heated to 95°C, return the K-D to the water as before. NOTE: losses of target compounds can occur at this step. Make sure solvent loss is rapid (10-15 min). Do not allow the extract to evaporate for more than 15 min.

8.4.13 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of hexane. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of hexane. Be sure to rinse all of the ground glass joints well, as compounds collect on the ground glass.

8.4.14 Carefully concentrate the extract to 10.0 mL under a gentle stream of nitrogen using the N-evap apparatus. If the extract is highly colored, forms a precipitate, or stops evaporating, consult a supervisor. Transfer the extract to a labeled 12 mL screw-top vial with a Teflon lined cap, mark the meniscus, and place on a rack with other samples with the same project number, if any. Include the following information on the label: project and sample number, matrix, parameter, solvent, final volume, your initials, and date of final concentration.

8.4.15 If the extract is colored or a precipitate forms, organochlorine pesticide extracts will need a florisil column cleanup; see Section 8.5. If the extract is to be analyzed for PCBs only, the appropriate cleanup procedure may be found in Section 8.6. If there is any question, consult a supervisor.

8.4.16 Complete the prep sheet for the extraction and concentration steps.

8.5 Extraction of Oil Samples by Acid Digestion

8.5.1 Place a rinsed 50 mL ground glass stopper graduated cylinder on the top loading balance and zero the balance.

8.5.2 Mix samples well to ensure a representative aliquot.

8.5.3 Weigh 1.0g of sample oil into the graduated cylinder and note the weight on the prep sheet.

8.5.4 Dilute the sample to 10.0 mL with hexane.

8.5.5 The total volume for all samples should be 10.0 mL. Note this volume on the prep sheet.

8.5.6 Bring the total volume to 50.0 mL with concentrated $H_2SO_4$. Wear appropriate protective clothing.

8.5.7 Stopper the cylinder and shake it vigorously for at least one minute.

8.5.8 Allow the layers to separate and wait at least 5 minutes after the apparent separation. Some separations may require a wait of 2 hours or more. Record the volume of hexane recovered as measured by the difference in the graduated cylinder.

8.5.9 Pipet the hexane layer into a 12 mL screw top vial with a Teflon lined cap.

8.5.10 The acid goes into "PCB Contaminated Acid Waste;" all of the glassware is to be treated as PCB contaminated.

8.5.11 Label the vial as "Acid Washed," mark the meniscus, and store it in the refrigerator to be column cleaned.

8.6 Florisil Cleanup for Pesticide Samples

8.6.1 Method Summary: A sample extract in hexane is subjected to a florisil adsorption column clean-up to remove interferences and separate pesticides into discrete

fractions. The extract is introduced onto the florisil and eluted into 2 discrete fractions with solvents of varying polarities.

8.6.2 Procedure

Prepare the eluting solvents in 250 mL stoppered graduated cylinders.

Fraction 1: 200 mL 6% diethyl ether in petroleum ether
Fraction 2: 200 mL 15% diethyl ether in petroleum ether
Fraction 3: 200 mL 50% diethyl ether in petroleum ether

8.6.3 A column performance standard, OCP CPS, accompanies every set of pesticide florisil columns.

8.6.4 Place a glass wool plug in the bottom of a 20 mm I.D. chromatographic column. Add 20 g of florisil, mesh size 60-100.

8.6.5 Tap the top of the column with a rubber stopper to settle the florisil. Add 1 cm of dried $Na_2SO_4$ to the top of the column and tap again.

8.6.6 Pre-elute the column with 30-50 mL of petroleum ether. Collect the eluate in a waste beaker. Just prior to the exposure of the $Na_2SO_4$ to the air, quantitatively transfer 10.0 mL (for industrial samples) of sample extract onto the top of the column. Continue collecting in a waste beaker.

8.6.7 Just prior to the exposure of the $Na_2SO_4$ to the air, add Fraction 1. Collect this eluate in a rinsed 400 mL beaker labeled "Column Cleaned-A".

8.6.8 Just prior to the exposure of the $Na_2SO_4$ layer to the air, add Fraction 2. Collect this eluate in the same beaker as above until the solvent volume reaches about 300 mL. Replace beaker with another rinsed beaker labeled "Column Cleaned-B".

8.6.9 Again, just prior to the exposure of the $Na_2SO_4$ to the air, add Fraction 3. Continue collecting the eluate in Beaker B.

8.6.10 Pour the eluate from Beaker A into a Kuderna-Danish (K-D) flask. It is not necessary to pass the eluate through $Na_2SO_4$.

8.6.11 Add a boiling chip to the K-D. Attach a 3-ball Snyder column and prewet by adding about 1 mL of petroleum ether through the top.

8.6.12 Place the K-D on a heated water bath set at 85°C so that the receiver tube is partially immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, remove the K-D from the water and allow it to cool.

8.6.13 After the flask has cooled, add the eluate from Beaker B. Add a fresh boiling chip and repeat the procedure.

8.6.14 Add 50 mL of hexane through the top of the Snyder column. Add a fresh boiling chip to the flask and shake the Snyder column to make sure the balls are not stuck.

8.6.15 When the bath has re-heated to 95°C, return the K-D to the water and concentrate as before, but this time to an apparent volume of 2-5 mL.

8.6.16 After the K-D has completely cooled, rinse the column and the middle flask with a small amount of hexane. Adjust the final volume to equal the volume of extract that was cleaned. Mark the meniscus, label the vial, and place the extract with the others in the same project, if appropriate.

8.7 Alumina/Silica Cleanup for PCB Samples

8.7.1 Method Summary: Interferences present in Polychlorinated Biphenyl (PCB) sample extracts in hexane are removed using a basic alumina and silica gel column clean-up. The chlorinated hydrocarbons are selectively eluted with a moderately polar solvent.

8.7.2 Materials:

8.7.2.1 Brockman Basic Alumina, Activity I, 80-200 mesh, or equivalent, 20g/sample. Deactivated alumina with carbon-filtered water at 0.5% by weight (w/w) (i.e., 100g alumina, 0.5g CF water). Store in a tightly capped glass bottle. Label bottle, including alumina type with mesh size and deactivation date. Re-activate and prepare at least every 10 days.

8.7.2.2 Acidified silica gel, 100-200 mesh, 6g/sample. Add $H_2SO_4$ (conc.) at 40% by weight (w/w) to the silica gel (i.e., 100g silica gel, 40g $H_2SO_4$) and shake well to distribute evenly. Silica gel should be activated prior to acidification at 200°C overnight and stored in the dessicating cabinet.

8.7.2.3 Anhydrous $Na_2SO_4$, 4g/sample. Dried at 400°C for 4 hr in furnace or 300°C in oven overnight.

8.7.2.4 Eluant, 20% methylene chloride in hexane, 50 mL/sample.

8.7.3 Procedure for Oil Samples:

8.7.3.1 The sample should first be acid-washed. One wash is generally enough; the sample does not need to be clear after the acid wash procedure.

NOTE: Record all cleanups, volumes, and weights in the record book and on the benchsheets.

8.7.3.2 Column preparation: Add 20g 0.5% deactivated basic alumina to a 20mm I.D. chromatographic column plugged with glass wool. Tap column to settle. Add 4g dried $Na_2SO_4$ (do not tap column), followed by 6g of acidified silica gel. Tap to settle.

8.7.3.3 Pre-elute the column with 40 mL of hexane. Collect the rinsate in a waste beaker.

8.7.3.4 Add 3.0 mL of the sample extract to the head of the column.

8.7.3.5 After the silica gel becomes exposed to the air, pre-elute the sample with 40 mL of hexane. Collect the rinsate in a waste beaker and discard as oil-contaminated.

8.7.3.6 Just as the silica gel becomes exposed to the air, exchange the waste beaker with a 50 mL graduated centrifuge tube (calibrated at 5.0 mL), which is the final sample collector.

8.7.3.7 Carefully add the 50 mL of 20% methylene chloride in hexane eluant, rinsing the column wall well.

8.7.3.8 After elution, columns, packing materials, glassware and waste must all be considered PCB contaminated.

NOTE: If time permits, continue with the concentration step; otherwise, double-wrap each sample with aluminum, seal with a rubber band, and store in the concentration refrigerator. Indicate at what place in the procedure you stopped on the benchsheet and in the record book.

8.7.3.9 Equilibrate the N-evap to 40°C. Immediately after the samples are placed on the N-evap, increase the bath to 63°C to ensure total removal of $CH_2Cl_2$. To prevent oils and PCB's (PCB's have a high affinity for glass) from sticking to the glass, continually rinse down the graduated centrifuge tube with hexane. Concentrate to slightly less than 5 mL. Before adjusting to a final volume of 5.0 mL, allow the samples to cool to room temperature.

NOTE: Label all sample vials as "column cleaned" before storing in the Pest/PCB extract refrigerator.

8.7.4 Procedure for Soils, Sludges and Water Samples

8.7.4.1 If the sample is extremely dark, it must be acid washed prior to column clean-up. If the sample extract is clear or only slightly discolored, an acid wash is not necessary. For acid washes a 10.0 mL aliquot of the extract should be used.

NOTE: Record all clean-ups, volumes and weights in the record book and on the benchsheet.

8.7.4.2 Column preparation: Add 20g of 0.5% deactivated basic alumina to a 20mm I.D. chromatographic column plugged with glass wool. Tap column to settle. (If alumina leaks from the tip of the column, repack the column or add more glass wool to the tip.) Add 4g dried $Na_2SO_4$ (do not tap column), followed by 6g of acidified silica gel. Tap to settle.

8.7.4.3 Pre-elute the column with 40 mL of hexane. Collect the rinsate in a waste beaker.

8.7.4.4 Add 5.0 mL of the sample extract to the head of the column

8.7.4.5 After the silica gel becomes exposed to the air, pre-elute the sample with 40 mL of hexane. Collect the rinsate in a waste beaker and discard as oil-contaminated.

8.7.4.6 Just as the silica gel becomes exposed to the air, exchange the waste beaker with a 50 mL graduated centrifuge tube (calibrated at 5.0 mL), which is the final sample collector.

8.7.4.7 Carefully add the 50 mL of 20% methylene chloride in hexane eluant, rinsing the column wall well.

8.7.4.8 After elution, columns, packing materials, glassware and waste must all be considered PCB contaminated.

NOTE: If time permits, continue with the concentration step; otherwise double-wrap each sample with aluminum, seal with a rubber band, and store in the concentration refrigerator. Indicate at what place in the precedure you stopped on the benchsheet and in the record book.

8.7.4.9 Equilibrate the N-evap to 40°C. Immediately after the samples are placed on the N-evap, increase the bath to 63°C to ensure total removal of $CH_2Cl_2$. To prevent oils and PCB's (PCB's have a high affinity for glass) from sticking to the glass, continually rinse down the graduated centrifuge tube with hexane. Concentrate to slightly less than 5 mL. Allow the samples to cool to room temperature. Adjust to a final volume of 5.0 mL.

NOTE: Label all sample vials as "column cleaned" before storing in the Pest/PCB extract refrigerator.

9. Analysis

9.1 Calibration of Instrument

9.1.1 Five point calibration curves are generated for all analytes except the multi-peak components (PCBs, chlordane, toxaphene), which have single point calibrations. Most compounds are linear in the range 0.005-0.25 ug/mL. Correlation coefficients $\geq$0.995 are expected for each analyte. External standard tables are used to quantify samples.

9.1.2 Run a mid-level check standard after every 10th injection and at the end of the sequence. Precede each check standard with a solvent blank. The change in response factors (RF), as defined by an RPD calculation, should be <20%. The calculation is performed as follows for each analyte.

$$\left|\frac{X_1 - X_2}{X_1}\right| (100\%) = RPD,$$

Where $X_1$ = area of first standard
$X_2$ = area of later standard

9.1.3 EVALB is the name given to the check standard run at the beginning of every sequence. It contains aldrin, endrin, p,p'-DDT, and dibutylchlorendate at 0.10 ug/mL. This standard is used to inspect the general performance of the chromatographic system by checking peak shape and on-column degradation of endrin and p,p'-DDT, and to set the scale for the Chromatography System.

9.1.4 The analyst should examine the shape of all peaks to ascertain that they are generally symmetrical. Tailing peaks indicate that the system requires routine maintenance.

9.1.5 Degradation of endrin or p,p'-DDT on the column is limited by the EPA to <20% of either compound and this criterion is applied to all work. By the time column degradation is approaching these values, maintenance as described above is mandatory.

9.1.6 Degradation in the EVALB mixture is calculated as follows:

Using area counts, calculate endrin degradation as a percentage of total endrin:

$$\frac{\text{endrin aldehyde + endrin ketone}}{\text{endrin + endrin aldehyde + endrin ketone}} \times 100\%$$

Similarly, calculate the degradation of p,p'-DDT:

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

9.1.7 There are no requirements for allowable changes in sensitivity for individual pesticide analyses. However, we impose CLP requirements. That is, changes in RFs on the quantitation column may vary by no more than 15% and on the confirmation column by no more than 20% throughout the course of the run. If these limits are exceeded, the run is stopped. Affected data is analyzed using the closest standard. The instrument must be recalibrated.

9.2 GC Conditions

9.2.1 Second column confirmation is required for all samples that contain any target compounds. Dissimilar columns may be operated simultaneously.

9.2.2 Analytical Conditions for Hewlett-Packard 5880 or 5890 equipped with 2 electron capture detectors and fitted with 2 megabore capillary columns as follows:

Column A: 007-608 (Methyl phenyl cyanopropyl silicone) 30m, 0.53mm I.D., 1.0 u film thickness.

Column B: 007-DB5 (Methyl phenyl 5% silicone) 30m, 0.53mm I.D., 1.0 u film thickness.

The following conditions are suggested for analysis with an H-P 5890 GC with dual megabore split capillary system.

| | | |
|---|---|---|
| Injection temperature | : | 200°C |
| Initial temperature | : | 60°C |
| Initial time | : | 2 min |
| Program rate | : | 40°C/min |
| Final temperature | : | 200°C |
| Final time | : | 2 min |
| Prog rate 2 | : | 3°C/min |
| Final temperature 2 | : | 230°C |
| Final time 2 | : | 2 min |
| Prog rate 3 | : | 2°C/min |
| Final temperature 3 | : | 240°C |
| Final time 3 | : | 10 min |
| Detector temperature | : | 325°C |
| Carrier flow | : | 7-10 mL/min $H_2$ |
| Makeup gas | : | $N_2$ |
| Total flow | : | 40-50 mL/min |
| Run time | : | 32 min. |

10. QC/QA Requirements

10.1 For a standard products test duplicate lab control samples (blank spikes) are generated for every 20 samples and a single control sample (method blank) is generated with every prep and analysis set. Project-specific Q.C., usually matrix spikes or duplicates, may be requested.

10.2 Acceptance limits for Q.C. follows Enseco's Internal QC program; 80% of the accuracy values and 80% of the precision values must be acceptable for analysis to proceed.

## METHOD 608
## ORGANOCHLORINE PESTICIDES by GC
## AQUEOUS MATRIX

| Analytes | Spike Conc. | Units: ug/L Measured LCS 1 | Conc. LCS 2 | \|-----Accuracy-----\| % Recovery LCS 1 | LCS 2 | Control Limits | \|-Precision-\| RPD | Control Limits |
|---|---|---|---|---|---|---|---|---|
| Lindane | 0.2 | ___ | ___ | ___ | ___ | 56-123 | ___ | 15% |
| Heptachlor | 0.2 | ___ | ___ | ___ | ___ | 40-131 | ___ | 20% |
| Aldrin | 0.2 | ___ | ___ | ___ | ___ | 40-120 | ___ | 22% |
| Dieldrin | 0.5 | ___ | ___ | ___ | ___ | 52-126 | ___ | 18% |
| Endrin | 0.5 | ___ | ___ | ___ | ___ | 56-121 | ___ | 21% |
| 4,4'-DDT | 0.5 | ___ | ___ | ___ | ___ | 38-127 | ___ | 27% |

## METHOD 8080
## ORGANOCHLORINE PESTICIDES by GC
## SOLID MATRIX

| Analytes | Spike Conc. | Units: ug/L Measured LCS 1 | Conc. LCS 2 | \|-----Accuracy-----\| % Recovery LCS 1 | LCS 2 | Control Limits | \|-Precision-\| RPD | Control Limits |
|---|---|---|---|---|---|---|---|---|
| Lindane | 27 | ___ | ___ | ___ | ___ | 46-127 | ___ | 50% |
| Heptachlor | 27 | ___ | ___ | ___ | ___ | 35-130 | ___ | 31% |
| Aldrin | 27 | ___ | ___ | ___ | ___ | 34-132 | ___ | 43% |
| Dieldrin | 67 | ___ | ___ | ___ | ___ | 31-134 | ___ | 38% |
| Endrin | 67 | ___ | ___ | ___ | ___ | 42-139 | ___ | 45% |
| 4,4'-DDT | 67 | ___ | ___ | ___ | ___ | 23-134 | ___ | 50% |

**METHOD 608**
**POLYCHLORINATED BIPHENYLS (PCBs) by GC**
**AQUEOUS MATRIX**

| Analytes | Spike Conc. | Units: ug/L Measured LCS 1 | Conc. LCS 2 | \|-----Accuracy-----\| % Recovery LCS 1 | LCS 2 | Control Limits | \|-Precision-\| RPD | Control Limits |
|---|---|---|---|---|---|---|---|---|
| Aroclor 1254 | 5.0 | ___ | ___ | ___ | ___ | 20-160 | ___ | 20% |
| | | ___ | ___ | ___ | ___ | | ___ | |
| | | ___ | ___ | ___ | ___ | | ___ | |
| | | ___ | ___ | ___ | ___ | | ___ | |
| | | ___ | ___ | ___ | ___ | | ___ | |
| | | ___ | ___ | ___ | ___ | | ___ | |

**METHOD 8080**
**POLYCHLORINATED BIPHENYLS (PCBs) by GC**
**SOLID MATRIX**

| Analytes | Spike Conc. | Units: ug/L Measured LCS 1 | Conc. LCS 2 | \|-----Accuracy-----\| % Recovery LCS 1 | LCS 2 | Control Limits | \|-Precision-\| RPD | Control Limits |
|---|---|---|---|---|---|---|---|---|
| Aroclor 1254 | 167 | ___ | ___ | ___ | ___ | 20-160 | ___ | 20% |

Corrective Action: See Current Q.C. manual

11. Calculations

For Waters: $\frac{\text{sample conc ug/mL X final volume (mL) X dilution factor}}{\text{L extracted}}$

For Soils: $\frac{\text{sample conc ug/mL X final volume (mL) X dilution factor}}{\text{Kg extracted}}$

12. Reporting

12.1 Reporting units. Aqueous samples are reported in ug/L. Solid samples are reported in mg/kg.

12.2 Reporting limits are test specific. Very often the reporting limit is equivalent to the detection limit. For tests such as A010CP RCR and A160CPTCLP the reporting limit is based on the regulatory limit.

12.3 Results are reported to 2 significant figures unless a client specifies otherwise. Values below the detection limit are reported as ND-not detected.

12.4 LIMS data entry is usually done by the analyst, and reviewed by data review section.

13. Method Source

13.1 Federal Register Vol. 49, no. 209, Friday October 26, 1984

13.1.1 Deviations

The 608 method presumes a 3 point calibration curve is run and that subsequent calibration checks are within 10% of the original curve. We run a 5 level curve of all analytes of interest. We are satisfied with the results if the subsequent standards are within 20% of the curve. The rational is:

13.1.1.1 It is unrealistic to run 30 standards before analyzing any samples.

13.1.1.2 The 10% window is too tight. EPA only requires 15% and 20%.

13.2 USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, November, 1986, SW-846, Third Edition.

## APPENDIX A

Appendix A. Analysis is run with the Nelson system:

1. Generate a sequence file (sample table) on the Compaq:

   From DOS type MENU (return). From the main menu enter "3".

   Sequence file: file names are up to 10 characters, ending in "A" for column A and "B" for column B e.g. INDPESTA, THMB. Use no punctuation except for periods.

   Sample names: the field is sufficiently large to provide complete description. Use no punctuation except for periods. e.g. TOXAPHENE STD 0.25 UG PER mL

Method name: AEPI for Column A
BEPI for Column B

Method names are up to 10 characters. Our naming convention is that the first character specifies the column (A or B), the second specifies the instrument, (in this case E), and the remainder should be relevant to the analysis. PI in AEPI stands for "pesticides, individual stds".

Data file name: up to 10 characters. The first letter followed by a colon designates the data storage disk currently in use. Our naming convention again specifies column and instrument as the first 2 characters after the colon followed by 3 numbers for the Julian date (001 to 366) e.g. D:AE080. To save the sequence file, enter F9 then enter the sequence file name. When the next screen comes up enter F10,YES.

Where the system is set up so that both GC channels are fed into 1 Nelson interface, it is only necessary to generate a sequence file for Column A. The system will automatically apply this list to Column B.

2. On the 5880 integrator, use CHANNEL B with SIGNAL B ENTER keyed into the integrator.

   GET PROGRAM "NELSON B" DEVICE 16.

3. Download a Nelson interface box:

   On the Compaq from the main menu enter option 0, acquire data. Then choose F2 DOWNLOAD. If using a sequence file, enter YES and name of file including drive specification, e.g. D:INDPESTA. As the method files are all stored on drive M:, enter M:AEPI (e.g.) Enter desired parameters to the questions which follow.

   Two channel acquisition is specified in method M:AEPI. Once the interface is downloaded for channel A, the system automatically seeks method M:BEPI for channel B and automatically labels the samples on column B and stores runs from column B in _:BE_ _ _ . Stored runs are sequentially numbered and should match run numbers e.g. D:AE0801=run 1, D:AE0802=run 2, etc.

4. Always run a PS005C first. When it has run, return to main menu (F10), select 1 to modify M:AEPI. When the METGEN MENU is displayed, choose option 2 and change the millivolt settings to the desired values. The value for "plot" is that for column A and the value for "Ch 1" is that for column B.

   Exit using F9, YES to overwrite and F10 followed by YES to return to main menu.

## APPENDIX B

Appendix B: Collecting and reducing all data with the Nelson system

1. Data collection:

Once the Nelson interface box is collecting data, the Compaq should be left in the acquire mode (0) in order that data is sent to disk and spooled to the printer as it is generated.

A. Setting up a library:

To reduce Nelson data, it is necessary to have a library against which to compare. Libraries are all on drive L:, access to libraries from DOS is gained by typing EDIT. Usually, each analyst has "private" libraries for individual work. Such libraries are prefixed by the analyst's initials and the privacy of these libraries should be respected. This is not to say they cannot be copied. For HP5880 instrument E, the base library is PEST101 for column A and PESTMIX for column B. To set up a new library from DOS, type:

COPY L;PEST101.LIB L:? ? ? 101.LIB and
COPY L:PESTMIX.LIB L:? ? ? MIX.LIB

The libraries typically have complete organochlorine pesticide lists, including irregularly used compounds such as the o,p'-series of DDT and its metabolites and the additional compounds required by Appendix IX.

It is important that each compound in the library have a correct retention time and reasonable retention time window.

To enter the program, type GCCOMP from DOS. The first screen gives 12 questions to be answered. Data drive, instrument, date, start cycle, end cycle are all obvious. For print standards answer yes. The sequence file does not require a drive to be specified. Libraries are usually set up for each instrument. Currently this program does all calculations on a single point, the mid-level standard which is always level 3 in the libraries. And, no, you do not wish to abort the program.

The program automatically goes through the cycles specified, first identifying standards and the components expected in them and updating the library with the new areas and the retention times. The program repeats the process with the sample runs, identifying hits and quantitating hits on both columns. Finally, it compares hits from both columns and procedures report of compounds found on both columns with calculated amounts and percent deviation from the expected retention time.

2. Nelson Libraries

All libraries are stored on drive L:, and access is gained from DOS through the EDIT program. With this program, it is possible to add (or delete) compounds to a library, change retention times and windows and method of quantitation.

Type EDIT from DOS and hit return when the screen shows Default drive = L: The option screen will show three options.

EDIT OLD LIBRARY
CREATE NEW LIBRARY
PRINT LIBRARY CONTENTS

Use the arrow keys to move the highlighted line and enter the desired option.

A. Edit Old Library

The screen will show available libraries. The basic libraries for instrument E are:

PEST101.LIB containing pesticide data for SP2100 column

PESTMIX.LIB containing pesticide data for the SP2250/SP2401 column

Use F2 to indicate which library is to be edited. The space at the end of the line is highlighted (LIBRARY:). Enter the library name, then F3, proceed and library entry 001 will come up on the screen.

Each entry consists of 2 pages and F1 allows you to go between pages. Page 1 contains RT information. Entries may be changed by moving the highlighted box using arrow keys and typing over the current entry. It is necessary to change absolute retention times if chromatographic conditions change significantly. Absolute RT are used for pesticide analyses and these entries rarely must be changed. Again, 'SINGLE' is the method primarily used for pesticide quantitation although there are 4 choices:

Average: The response factors of all active levels will be averaged and the average response factor will be used to calculate concentration.

Line: A least squares regression line ("best fit") will be drawn to encompass all active levels and the slope of this line will be the response factor used to calculate concentration.

Interpolation: Lines will connect all active levels (point-to-point) and the concentration will be calculated using the slope of the appropriate line segment as the response factor.

Single: The response facto from a single calibration level will be used to calculate concentration. The level to be used is specified in the next line "Calculation level."

Page 2 contains quantitative information. There are 7 levels of calibration possible for each entry with the first being 0,0.00. Amounts must be entered on this page. Entries for level 3 are quantities for mid-level standards (e.g. CM019C) and entries for level 4 are quantities for B-level standards (e.g. CM019B). The absolute response factor and reference data file are updated automatically by using the REPORT program.

When all information for an entry is correct exit that entry by F2. This exits but remains in the library so that other entries can be edited.

List entries - lists library entries on the screen
Print entries - makes a hard copy of the list of library entries
Change entries - allows you to change info on another entry
Add entry - appends an entry to the library
Exit to Edit

To change another entry, move highlight to "Change Entry" and enter. Answering YES to save last entry, saves any changes, NO does not. Enter the number of the library entry you wish to go to next (1-100). Exit using F3.

B. Create New Library

The screen will display a directory of libraries currently stored on Drive L: Use F2, create library and enter the name you wish to give the new library in the highlighted box. Then push F3-proceed.

Each entry has 2 pages of information associated with it. See section 1 for detailed information. Fill in both pages for all entries desired.

Use F3 to exit and save the new library.

C. Print Library Contents

This prints both pages for each library entry and can run to quite a few pages. The screen displays the libraries stored on drive L: Use F2 and enter the name of the desired library in the highlighted box. Enter F3, proceed. You have a choice of printing all entries or a range of sequential entries.

It is important to be aware that any library is accessible to all comers. If two people try to use the same library, collisions will inevitably occur. To avoid this, establish your own library by copying the base library.
B> COPY L:PEST101.LIB L:MJW101.LIB. Libraries prefixed by such initials are for the owner's use only.

3. Reprocessing Nelson Data

If chromatograms are missing but the data is available on disk (only the .PTS and .HDR file are needed); they can be reconstructed and .ATB generated using option 10 of the Nelson menu - batch reprocessing.

The answers to the questions on the screen are fairly self-evident. The base file name must include drive specification e.g. F:AE083. The default method file name is the method file to be used for integration.

When all the data has been spooled to the printer, the screen will display the status of all the Nelson interfaces. Activate the printer by simultaneously pressing F1.

Occasionally, both .ATB and .HDR files may be missing, but so long as a .PTS file is in memory, all is not lost. A .HDR file can be copied and, while some information may have to be corrected by hand, this copy and the .PTS file can be used in batch reprocessing to generate a chromatogram and .ATB file. To copy a header file:

```
B:/>COPY F:AE08013.HDR F:BE08013.HDR
```

If it is necessary or desirable to reintegrate peaks or to plot them at a different scale, this may be done using option 14 of the Nelson menu - Process Disk Data.

Once the raw data file has been entered, default values for threshold, peak width may be accepted. The timed events may or may not be edited-generally not.

After some working of the computer, a new screen will ask for a new area reject and whether or not to print a new area percent table. Internal and external standard tables are not printed for current use.

Usually the default values are accepted for amount injected and dilution factor are accepted.

The number of pages for replot permits compressing or expanding the horizontal axis of a plot. This is sometimes useful for making it easier to see complex chromatograms and to examine how baselines are drawn. Accepting the default value will leave the horizontal axis unchanged.

Answer "yes" to get a hard copy only if a copy of the original chromatogram is desired. At this point, peaks are not identified and named.

The first screen plots the first portion of the chromatogram. If this segment does not contain the peak of interest use F10 to proceed to the next segment or until the segment of interest is reached.

The first screen plots the first portion of the chromatogram. If this segment does not contain the peak of interest use F10 to proceed to the next segment or until the segment of interest is reached.

To change the way a peak is integrated (to redraw a baseline) move the cursor using the arrow keys on the right side of the keyboard. Move the cursor to the desired baseline point at the beginning of the peak and press F7 (point 1). Then move the cursor to the desired baseline at the trailing edge of the peak and press F8 (area). The new area will be printed alongside the reintegrated peak. To insert this number into the area table replacing the old value, press F9 (replace). The peak number to be entered is the appropriate peak number from the Nelson chromatogram.

A hard copy of the desired screen is obtained by pressing F6, then F1. The next screen provides the opportunity to replot the chromatogram larger or smaller than the original, stretched or compressed, or a combination. A range of times or the entire chromatogram may be replotted on as many pages (screens) as desired. Names are not put on the plot, offset is usually 0, screens are not hard copied until desired. Absolute mV full scale permits "electronic dilution." Replotting a chromatogram at 200mV which was originally plotted at 20mV will result in a 10-times down-scaling. Chromatograms may be re-scaled, reintegrated and hard copied as many times as desired before exiting the program. Screens that are hard copied by F6 are not printed until F1 is pressed and may be accumulated.

STANDARD
OPERATING
PROCEDURE

Subject or Title: 

GC/MS Analysis of Semivolatile Organics

| SOP No.: LM-RMA-3013 BNA 625 | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes: Original and LM-RMA-3002

1. Scope and Application

Capillary GC/MS is used to analyze a wide range of extractable organic compounds in a variety of matrices. Although the method for preparation of samples is dependent on the matrix, most aspects of the instrumental analysis are not dependent on the matrix or compounds to be determined, consequently the scope of the method may be extended to include quantitative analysis of extractable, chromatographable compounds not explicitly listed in Appendix A provided they are amenable to analysis as semivolatiles and a standard is analyzed and evaluated for those compounds.

Several Analytical Test References are contained in Appendix F. The Analytical Test References document analyses currently performed at RMAL which deviate in target compound list, analytical protocol and/or data package requirements from those specified in Appendix A and Sections 8.7 through 12.3.

The applicability of this Standard Operating Procedure LM-RMA-3013 to particular matrices and/or analytical requests not specifically stated in this S.O.P. is determined by the GC/MS department manager and communicated to the client manager. The GC/MS department manager has the authority to make modifications to the S.O.P. for certain matrices and/or analyses if his technical judgment deems it necessary and appropriate.

1.1 Analytes: Quantitative analysis is performed for the compounds listed in Appendix A. Qualitative analysis by mass spectral identification can be performed on other extractable, chromatographable compounds.

| Prepared by: | Date: 12-4-89 |
|---|---|
| Management Approval: | Date: 2/15/90 |
| QA Officer Approval: | Date: 2-23-90 |

1.2 Reporting Limits: For most compounds, the instrument reporting limit is 10 ug/mL. The extraction recovery of some compounds and consequently their detection limits depend on the sample matrix (e.g. soil, water, waste). The detection limit also depends on the characteristics of individual samples. All aspects of this S.O.P. assume target compounds will be found and quantitated at levels within the specified range of calibration for each analysis. Therefore, if a sample contains a high concentration of target compounds or a large amount of interfering material, it will be diluted prior to analysis. This increases the reporting limit proportionally to the dilution. If the sample extract is diluted to 10% (i.e. 1:10 dilution) or less, the surrogates and spiked compounds will be diluted to levels below reporting limits and will not be reported. Refer to Appendix A for HSL (Hazardous Substance List) and PP (Priority Pollutant) semivolatiles and their nominal reporting limits for each matrix. Refer to Appendix F for summaries of other common analyses performed at RMAL. Included in each Analytical Test Summary is the typical range of calibration for analytes.

1.3 Applicable matrices: Water, soil, solids, sludge, waste and TCLP leachates (Toxicity characterization leaching procedure.)

1.4 Dynamic range: Analytes can typically be quantitated between 5ug/mL and 160 ug/mL. Samples which exceed this concentration range for any analyte in Table A-1 should be diluted to be within the dynamic range of the method. Sample extracts should also be diluted when any other semivolatile component of the sample exceeds this range. Reporting limits will be adjusted to reflect the dilution performed.

1.5 Analysis time: Approximate analytical time is 50 minutes per GC/MS run. The time required for data reduction will be dependent upon the complexity of the sample.

2. Method Summary

2.1 Aqueous samples are extracted with dichloromethane (methylene chloride) into base/neutral and acid fractions. The base/neutral and acid fractions are concentrated and combined for quantitative and/or qualitative capillary GC/MS analysis.

2.2 Solid samples are extracted with an appropriate solvent. The extract is concentrated and then analyzed by capillary GC/MS for quantitative and/or qualitative identification.

3. Comments

3.1 All deviations made from this SOP should be approved by the supervisor and be well documented.

3.2 All reusable glassware should be solvent rinsed prior to use.

3.3 Care should be taken not to introduce contamination into the samples. Particular attention should be paid to selecting a protective glove that does not contribute contamination during sample preparation.

3.4 pH adjustments required for the preparation of aqueous samples are critical. Analyte recoveries can be adversely affected if the pH of the sample is not sufficiently basic or acidic.

4. Safety Issues

4.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely defined, however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible.

4.2 A Material Safety Data Sheet (MSDS) is available for all laboratory standard and reagent chemicals. The appropriate MSDS must be read before handling the chemical(s).

4.3 All laboratory personnel should be thoroughly familiar with the laboratory Safety Manual and Hazard Communication Standard before undertaking any laboratory work.

5. Sample Collection, Preservation, Containers and Holding Times

5.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed, except that the bottle must not

be pre-rinsed with sample before collection. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination. Solid samples should be collected in wide mouth glass jars.

5.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction.

5.3 All aqueous samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction. Soil/waste samples must be extracted within 10 days of collection and completely analyzed within 40 days of extraction.

5.4 Refer to Enseco, Inc. QA Program Plan for Environmental Chemical Monitoring Rev. 3.3, Sections 4-5 and Appendix I for more information.

6. Apparatus

6.1 Separatory funnel - 2 L, with Teflon stopcock.

6.2 Filtering Funnel - 75 mm top diameter.

6.3 Pyrex glass wool

6.4 Disposable 1 mL pipet

6.5 Concentrator tube, Kuderna-Danish - 10 mL graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

6.6 Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

6.7 Snyder column, Kuderna/Danish - Three ball macro (Kontes K-503000-0121 or equivalent).

6.8 Vials - 2 mL capacity with Teflon-lined screw cap suitable for GC autosampler.

6.9 Boiling chips - Approximately 10/40 mesh.

6.10 Water bath - Heated, with concentric ring over cover, capable of temperature control (± 2°C). The bath should be used in a hood.

6.11 Toploading balance capable of accurately weighing 0.01 g.

6.12 Analytical balance capable of accurately weighing 0.0001 g.

6.13 Spatula - stainless steel or teflon

6.14 Sonicator - A horn-type sonicator equipped with a titanium tip should be used.

6.15 Sonabox - Recommended with above disrupters for decreasing cavitation sound.

6.16 Gas Chromatograph for screening - A capillary chromatograpny system suitable for splitless injection and all required accessories such as syringes, analytical columns, gases, flame ionization detector, and recording device. A data system is recommended for data acquisition, integration and output.

6.17 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C.

6.18 Continuous liquid-liquid extractors-equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication.

6.19 Capillary column. One of the following or an approved substitute should be used.

6.19.1 J&W 30 m DB-5, 0.25 mm id, 0.25 um film thickness

6.19.2 Quadrex 25 m Methy Phenyl (5%) Silicone, 0.25 mm id, 0.5 um film thickness.

6.19.3 Restec 30 m RTX-5, 0.32 mm id, 1.0 um film thickness.

6.20 Syringes and/or micro pipets - 10 uL, 25 uL, 50 uL, 100 uL, 500 uL and 1 mL, as appropriate for making injections, adding internal standard and preparing sample dilutions.

6.21 Gas Chromatograph/Mass Spectrometer

6.21.1 Gas chromatograph: An analytical system that includes a temperature-programmable gas chromatograph suitable for splitless injection including all required accessories such as syringes, columns, and gases.

6.21.2 Mass spectrometer: Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

6.21.3 GC/MS interface: An interface that allows direct coupling of the capillary column and adequate analyte response.

6.21.4 Data system: A computer system must be interfaced to the mass spectrometer which will allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the analytical run. The software should be capable of producing extracted ion profiles and integrating these abundances. The EPA/NIH (NBS) Mass Spectral Library should also be available on the system.

7. Reagents and Standards

7.1 Reagent water - Carbon filtered house distilled water. Interferents not observed at or above the reporting limit of the parameters of interest.

7.2 Sodium hydroxide solution (10 N)-Dissolve 40 g NaOH in reagent water and dilute to 100 mL.

7.3 Sulfuric Acid (1+1)-Slowly add 50 mL of $H_2SO_4$ (ACS sp. gr. 1.84) to 50 mL of reagent water.

7.4 Acetone, methanol, methylene chloride - Pesticide quality or equivalent.

7.5 Sodium sulfate-(ACS), anhydrous. Granulated for drying extracts, powdered for extracting soil samples.

7.6 Surrogate standard spiking solution. (See Appendix B)

7.7 Laboratory Control Sample standard spiking solution. (See Appendix B).

7.8 GC calibration standard. (See Appendix B)

7.9 Internal standard solution. (See Appendix B)

7.10 Calibration standard solutions. (See Appendix B)

8. Procedure

8.1 Scheduling Samples for Extraction

8.1.1 Samples will be scheduled for extraction by the section supervisor. Samples should be scheduled for preparation according to the following priorities:

a) Meet holding times

b) Meet promised due date

8.2 Preparation of Water Samples

NOTE: ALL GLASSWARE MUST BE SOLVENT RINSED BEFORE USE. RINSE WITH ACETONE AND METHYLENE CHLORIDE.

8.2.1 Emulsion test. This test is performed if a sample contains particulate matter. It is used to determine if the sample will be extracted using a separatory funnel or a continuous liquid-liquid extractor.

8.2.1.1 3-4 mL of sample is transferred to a 12 mL screw top glass vial with a Teflon liner.

8.2.1.2 Approximately 1 mL of $CH_2Cl_2$ is added. The vial should be shaken vigorously for about 30 seconds and the layers allowed to separate.

8.2.1.3 If no emulsion forms, the prep should proceed with the shakeout technique in Section 8.2.2.

8.2.1.4 If an emulsion forms, the prep should proceed with the continuous extractor technique in Section 8.2.3.

8.2.2 Separatory Funnel Extraction (Shakeout Technique)

8.2.2.1 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle, all others are mixed by shaking the bottle.

8.2.2.2 If the sample is in a larger bottle or requires decanting, a 1 L graduated cylinder is used to measure the sample volume. The sample is poured into a 2 L separatory funnel.

8.2.2.3 If the sample does not require decanting and is in a 1 L or smaller bottle, the water meniscus is marked on the side of the sample bottle and used to measure the sample volume. The sample is then poured into a 2 L separatory funnel. After the sample bottle is rinsed with methylene chloride (Section 8.2.2.11), the sample volume used is measured by adding tap water to the bottle to the marked level. The volume added is measured with a graduated cylinder.

8.2.2.4 The volume of each sample is recorded on the benchsheet.

8.2.2.5 For blanks and quality control samples, 1 L of carbon filtered water is poured into the separatory funnel.

8.2.2.6 For matrix specific QC, 1 L of the appropriate sample required for each QC sample is poured into a separatory funnel. If insufficient sample is available to use at least 500 mL per aliquot, the supervisor is consulted. The volume of sample used for each portion is recorded on the benchsheet.

8.2.2.7 1 mL of the surrogate standard spiking solution is added to each sample and QC sample using a 1 mL disposable pipet, making sure the pipet tip is below the surface of the sample while the standard is being added. The sample is mixed immediately by stoppering and shaking the separatory funnel. The addition of the surrogate spiking solution is noted on the bench sheet, including who added it and the date and by whom the standard was made.

8.2.2.8 0.5 mL of the DCS or matrix standard spiking solution is added if appropriate, using the procedure described in 8.2.2.7.

8.2.2.9 The pH is checked by removing a small aliquot of the sample with a pasteur pipet, placing enough of the sample on a pH paper to thoroughly soak the paper. The pH paper is compared with the chart on the container and the initial pH is recorded on the prep sheet.

8.2.2.10 The pH is adjusted to >12 with 10 N NaOH and the adjusted pH is noted on the prep sheet.

8.2.2.11 For samples that were mixed before pouring, 60 mL methylene chloride ($CH_2Cl_2$) is added to the sample container to rinse the inner walls. The solvent is then transferred to the separatory funnel. For samples that were decanted, the first aliquot of $CH_2Cl_2$ is added directly to the separatory funnel.

8.2.2.12 The sample is extracted by shaking it for two minutes with frequent ventilation. The layers are allowed to separate. If there is an emulsion it is broken up using one of several techniques. The optimum technique will depend upon the sample, and may include: stirring, centrifugation, filtration through glass wool or other physical methods. If the emulsion cannot be broken, the sample must be transferred to a continuous extractor. See section 8.2.3.

8.2.2.13 After separation, the bottom layer ($CH_2Cl_2$) is drained into a 400 mL beaker.

8.2.2.14 The extraction is repeated twice more using a 60 mL aliquot of $CH_2Cl_2$ each time. The solvent is collected in the same beaker described in 8.2.2.13.

8.2.2.15 The pH is then adjusted to $< 2$.

NOTE: THIS pH ADJUSTMENT IS CRITICAL. PHENOL RECOVERIES MAY BE LOW IF THE pH IS NOT LESS THAN 2.

8.2.2.16 The extraction is repeated another three times at the acidic pH using a 60 mL aliquot of $CH_2CL_2$ each time. The solvent is collected in the same beaker described in 8.2.2.13.

8.2.2.17 A plug of glass wool is placed in a funnel and the funnel is filled about 2/3 full with granular $Na_2SO_4$. The funnel and $Na_2SO_4$ are rinsed with 30-40 mL of $CH_2Cl_2$ which is discarded after being used for rinsing.

8.2.2.18 The sample extract is poured through the $Na_2SO_4$ into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. The beaker which contained the extract and the $Na_2SO_4$ in the funnel are rinsed with small amounts of $CH_2Cl_2$. These rinses are added to the K-D.

8.2.2.19 A boiling chip is added to the K-D and a 3- ball Snyder column is attached to the top. The column is prewet by adding about 1 mL of $CH_2Cl_2$ to the top of the Snyder column.

NOTE: THE CONCENTRATION STEP IS CRITICAL; LOSSES OF TARGET COMPOUNDS CAN OCCUR IF CARE IS NOT TAKEN.

8.2.2.20 The K-D is placed in a 95°C hot water bath so that the receiver tube is immersed in hot water below the joint and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, the K-D is removed from the bath and allowed to cool completely.

8.2.2.21 When the K-D has cooled, the column and flask are rinsed with a small amount of $CH_2Cl_2$. The extract is transferred to a calibrated 15 mL centrifuge tube, rinsing with a small amount of $CH_2Cl_2$. It is important to rinse all of the ground glass joints of the K-D well, as compounds collect on the ground glass.

8.2.2.22 The extract is carefully concentrated to 1.0 mL under a gentle stream of nitrogen using the N-evap apparatus. The temperature of the water bath should be 35-40°C. If the extract is highly colored, forms a precipitate, or stops evaporating, the supervisor is consulted before the procedure is continued.

8.2.2.23 The extract is transferred to a labelled autosampler vial with Teflon lined cap. The meniscus is marked and the extract is placed on a tray in the BNA extract refrigerator with any other samples having the same QC Lot number. The tray number is recorded on the outside of the prep folder containing the bench sheets.

8.2.2.24 As the final step, the prep sheet is completed for the extraction and concentration steps.

8.2.3 Continuous Extractor

8.2.3.1 The continuous extractor is set up in a hood or well-ventilated area.

8.2.3.2 250 mL of $CH_2Cl_2$ is placed in a round bottom flask and a few boiling chips are added. 300 mL of $CH_2Cl_2$ is added to the extractor flask.

8.2.3.3 500 mL of carbon filtered water is carefully poured into the extractor, minimizing the disturbance of the solvent layer and preventing water from entering into either sidearm by pouring the water down the back of the extractor.

8.2.3.4 The pH of the sample is checked by removing a small aliquot with a pasteur pipet, placing enough of the sample on a pH paper to thoroughly soak the paper. The pH paper is compared with the chart on the container and the initial pH is recorded on the prep sheet.

8.2.3.5 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle, all others are mixed by shaking the bottle.

8.2.3.6 If the sample is in a larger bottle or requires decanting, a 1 L graduated cylinder is used to measure the sample volume. The sample is then carefully poured into the extractor flask.

8.2.3.7 If the sample does not require decanting and is in a 1 L or smaller bottle, the water meniscus is marked on the side of the sample bottle and later used to measure the sample volume. The sample is carefully poured into the extractor flask. After the sample bottle is rinsed with methylene chloride (section 8.2.2.11), the sample volume used is measured by adding tap water to the bottle to the marked level. The volume added is measured with a graduated cylinder.

8.2.3.8 The volume of each sample is recorded on the benchsheet.

8.2.3.9 For blanks and quality control samples, 1 L of carbon filtered water is poured into the extractor flask.

8.2.3.10 For matrix specific QC, 1 L of the appropriate sample required for each QC sample is measured into the extractor flask. If insufficient sample is available to use at least 500 mL per aliquot, the supervisor is consulted. The volume of sample used for each portion is recorded on the bench sheet.

8.2.3.11 Sufficient carbon filtered water is added to the extractor to allow the solvent in the removable sidearm to begin to drip into the round bottom flask. It is important that the extractor stays in a strictly vertical position.

8.2.3.12 1 mL of the surrogate spiking solution is added to each sample and QC sample using a 1 mL disposable pipet, making sure that the tip of the pipet is below the surface of the liquid in the extractor as the standard is being added. The sample is mixed immediately using a glass stirring rod. The addition of the surrogate spiking solution is noted on the bench sheet, including who added it and the date and by whom the standard was made.

8.2.3.13 0.5 mL of DCS or matrix spike mix is added if appropriate, using the procedure described in 8.2.3.12

8.2.3.14 The pH is adjusted to >12 with 10 N NaOH, stirring the sample carefully with a glass rod. If an excessive amount of NaOH is used (more than about 15 mL), this is recorded along with the adjusted pH on the prep sheet.

8.2.3.15 The condenser is wiped clean with solvent at the lower joint and lip and then placed on the top of the extractor. The water chiller (set at 10°C) is turned on and flow indicators checked.

8.2.3.16 The heating mantle beneath the round bottom (rb) flask is turned on and the extraction starting time is recorded on the bench sheet. The extractor is checked after 15 minutes to ensure it is operating correctly and that there are no leaks.

8.2.3.17 The extraction is allowed to proceed for a minimum of eighteen hours.

8.2.3.18 The heating mantle is turned off and the apparatus is allowed to cool (30-60 minutes) with water flowing through the condenser.

8.2.3.19 The solvent contained in the round bottom flask is the base/neutral extract. The flask is removed, capped, refrigerated and replaced with a clean round bottom flask containing 250 mL $CH_2Cl_2$ and a few boiling chips.

8.2.3.20 The condenser is removed and the pH is adjusted to $< 2$.

NOTE: THIS pH ADJUSTMENT IS CRITICAL. PHENOL RECOVERIES MAY BE LOW IF THE pH IS NOT LESS THAN 2.

8.2.3.21 Steps 8.2.3.16-8.2.3.19 are then repeated for the acid extraction.

8.2.3.22 The solvent contained in the round bottom flask is the acid fraction. The round bottom flask is removed, capped and refrigerated.

8.2.3.23 The contents of the extractor are poured into a separatory funnel to separate the water from the methylene chloride. The water layer from the extractor flask is discarded into the sink with plenty

of cold tap water. The solvent layer from the extractor flask is discarded into a contaminated waste bottle.

8.2.3.24 The drying and concentration steps are similar to those described in section 8.2.2.17 to 8.2.2.18. Due to the larger volume of solvent the base/neutral fraction must be concentrated first to an apparent volume of 10-15 mL. Then a new boiling chip is added and the acid fraction is poured through $Na_2SO_4$ into the same K-D. The concentration is then completed as described in sections 8.2.2.19 to 8.2.2.23.

8.2.3.25 As the final step, the prep sheet is completed for the extraction and concentration steps.

8.3 Preparation of Soil Samples

8.3.1 Percent Moisture Determination (optional)

5-10 g of the sample is added to a tared weighing dish. The wet weight of the sample is recorded. The weighed sample is allowed to dry overnight at 105°C and then reweighed. The dry weight of the sample is recorded, correcting for weight of the weighing dish. The percent moisture of the sample is then calculated using equation 10.2.7.

8.3.2 pH Determination (optional)

50 g of sample is transferred to a 100 mL disposable beaker. 50 mL of water and a teflon-coated magnetic stirring bar are added and the beaker is stirred for one hour on a magnetic stir plate. The pH of the sample is determined with glass electrode and pH meter while stirring. The pH value is reported on appropriate data sheets.

8.3.3 Extraction by Sonication Technique

NOTE: ALL GLASSWARE MUST BE SOLVENT RINSED BEFORE USE. RINSE WITH ACETONE AND METHYLENE CHLORIDE.

8.3.3.1 Any water layer on a sediment sample is discarded. Samples are mixed thoroughly, discarding any foreign objects such as sticks, leaves and rocks.

8.3.3.2 Approximately 30 g (to the nearest 0.1 g) of sample is weighed into a 250 mL bottle. 60 g of anhydrous powdered sodium sulfate is added and mixed well. 100 mL of 1:1 methylene chloride-acetone is added to the sample, followed by 0.5 mL of surrogate spiking solution. The addition of the surrogate spiking solution is noted on the bench sheet, including who added it and the date and by whom the standard was made.

8.3.3.3 1.0 mL of the DCS or matrix spike mix is added, if appropriate, using the procedure described in Section 8.3.3.2.

8.3.3.4 The bottom surface of the tip of the sonicator horn is placed about 1/2 inch below the surface of the solvent but above the sediment layer.

8.3.3.5 The sample is sonicated for 1 1/2 minutes.

8.3.3.6 The solvent layer is decanted into a rinsed 400 mL beaker.

8.3.3.7 The extraction is repeated twice more using 100 mL aliquots of 1:1 $CH_2Cl_2$-acetone for each extraction making certain that the sodium sulfate is free flowing and not a consolidated mass. As required, large lumps are broken up with a clean spatula. These extracts are collected in the the same beaker described in 8.3.3.6.

8.3.3.8 A funnel is plugged with glasswool and filled 2/3 full with $Na_2SO_4$.

8.3.3.9 A boiling chip is added to the K-D and a 3- ball Snyder column attached to the top. The column is pre-wet by adding about 1 mL of $CH_2Cl_2$ to the top of the Snyder column.

NOTE: THE CONCENTRATION STEP IS CRITICAL; LOSSES OF TARGET COMPOUNDS CAN OCCUR IF CARE IS NOT TAKEN.

8.3.3.10 The K-D is placed in a heated water bath set at 95°C so that the receiver tube is immersed in hot water below the joint and the entire lower rounded surface is bathed in steam. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, the K-D is removed from the bath and allowed to completely cool.

8.3.3.11 After the K-D has cooled, the Snyder column and flask are rinsed with a small amount of methylene chloride. The extract is transferred to a calibrated 15 mL centrifuge tube, rinsing the K-D flask with a small amount of methylene chloride. It is important to rinse all of the ground glass joints of the K-D well, as compounds collect on the ground glass.

8.3.3.12 The extract is carefully concentrated to 1.0 mL under a gentle stream of nitrogen using the N-evap apparatus. The temperature of the water bath should be 35-40°C. If the extract is highly colored, forms a precipitate, or stops evaporating, a supervisor is consulted before the procedure is continued.

8.3.3.13 The extract is transferred to a labelled autosampler vial with Teflon lined cap. The meniscus is marked and the extract is placed on a tray in the BNA extract refrigerator with any other samples having the same QC Lot number. The tray number is recorded on the outside of the prep folder containing the bench sheets.

8.3.3.14 The prep sheet is completed for the extraction and concentration steps.

8.3.3.15 If GPC cleanup is required, see section 8.5.

8.3.4 Other Extraction Techniques

8.4 Preparation of Sludge Samples

8.4.1 Approximately 10 g of sample is weighed into a pre-weighed aluminum weigh boat and allowed to air-dry overnight. The percent moisture is determined using equation 10.2.7.

8.4.2 A continuous extractor is set-up as described in Sections 8.2.3.1 to 8.2.3.4.

8.4.3 Weigh the bottle containing the sample and carefully transfer {insert approximate amount of sample used} g of the sample to the extractor flask. Re-weigh the bottle, calculate the difference, and record it as the amount extracted on the prep sheet.

8.4.4 The continuous extractor method is followed as described in Sections 8.2.3.9 to 8.2.3.26

8.5 Screening Extracts

8.5.1 Chromatographic conditions.

8.5.1.1 Column: 15 m DB-5, 0.25 mm id, 0.25 um; or equivalent

8.5.1.2 Temperature Program: 50°(1)-20°/min-280°C

8.5.2 Inject 1 uL of the GC Calibration Standard on the screening GC. This solution contains 50 ug/mL of phenol, phenanthrene, and di-n-octyl phthalate. The response of phenanthrene should be approximately 50% full scale deflection (FSD). The response of di-n-octyl phthalate should be > 25% FSD.

8.5.3 For water samples screen only if the extract is colored. If the extracts were not combined during the concentration step, screen both fractions. If there are no non-surrogate peaks greater than 400% of the peak height of the phenanthrene peak in either fraction, submit as is. If either fraction has peaks greater than 400% of the peak height of the phenanthrene peak, dilute both fractions so that the largest peak is approximately four times the peak height of phenanthrene, i.e., both fractions will always have the same dilution factor. Note the dilution factor on the benchsheet.

8.5.4 If acid and base/neutral fractions were not combined in the concentration step, combine 0.5 mL aliquots of each fraction in one autosampler vial. Clearly label as BNA with the sample number.

8.5.5 For solid samples screen the extract at an appropriate volume (i.e. a clear extract may be screened at 1 mL; a dark extract at 10 mL.) Adjust final volume such that the largest non-surrogate peak is between 200% and 400% of the phenanthrene peak. Note the dilution factor on the benchsheet.

8.5.6 Put aliquot of any dilutions performed in a labeled autosampler vial. If any of the undiluted extract remains, label and store this autosampler vial with the dilution.

8.6 Instrument Set-Up for the GC/MS

8.6.1 Chromatographic Conditions

8.6.1.1 Column: See section 6.19.

8.6.1.2 Temperature Program:

| | |
|---|---|
| Initial Temperature: | 30°C |
| Initial hold time: | 1 minute |
| Temperature ramp: | 10°C/min |
| Final temperature: | 325°C |
| *Final hold time: | 10 minutes |

*The final hold time should be adjusted according to the retention time of the final compound in the standard.

8.6.1.3 These parameters are verified and/or changed using the DESC procedure for the Finnigan 4500's or GC method for the Finnigan Incos 50 instruments.

8.6.2 Mass Spectrometer Parameters:

8.6.2.1 Mass range: 35-500 amu

8.6.2.2 Scanning rate: 0.75 second/scan

8.6.2.3 Zone temperatures:

| | |
|---|---|
| Injection port: | 250°C |
| Transfer line: | 275°C |
| Manifold temperature: | 100°C |
| Source temperature: (if applicable) | 150°C |

8.6.3 Acquisition Parameters (Program ACQU)

Consult your supervisor if your acquisition parameters differ from the following:

| | |
|---|---|
| minimum peak width | = 3 |
| minimum area | = 30 |
| threshold | = 1 |

8.7 File Naming Conventions

For each semivolatiles instrument, the filenames for DFTPP, calibration standards, QC samples and client samples acquisitions are based on the use of common prefix abbreviations followed by instrument letter designation and a consecutive integer progression from 001 to 999. This numeric portion of the filename is commonly referred to as the "run number". When the run number on a particular instrument reaches 999, the progression begins again with 001.

Filenames are recorded in the instrument log book at the time of data acquisition along with other pertinent sample information (e.g. project & sample number, extract dilution, and analyst's initials and date of acquisition).

8.7.1 The filename for DFTPP uses the prefix DF followed by instrument letter designation and run number (next consecutive integer - up to 999 - in instrument log book).

For Example:

DFU006 is the filename for DFTPP analyzed on instrument U at run number 006 in the consecutive integer count maintained in the log book for instrument U.

8.7.2 The filenames for standards use the prefix STD followed by instrument letter designation and run number (next consecutive integer - up to 999 - in instrument log book).

For Example:

STDU007 is the filename for a standard analyzed on instrument U at run number 007 in the consecutive integer count maintained in the log book for instrument U.

8.7.3 The filenames for QC samples (Refer to Section 9. for a discussion of DCS and Blank QC Samples) use either the prefix DCS or Blank, depending on the type of QC sample, followed by instrument letter designation and run number (next consecutive integer - up to 999 - in the instrument log book).

For Example:

DCSU008 is the filename for a Duplicate Control Sample (DCS) analyzed on instrument U at run number 008 in the consecutive integer count maintained in the log book for instrument U.

8.7.4 The filenames for client samples use the prefix S followed by the RMA project number, instrument letter designation and run number (next consecutive integer - up to 999 - in the instrument log book).

For Example:

S6450U009 is the filename for a sample belonging to RMA project number 6450, run on instrument U at run number 009 in the consecutive integer count maintained in the log book for instrument U.

8.8 Instrument Calibration

8.8.1 DFTPP

8.8.1.1 Each GC/MS **MUST** be hardware tuned to meet the EPA ion abundance criteria for DFTPP. The instrument tune **MUST** be verified at the start of every 12 hours of operation or as specified by client contract. Ion abundance criteria are listed in Appendix C, Table C-1.

8.8.1.2 50 ng of DFTPP is directly injected into the GC using the following chromatographic conditions:

| | |
|---|---|
| Initial temperature: | 175°C |
| Initial hold: | 1 min |
| Rate: | 35°C/min |
| Final temperature: | 250°C |

DFTPP will generally elute between scans 250-450.

8.8.1.3 The spectrum of DFTPP is evaluated for overall intensity and ion abundances. Ion abundance criteria given in Table C-1 must be met. Averaging scans across the peak and reasonable background subtraction is acceptable. Manipulations which will distort the spectrum, such as excessive background subtraction, are not acceptable.

8.8.1.4 If the ion abundance criteria are not met, the instrument must be retuned and DFTPP reinjected.

8.8.1.5 If the ion abundance criteria are met, daily instrument calibration may proceed. Hardcopies of the DFTPP spectrum, mass list and ion abundance summary are kept on file for QC purposes.

8.8.2 Initial Calibration

An initial calibration must be performed before analysis of sample extracts can begin. This consists of a five point calibration curve for all of the analytes listed in Appendix A. Standard solutions are prepared at concentrations of 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL and 160 ug/mL.

Certain compounds have been selected to monitor instrument performance. These compounds are identified as System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC).

The average response factor for each System Performance Check Compound (SPCC) MUST be greater than 0.05.

The percent relative standard deviation (%RSD) for or equal to each Calibration Check Compound (CCC) MUST be less than or equal to 30%. The %RSD for all compounds should also be reviewed. Any values greater than 50% should be evaluated before analyses are continued.

If samples are NOT being analyzed for these specific compounds, the criteria for these compounds need not be met. The fact that these compounds are not in the analyte set should be documented.

See Table C-2 for a summary of CCC and SPCC criteria.

8.8.2.1 The following five standard solutions are analyzed: 50 ug/mL, 20 ug/mL, 80 ug/mL, 120 ug/mL and 160 ug/mL.

For each standard to be analyzed, a 1-2 uL injection is made under the conditions specified in 8.7.

8.8.2.2 Immediately following the analysis of the 50 ug/mL standard, the data acquired should be processed and evaluated to ensure that certain criteria are met before proceeding with initial calibration.

8.8.2.2.1 To minimize saturation in the higher level standards, the peak area of the quantitation ion for the 1st internal standard, d4-1,2-dichlorobenzene, should be between 10,000 and 30,000 counts, depending on the instrument. If the peak area is outside these limits, the analyst should adjust the electron multiplier and re-inject the 50 ug/mL standard. (DFTPP criteria must first be met under the new instrument operating conditions. See Section 8.9.1).

8.8.2.2.2 The SPCC limits **MUST** be verified. If the response factor of an SPCC compound is <0.05, a supervisor should be consulted to determine if corrective action will be taken.

8.8.2.3 The following target compounds are at 100 ug/mL in the 50 ug/mL standard:

Benzidine
3,3'-Dichlorobenzidine

8.8.2.4 The concentration of acid surrogate compounds is 100 ug/mL. The concentration of base/neutral surrogate compounds is 50 ug/mL. The concentration of internal standard compounds is 40 ug/mL.

8.8.2.5 The quantitation list is reviewed to ensure that the internal standards are correctly identified and that the analytes are properly quantitated. The following compounds should be checked for proper integration:

Benzyl Alcohol
Benzoic Acid
4-Nitrophenol
4-Nitroaniline
Benzo(b &/or k)fluoranthene
Indeno(1,2,3-cd)pyrene
Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene

These compounds do not chromatograph well, particularly as a column is used.

8.8.2.6 Structural isomer pairs are checked for correct peak assignment. Identification is based on elution order, as they appear in the library. These pairs are:

2,4,6-/2,4,5-Trichlorophenol
Anthracene/Phenanthrene
Chrysene/Benzo(a)anthracene
Benzo(b)/Benzo(k)fluoranthene

8.8.2.7 If the standard evaluation criteria have been met, the component libraries are updated with the response factors and retention times of the 50 ug/mL standard just analyzed.

8.8.2.8 Each of the other standards analyzed should be evaluated in the same manner as the 50 ug/mL standard with particular attention focused on the "troublesome" compounds noted in Sections 8.9.2.5 and 8.9.2.6. Some additional compounds in the 120 and 160 ug/mL standards may need to be manually quantitated due to poor chromatography at higher concentrations.

8.8.2.9 Following analysis and evaluation of the 5 calibration standards, a table which lists each standard's response factors, the average RF and % RSD's for all compounds is generated using the RMA program RFAC. Refer to Appendix E for examples of the initial

calibration table and a continuing calibration table. The % RSD's for CCC compounds and the average response factors for SPCC compounds are then evaluated. Compounds having % RSD's greater than 30% are checked to verify accuracy of quantitation for each standard. Individual response factors which differ greatly from the compound average are also investigated.

8.8.2.10 If all CCC and SPCC criteria are met, the data are stored in an initial calibration file. This file is then referred to during continuing calibration. Quan lists are then copied to unit 1.

8.8.3 Continuing Calibration

The 50 ug/mL standard is used as the continuing calibration standard. It MUST be analyzed for each 12 hour period immediately following a successful DFTPP analysis.

The response factor of each CCC compound should be compared to the initial calibration average response factor. A table which lists the initial calibration average response factors, continuing calibration response factors and % differences for all compounds is generated for comparison using the RMA procedure RFAC. See Appendix E for an example of this table. The percent difference for each CCC compound should be less than or equal to 25% for analysis to proceed. Deviations from the specified criteria for continuing calibration are allowed, provided a supervisor is informed and rationale is well documented. Response factors for all analytes should be reviewed. Any significant deviations from the initial calibration should be evaluated before analyses are continued. (e.g. deviations of greater than 50%)

All SPCC criteria should be met (i.e. RF > 0.05).

8.8.3.1 A 1 or 2 uL injection of the 50 ug/mL standard is made under the conditions specified in Section 8.7.

8.8.3.2 The 50 ug/mL continuing calibration standard should be evaluated and quantitation checked as outlined in Sections 8.9.2.2 through 8.9.2.6.

8.8.3.3 The continuing calibration standard is compared to the initial calibration curve by generating the table described in Section 8.9.3. The CCC and SPCC compounds are evaluated as outlined in Section 8.9.3. If any large percent differences between initial and continuing calibration response factors are noted, the peak assignment and integration for these compounds should be checked.

8.8.3.4 If the standard evaluation criteria are NOT met, and analyses to be performed require meeting SPCC & CCC criteria then corrective action must be taken. Corrective action may include replacement of injection port liner, cutting a portion off the front end of the column, or replacing the column. If corrective action is taken, the instrument must be recalibrated beginning with injection of DFTPP (See Section 8.9.1.2 through 8.9.1.3). Following successful analysis of DFTPP, the 50 ug/mL continuing calibration standard is reinjected. If deviations between initial and continuing calibrations still exist, a new initial calibration may need to be performed (See Section 8.9.2).

8.8.3.5 If these criteria are not met and the decision is made to continue analysis, the rationale must be documented by the individual making that decision. This documentation must be kept on file with the continuing calibration record.

8.8.3.6 If the standard evaluation criteria have been met or the decision has been made to continue, the component libraries are updated with the response factors and retention times of the continuing calibration standard just analyzed.

8.9 Sample Analysis

8.9.1 Scheduling

8.9.1.1 Analyses are scheduled by the supervisor or a senior operator and assigned to analysts on a daily basis.

8.9.1.2 A LIMS generated semivolatile sample backlog is used for prioritizing sample analyses and for recording the status of analyses, (e.g. prep completed, analysis in progress, analysis completed). The analytical test (i.e. target compound list) required is indicated on the sample backlog. Modifications to the normal analytical protocol, such as low detection limits, special compounds or special criteria are also flagged on the backlog and pertinent information is accessed by the analyst or supervisor via LIMS.

8.9.2 Extract Preparation

8.9.2.1 Extracts are located in numbered trays stored in the BNA extract refrigerator. The tray number for a given set of samples is recorded on the project folder for those samples.

8.9.2.2 Appropriate sample extract vials are located by the analyst and placed in the semivolatiles hood for aliquoting. Acid and B/N fractions are combined if necessary and the internal standard solution (400 ug/mL) is added to each extract aliquot at a ratio of 1:10 resulting in I.S. concentration of 40 ug/mL in the extract. The vial is then marked with the letters "WIS" (with internal standard) to indicate the addition of internal standard.

8.9.2.3 The syringe is rinsed with $CH_2Cl_2$ between each use.

8.9.2.4 The prepared extracts are then transferred to the appropriate instrument for analysis. If the samples are to be acquired using an autosampler (rather than being injected and acquired manually by the analyst), the vials are placed in the autosampler (either HP or Varian) rack as per the manufacturer's instructions.

8.9.3 Data Acquisition

8.9.3.1 Samples are analyzed using the same instrument parameters as the calibration standards. (conditions specified in Section 8.7)

8.9.3.2 Sample data can be acquired manually as a single run (analysis) or as a series of runs in an automated sequence of samples. Two custom RMA procedures programs which utilize the Finnigan ACQU command are used for data acquisition. The RMA procedure DOIT allows the analyst to set acquisition parameters and input sample header information for manual acquisition of a single sample. The RMA procedure ATAQ enables the analyst to set acquisition parameters and input sample header information for a series of samples. The ATAQ procedure automatically coordinates sample acquisition with the autosampler, the GC ready condition, and sample header information and filename. The analyst is responsible for making sure extract vials are placed in the autosampler rack in the order intended for analysis and that sample header information and filenames are loaded into the computer in the correct sequence as well.

8.9.3.3 If a limited list of compounds is being used, the analysis time may be shortened, as appropriate to the compounds being analyzed.

8.9.3.4 Filenames, sample identification information (project number and sample number, e.g. 6450-02), extract dilution, analyst's initials and date of analysis are recorded by the analyst in the instrument log book.

8.9.4 Analysis Sequence

8.9.4.1 QC samples (refer to Section 9 for a discussion of DCS and blank samples) should be analyzed and QC criteria met before client samples from that QC Lot number can be analyzed. See Section 9 for QC criteria for DCS and blank samples.

8.9.4.2 If QC criteria are met, client samples will be analyzed according to scheduling priority. Matrix spikes, matrix spike duplicates, and sample duplicates when available are analyzed in conjunction with the associated samples.

8.9.4.3 If the QC criteria are not met, the supervisor is informed and the decision to reprep samples from the QC lot or to continue analysis is made and documented. (See Section 9.)

8.10 Data Processing & Reduction & Data Package Assembly

"The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data." Quote from Enseco, Inc. QAPP for Env. Chem. Monitoring, Rev. 3.3.

For a general overview of Enseco data reduction, validation and reporting guidelines from a quality assurance standpoint refer to Enseco, Inc. QAPP for Env. Chem. Monitoring, Rev. 3.3, Section 8.

8.10.1 Following sample data acquisition, each sample data file is processed using the RMAL procedure TC which utilizes Finnigan AUTOQUAN software and semivolatile component libraries developed by RMAL. A number of "Library Lists" - subsets of the semivolatile libraries - reside on each instrument computer and are selected by the analyst for use in conjunction with the procedure TC to generate a data package for each sample that specifically covers the target compound list (analytical test) requested by the client. In addition to processing one data file at a time using TC, a batch of data files can be processed

automatically using the RMAL procedure AUTOB. For more detailed instructions on using the RMAL procedure "TC" to process data files, refer to "RMAL GC/MS Software Manual" (Ver. 6/28/88).

8.10.2 The data package generated contains the following print outs:

8.10.2.1 **Log Page**

Included on this page are the header information, GC conditions, scan parameters, and instrument tuning parameters that were used at the time of data acquisition.

8.10.2.2 **Chromatogram**

Included on this page is the header information and the reconstructed ion chromatogram displaying the range of scans used during data acquisitions.

8.10.2.3 **Diagnostic Report**

Included on this page is a table of library entry numbers and chromatographic and library search information used in the computerized target compound evaluation. In addition, if any peak found is saturated, this is flagged on the diagnostic report.

8.10.2.4 **Quantitation Report**

Included in this section is the list of target compounds, internal standards and surrogate standards used for processing the datafile and their corresponding library entry numbers. This section contains (1) chromatographic information such as retention times (scan numbers) and relative retention times and (2) quantitation information such as peak areas, updated library response factors, and quantitated amounts for the library entries.

8.10.2.5 **Quantitation Summary**

This summary page lists the internal standards in elution order, followed by base/neutral surrogates in elution order, acid surrogates in elution order, and then all target compounds searched for by the processing routine, also in elution order. Tabulated with each compound are the library entry number, quantitation mass, indication of reference internal standard, and for those compounds with amounts calculated to be > 1.0 ug/mL, peak area, scan number (directly related to R.T.) at which peak maximized, calculated response factor and quantitated amount. Those compounds not found by the processing routine or with amounts calculated to be < 1.0 ug/mL are flagged with the words "not found".

8.10.3 For each processed data file the following steps should be taken:

8.10.3.1 Internal standard retention times should be reviewed and compared to the daily (continuing calibration) standard. No internal standard retention time should vary more than 30 seconds from the daily standard. If this occurs, the chromatographic system should be checked and corrections made as necessary. Affected samples will be reanalyzed.

8.10.3.2 Internal standard areas should be reviewed and compared to the daily standard. An internal standard comparison report should be generated using the RMA program "ISCHK" and placed permanently into the log book. The areas should not vary by more than a factor of two (50% to 200% of daily standard internal standard area) from the daily standard. If the areas do vary by more than a factor of two, the analyst should first check to see if the peak has been correctly integrated. If the area is still outside

the limits, the system should be checked and corrected if necessary. The sample should be reanalyzed once problems with the GC/MS system have been corrected. If, upon verification that the instrument is operating properly, the areas are still outside of the limits both analyses will be submitted with the data package and the actions taken documented by the analyst.

8.10.3.3 Surrogate standard amounts should be reviewed to verify acceptable surrogate recoveries. If any one acid and/or base/neutral surrogate falls outside the acceptable recovery limits, the quantitation should be checked.

8.10.3.3.1 If the quantitation is correct, the unacceptable surrogate recovery and affected sample(s) should be shown to a supervisor. Once a decision is made to use the data or submit for a reprep, all action should be documented on the anomaly sheet.

8.10.3.3.2 If more than one acid and/or base/neutral surrogate is outside the acceptable recovery limits, the quantitation should be checked. If the quantitation is correct, the analyst should inform a supervisor and the affected sample(s) may be submitted for repreparation. The analyst should document all action taken on the anomaly sheet used for analysis.

8.10.3.4 The same general guidelines outlined in sections 8.9.2.5 and 8.9.2.6 regarding "troublesome" compounds and structural isomers should be followed whenever any of these compounds are found in a sample by the processing routine.

8.10.3.5 Target compound amounts should be reviewed to verify that the quantitated amounts are within the upper linear range of calibration (160 ug/mL +/- 10%).

8.10.3.5.1 If any target compound amounts appear to be greater than the upper limit, the quantitation should be checked by the analyst. If quantitation is correct, the sample extract must be diluted appropriately and reanalyzed for the target compound involved. The initial run may be used for any target compound data which fall within the linear range of calibration (10 ug/mL - 160 ug/mL).

8.10.3.5.2 The diagnostic report (see Section 8.11.2.3) should also be reviewed for any peak saturation of target compounds. If any target compound peak is saturated, the quantitation should be verified by the analyst and the sample extract diluted appropriately and reanalyzed.

8.10.4 Spectra Verification

The data processing routine (1C) generates a "scan list" file for each data file which contains the scan numbers for all target compounds found in the sample at amounts > 1.0 ug/mL (i.e. all target compounds listed on the data processing quantitation summary, Section 8.11.2.5). Following initial processing of a datafile, the RMAL procedure NDUAL is used to hardcopy the unenhanced and enhanced spectra for all scans in the scan list. In addition to hardcopying spectra for one data file at a time using NDUAL, a batch of data files' spectra can be hardcopied automatically using the RMAL procedure AUTOB.

Prior to qualitative identification of target compounds in a sample, "standard" mass spectra of known identity should be obtained on the instrument(s) used for sample analysis. Hardcopies of mass spectra from a calibration standard (e.g. 50 ug/mL continuing calibration standard) run on the instrument under similar tuning conditions would serve this purpose.

8.10.4.1 Each mass spectrum generated for a sample data file should be compared to the standard spectrum. EPA criteria for qualitative identification by comparison of mass spectra should be followed. The EPA criteria for comparing spectra are:

8.10.4.1.1 All ions present in the standard spectrum at a relative intensity greater than 10% of the base peak MUST be present in the sample spectrum.

8.10.4.1.2 The relative intensities of the ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

8.10.4.1.3 Ions greater than 10% in the sample spectrum, but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labeled with the contaminant.

8.10.4.2 When a spectra meets the criteria listed in Section 8.11.4.1 the compound name in the header should be circled. All spectra that do not meet the EPA criteria should have the compound name crossed out in the header, and the corresponding entries on the quantitation summary should be crossed out (line out the amounts calculated for those compounds which are not "hits") by the analyst making the judgment. If a compound cannot be verified by all of the criteria listed in 8.11.4.1, but in the technical judgment of the analyst the identification is correct, the compound should be reported. If the analyst is uncertain of an identification, a senior operator or a supervisor should be consulted.

8.10.5 Summary Sheets

A number of summary sheets are computer generated at the instrument following data file processing and pulling (hardcopying) target compound spectra. These summaries include:

8.10.5.1 **Target Compound Summary**
(also referred to as "sum sheets" or "sums")

This one-or two page summary is generated with the RMAL procedure "SUM1" and tabulates pertinent information about a sample analysis including header information (e.g. sample number, dilution used for analysis, sample matrix), date analyzed, analyst's initials, run factor (refer to calculations in Section 10.1.1), final reporting units, surrogate % recoveries, target compounds listed in elution order, and target compound CAS #'s when available. For those target compounds with amounts > 1.0 ug/mL in the extract the scan number, amount listed on quantitation summary (i.e. concentration in the analyzed extract ug/mL) and concentration in sample (expressed in final reporting units, usually ug/l for waters and ug/kg for soils) are tabulated.

8.10.5.2 **DCS Summary**
(Refer to Section 9 for discussion of DCS QC samples)

This one-page summary is generated with the RMA procedure DCS and tabulates pertinent information about the DCS samples representing a specific QC lot. The information tabulated includes QC lot number, matrix (soil or water), date analyzed, analyst's initials, DCS data file numbers, list of samples belonging to the QC lot, names of spike components and QC limits for spike % recoveries and RPD's, calculated amount and percent recovery for each spike compound in both DCS samples, and calculated RPD between the two

DCS recoveries for each spike component. Percent recoveries and RPD's that fall outside the acceptable QC limits are flagged with asterisks and require further action by the analyst (see Section 9). When generating the summary sheet it is imperative that the analyst input the complete list of samples belonging to the QC lot for QC tracking purposes. This information can be found in the QC summary notebook kept in the prep lab.

8.10.5.3 **SCS Summary**

(Also referred to as "SCS Summary"- see section 9 for discussion of SCS QC samples)

This one page summary tabulates pertinent information about the SCS sample representing a specific analytical lot (also called "QC" or "SCS" lot) of client samples including QC lot number, matrix (soil or water), date analyzed, analyst's initials, SCS data file number, list of samples belonging to the SCS lot, names of surrogates and QC limits for surrogate % recoveries, and calculated amount and % recovery for each surrogate. Percent recoveries that fall outside the acceptable QC limits are flagged with asterisks and require further action by the analyst (see Section 9). When generating this summary sheet, it is imperative that the analyst input the complete list of samples associated with the SCS sample for QC tracking and data reporting (blank subtraction) purposes. This information can be found in the QC summary notebook kept in the sample preparations laboratory.

8.10.6 Library Searches (Optional)

Some data packages will include mass spectral library searches on non-target compounds (excluding internal standards, surrogates and spikes) as outlined in contract requirements or requested by the client. If the library searches (also referred to as "TIC's" or Tentatively Identified Compounds) are

necessary to the data package, the 20 largest unidentified peaks that are greater than 10% of the peak (RIC) area of the nearest uninterfered internal standard will be searched against the NBS library for possible matches.

8.10.6.1 The generation of TIC data is partially automated through the use of several RMA procedures which perform the following functions:

1. Select non-target, unidentified peaks from the chromatogram for tentative identification,

2. Quantitate unknown peaks using RIC area of closest uninterfered internal standard as reference peak and a response factor of 1.0 (See calculations, Section 10.2.11),

3. Perform and generate hardcopy mass spectral library searches on the unknown peaks (searching against the NBS library of mass spectra),

4. Select preliminary "Best Match" spectra from the library searches and tabulate them with quantitation information for all unknowns,

5. Allow editing of computer's final selections for compound names,

6. Allow manual quantitation of unusually shaped peaks,

7. Generate final summary with amounts calculated to reflect concentration in sample (i.e., run factor applied).

8.10.6.2 Listed below, in the order that they are generally used by the analyst, are the RMA procedures used for TIC generation and their main functions:

TICP - Used to verify and/or change peak selection criteria (e.g. can specify to search all unknown peaks >10% internal standard RIC area.

TIC1 - Generates TIC data (hardcopies of library search spectra and quantitation information page) for one data file.

TIC - Generates TIC data (hardcopies of library search spectra and quantitation information page) for a batch of data files.

TICQS- Allows for manual quantitation by the analyst.

TICE - Allows the analyst to edit the names of compounds selected and generates final TIC summary page after input from the analyst.

8.10.6.3 After reviewing each library search spectrum, the analyst should indicate the compound name selected by labelling the spectrum or circling the correct name (if it appears on the library search spectrum as one of the computer's selections). If the compound is to be reported as "Unknown" (See Section 8.11.6.5) the spectrum should be labelled as such.

8.10.6.4 The EPA criteria to be used for making these decisions for tentative identifications are as follows:

8.10.6.4.1 Relative intensities of major ions present in the library spectrum at a relative intensity greater than 10% of the base peak should be present in the sample spectrum.

8.10.6.4.2 The relative intensities of the ions specified in (1) must agree within plus or minus 20% between the library and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%.)

8.10.6.4.3 Molecular ions present in the library spectrum should be present in the sample spectrum.

8.10.6.4.4 Ions present in the sample spectrum, but not present in the standard spectrum should be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labelled with the contaminant.

8.10.6.4.5 Ions present in the library spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds.

8.10.6.5 If in the technical judgment of the analyst, no valid tentative identification can be made, the compound should be reported as "Unknown." If possible, assign additional compound class information (e.g. unknown hydrocarbon). The analyst should consult with a more experienced analyst or a supervisor if there are questions concerning compound identification.

8.10.6.6 For more detailed instructions on the generation of TIC data using the RMA TIC procedures, refer to "RMA GC/MS Software Manual", version 6/28/88.

8.10.7 Reconstructed Ion Chromatogram

The RIC generated by data file processing should be labelled to identify internal standards, surrogates, spikes if applicable, target compounds and tentatively identified compounds (if library searches are performed - see Section 8.11.6). For industrial, non-contract work, the abbreviations IS - internal standard, SS - surrogate standard, SP or SC - spike compound, TC - target compound, and TIC - tentatively identified compound, are sufficient. In contract work the internal standards and surrogates are usually labelled with the compound names; the abbreviations SC, TC and TIC are normally sufficient for contract work for spike compounds, target compounds and tentatively identified compounds provided a legend is clearly indicated on each chromatogram page. Refer to Appendix F for more specific criteria.

8.10.8 Anomaly Sheet

Each data package submitted by the analyst for secondary review must include an anomaly sheet summarizing any problems or unusual circumstances incurred during sample analysis. If no problems were encountered during analysis, the analyst must still submit an anomaly sheet to that effect. All anomaly sheets must be singed by a supervisor.

8.10.9 QC Summary Sheets (DCS & SCS)

Each data package submitted for secondary review must include copies of DCS and SCS summary sheets for all QC related to the sample set in the data package.

8.10.10 Summary of Data Package Requirements

When initial data review has been completed by the analyst, the data package should be assembled to include the following:

- Anomaly Sheet
- DCS Summary Sheet
- SCS Summary Sheet
- SCS Target Compound Summary Sheet, TIC Summary and Data
- Out-of-Control forms (if necessary - See Section 9)
- Sample Raw Data
- Sample Target Compound Summaries (Attached to sample raw data)
- TIC Data & Summary (when requested)

In some cases, contract requirements or client requests will necessitate including tuning and calibration information (i.e. DFTPP), daily standard raw data and initial & continuing calibration response factor tables. Refer to Appendix F for specific data package criteria.

8.10.11 Upon completion of the data package assembly and review, the data package is passed on to the data review specialist for secondary review.

9. QA/QC Requirements (See SOP no.: M-EQA-002)

9.1 DCS. Laboratory Control Samples are extracted and analyzed for every 20 samples. DCS components and spike concentrations are given in Appendix D. For aqueous samples, DCS components are spiked into organic-free water. For solid samples, the DCS components are spiked into the extraction solvent. No solid matrix (sand or Celite) is incorporated since there is no true representative solid matrix.

9.2 SCS. A Surrogate Control Sample is analyzed with every analytical lot. This sample serves as the method blank. An analytical lot for extractable organics is defined as samples extracted or prepared at the same time up to a maximum of 20 samples. An SCS sample is also referred to as an SCS.

SCS components and spike concentrations are given in Appendix D.

9.3 Blank. For this method the blank and the SCS are one and the same. In order for data associated with an SCS to be acceptable, blank values for target compounds must be acceptable. If the blank (SCS) contains target compounds at values above the reporting limit, the supervisor should be consulted.

If the blank value lies between the reporting limit and three times the reporting limit, the reporting limit is adjusted to the level found in the blank.

9.4 Surrogates spiked into samples. Surrogate compounds are required to be spiked into all samples and QC samples for this method. Surrogate spike mix concentrations are given in Appendix D.

9.5 Matrix specific QC. Matrix spike and matrix spike duplicates are performed at the request of the client. Matrix spike compounds will be the same as those used for the DCS. See Appendix D. These compounds will be spiked into aliquots of the sample specified by the client at the same concentration level as the DCS.

9.6 Data acceptability is initially based upon the results of the Duplicate Control Samples. If DCS criteria is not in control, consult a supervisor and document any corrective action and/or alternative data acceptability criteria on an Out-of Control form. QC data must fall within established control limits in order for the laboratory to be considered "in control" when samples from that QC lot were analyzed.

9.6.1 At least 80% of the DCS recovery data, and at least 80% of the DCS precision data and at least 80% of the SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Blank values must be acceptable. (See Section 9.3) For this method the maximum number of values permitted outside control limits are

| | recovery | precision |
|---|---|---|
| DCS: | 4 | 2 |
| SCS: | 1 | |

9.7 Control Limits: Control limits will be determined as described in SOP No.: M-EQA-002. At a minimum, CLP limits must be met. These limits are listed in Table D-2. Current historical limits generated by the laboratory will be available through LDMS.

10. Calculations

10.1 Calculations required to reduce data

10.1.1 Acid and B/N run factors (rf):

$$rf = \frac{Vf\ (mL)}{Vi\ (L\ or\ kg)} \times \frac{100\%}{(\%\ Dilution\ of\ extract\ used\ for\ analysis)} \times (Column\ Clean\ up\ Factor)$$

Where Vf = final extract volume as prepped
Vi = initial sample volume (or weight) used for extraction

$$\text{Column Clean up Factor} = \frac{\text{Volume available for clean up}}{\text{Volume put through column}}$$

10.1.2 Expected (theoretic) Acid and B/N surrogate and/or spike concentrations in extract (TSC):

$$TSC = \frac{C_s\ (ug/_{mL}) \times Vs\ (mL)}{Vf\ (mL)} \times (\%\ Dilution\ of\ extract\ used\ for\ analysis) \times \frac{1}{(Column\ Clean\ up\ Factor)}$$

Where TSC = theoretic surrogate or spike concentration in extract

Cs = concentration of surrogate or spike in mix

Vs = volume of surrogate mix or spike mix spiked into sample during extraction procedure

10.2 Calculations required to verify automated data reduction

10.2.1 Response factors (RF):

$$RF = \frac{A_x \times Cis\ (ug/mL)}{A_{is} \times C_x\ (ug/mL)}$$

where $A_x$ = area of characteristic (quantitation) ion for the compound to be measured

$A_{is}$= area of characteristic (quantitation) ion for the reference internal standard

$C_{is}$= concentration of the internal standard

$C_x$ = concentration of the compound to be measured

10.2.2 Average Response Factor ($RF_{ave}$):

$$RF_{ave} = \frac{(RF_{20} + RF_{50} + RF_{80} + RF_{120} + RF_{160})}{5}$$

Where $RF_x$ = response factor of a compound in each of the calibration standards

10.2.3 Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

Where SD = standard deviation of individual initial calibration response factors (per compound)

and where

$$SD = \sum_{i=1}^{N} \frac{(X_i - X)^2}{N-1}$$

10.2.4 Percent Deviation from average response factor (%D):

$$\%D = \frac{RF_I - RF_C}{RF_I} \times 100$$

where $RF_I$ = average response factor from initial calibration

$RF_C$ = response factor from current calibration daily standard

10.2.5 Percent Recovery (%Rec):

Spikes -

$$\%Rec = \frac{SSR\ (ug/mL) - SR\ (ug/mL)}{TSC\ (ug/mL)} \times 100$$

Where SSR = spiked sample results
SR = sample results
TSC = theoretic spike concentration

Surrogates -

$$\%Rec = \frac{SR\ (ug/mL)}{TSC\ (ug/mL)} \times 100$$

Where SR = surrogate concentration (results in sample)

TSC = theoretic surrogate concentration

10.2.6 Relative Percent Difference (RPD):

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where $D_1$ = first sample value

$D_2$ = second sample value (e.g. duplicate or matrix spike duplicate)

10.2.7 Percent moisture

$$\%moisture = \frac{\text{Wet wt (sample plus dish)} - \text{Dry wt (sample plus dish)}}{\text{Wet wt (sample plus dish)}} \times 100$$

10.2.8 Analyte concentration in an aqueous sample

$$\text{Concentration } \left(\frac{ug}{L}\right) = \frac{A_x \times C_{is}\ (ug/mL)}{A_{is} \times RF} \times rf \left(\frac{mL}{L}\right)$$

Where $A_x$ = area of quantitation ion for compound being measured

$A_{is}$ = area of quantitation ion for reference internal standard

$C_{is}$ = concentration of internal standard in sample extract

RF = response factor for compound being measured from ongoing (continuing) calibration standard

rf = run factor (See 10.1.1)

10.2.9 Analyte concentration in a soil sample (wet weight)

$$\text{Concentration } \left(\frac{ug}{kg}\right) = \frac{A_x \times C_{is}\ (ug/mL)}{A_{is} \times RF} \times rf\ \left(\frac{mL}{kg}\right)$$

Where $A_x$, $A_{is}$, $C_{is}$, RF, rf = same as given for water in 10.2.8

10.2.10 Analyte concentration in a soil sample (dry weight)

$$\text{Concentration } \left(\frac{ug}{kg}\right) = \frac{A_x \times C_{is}\ (ug/mL)}{A_{is} \times RF} \times \frac{1}{\left(1-\frac{\%moisture}{100}\right)} \times rf\left(\frac{mL}{kg}\right)$$

Where $A_x$, $A_{is}$, $C_{is}$, RF, rf = same as given for water in 10.2.8

and % moisture as defined in 10.2.7

10.2.11 Tentatively identified compound concentration in sample

$$\text{Concentration } \left(\frac{ug}{L} \text{ or } \frac{ug}{kg}\right) = \frac{RIC_x \times C_{is}\ (ug/mL)}{RIC_{is} \times 1.0} \times rf\ \left(\frac{mL}{L} \text{ or } \frac{mL}{kg}\right)$$

Where $RIC_x$ = area of reconstructed ion chromatogram for unknown compound being measured

$RIC_{is}$ = area of reconstructed ion chromatogram for closest uninterfered internal standard

$C_{is}$ = concentration of internal standard in sample extract

rf = run factor for sample extract as analyzed

11. Reporting requirements

11.1 Units: Aqueous samples will be reported in ug/L. Solid and sludge samples will be reported in ug/kg on a wet weight basis, unless requested otherwise by the client.

11.2 Reporting limits:

See Appendix A

11.3 Significant Figures:

All data <10 ppb will be reported with one significant figure. All other data will be reported with two significant figures.

11.4 LDMS data entry

12. References

12.1 Method Source: Method 8270, SW-846 Third Edition, USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, September 1986.

12.2 Deviations from Source Methods

12.2.1 Daily (continuing) calibration is considered to be valid for 12 hours from time of injection of DFTPP.

12.2.2 Deviations from the specified criteria for initial and continuing calibration are allowed, provided the rationale is documented.

12.2.3 An internal QA/QC program has been adopted by Enseco, Inc. This is described in Enseco's "Quality Assurance Program Plan for Environmental Chemical Monitoring" (Rev. 3.3). Specific elements of the QC program are described in SOP NO. M-EQA-002: "Internal QC Checks--Laboratory Performance QC." The Laboratory Control Samples generated in this program are used to monitor method performance. Matrix spikes are performed at the request of the client.

12.2.4 Additional Sources

12.2.4.1 USEPA Contract Laboratory Program Statement of Work 10/86 Rev:1/88, 2/88, 7/88

12.2.4.2 RMAL GC/MS Software Manual (Ver. 6/28/88)

12.2.4.3 Enseco, Inc. Quality Assurance Program Plan for Environmental Chemical Monitoring (Rev 3.2).

APPENDIX A Analytes & Detection Limits

TABLE A-1 HSL LIST

| Analyte | Detection Limit Water (ug/mL) | Detection Limit Soil (ug/mL) |
|---|---|---|
| Phenol | 10 | 330 |
| bis(2-Chloroethyl)ether | 10 | 330 |
| 2-Chlorophenol | 10 | 330 |
| 1,3-Dichlorobenzene | 10 | 330 |
| 1,4-Dichlorobenzene | 10 | 330 |
| Benzyl alcohol | 10 | 330 |
| 1,2-Dichlorobenzene | 10 | 330 |
| 2-Methylphenol | 10 | 330 |
| bis(2-Chloroisopropyl)ether | 10 | 330 |
| 4-Methylphenol | 10 | 330 |
| N-Nitroso-di-n-propylamine | 10 | 330 |
| Hexachloroethane | 10 | 330 |
| Nitrobenzene | 10 | 330 |
| Isophorone | 10 | 330 |
| 2-Nitrophenol | 10 | 330 |
| 2,4-Dimethylphenol | 10 | 330 |
| Benzoic acid | 50 | 1600 |
| bis(2-Chloroethoxy)methane | 10 | 330 |
| 2,4-Dichlorophenol | 10 | 330 |

TABLE A-1 HSL LIST (Cont.)

| Analyte | Detection Limit Water (ug/mL) | Detection Limit Soil (ug/mL) |
|---|---|---|
| 1,2,3-Trichlorobenzene | 10 | 330 |
| Naphthalene | 10 | 330 |
| 4-Chloroaniline | 10 | 330 |
| Hexachlorobutadiene | 10 | 330 |
| 4-Chloro-3-methylphenol | 10 | 330 |
| 2-Methylnaphthalene | 10 | 330 |
| Hexachlorocyclopentadiene | 10 | 330 |
| 2,4,6-Trichlorophenol | 10 | 330 |
| 2,4,5-Trichlorophenol | 50 | 1600 |
| 2-Chloronaphthalene | 10 | 330 |
| 2-Nitroaniline | 50 | 1600 |
| Dimethyl phthalate | 10 | 330 |
| Acenapnthylene | 10 | 330 |
| 3-Nitroaniline | 50 | 1600 |
| Acenaphthene | 10 | 330 |
| 2,4-Dinitrophenol | 50 | 1600 |
| 4-Nitrophenol | 50 | 1600 |
| Dibenzofuran | 10 | 330 |
| 2,4-Dinitrotoluene | 10 | 330 |
| 2,6-Dinitrotoluene | 10 | 330 |
| Diethyl phthalate | 10 | 330 |
| 4-Chlorophenylphenyl ether | 10 | 330 |
| Fluorene | 10 | 330 |
| 4-Nitroaniline | 50 | 1600 |
| 4,6-Dinitro-2-methylphenol | 50 | 1600 |
| N-Nitrosodiphenylamine | 10 | 330 |
| 4-Bromophenylphenyl ether | 10 | 330 |
| Hexachlorobenzene | 10 | 330 |
| Pentachlorophenol | 50 | 1600 |
| Phenanthrene | 10 | 330 |
| Anthracene | 10 | 330 |
| Di-n-butyl phthalate | 10 | 330 |
| Fluoranthene | 10 | 330 |
| Pyrene | 10 | 330 |

| | | |
|---|---|---|
| Butyl benzyl phthalate | 10 | 330 |
| 3,3'-Dichlorobenzidine | 20 | 660 |
| Benzo(a)anthracene | 10 | 330 |
| bis(2-Ethylhexyl)phthalate | 10 | 330 |
| Chrysene | 10 | 330 |
| Di-n-octyl phthalate | 10 | 330 |
| Benzo(b)fluoranthene | 10 | 330 |
| Benzo(k)fluoranthene | 10 | 330 |
| Benzo(a)pyrene | 10 | 330 |
| Indeno(1,2,3-c,d)pyrene | 10 | 330 |
| Dibenzo(a,h)anthracene | 10 | 330 |
| Benzo(g,h,i)perylene | 10 | 330 |

TABLE A-2 PRIORITY POLLUTANT LIST

| Analyte | Detection Limit Water (ug/mL) | Detection Limit Soil (ug/mL) |
|---|---|---|
| Phenol | 10 | 330 |
| bis(2-Chloroethyl)ether | 10 | 330 |
| 2-Chlorophenol | 10 | 330 |
| 1,3-Dichlorobenzene | 10 | 330 |
| 1,4-Dichlorobenzene | 10 | 330 |
| 1,2-Dichlorobenzene | 10 | 330 |
| bis(2-Chloroisopropyl)ether | 10 | 330 |
| N-Nitroso-di-n-propylamine | 10 | 330 |
| Hexachloroethane | 10 | 330 |
| Nitrobenzene | 10 | 330 |
| Isophorone | 10 | 330 |
| 2-Nitrophenol | 10 | 330 |
| 2,4-Dimethylphenol | 10 | 330 |
| bis(2-Chloroethoxy)methane | 10 | 330 |
| 2,4-Dichlorophenol | 10 | 330 |
| 1,2,4-Trichlorobenzene | 10 | 330 |
| Naphthalene | 10 | 330 |
| Hexachlorobutadiene | 10 | 330 |
| 4-Chloro-3-methylphenol | 10 | 330 |
| Hexachlorocyclopentadiene | 10 | 330 |
| 2,4,6-Trichlorophenol | 10 | 330 |
| 2-Chloronaphthalene | 10 | 330 |
| Dimethyl phthalate | 10 | 330 |
| Acenaphthylene | 10 | 330 |
| Acenaphthene | 10 | 330 |
| 2,4-Dinitrophenol | 50 | 1600 |
| 4-Nitrophenol | 50 | 1600 |
| 2,4-Dinitrotoluene | 10 | 330 |
| 2,6-Dinitrotoluene | 10 | 330 |
| Diethyl phthalate | 10 | 330 |

TABLE A-2 PRIORITY POLLUTANT LIST (Cont.)

| Analyte | Detection Limit Water (ug/mL) | Detection Limit Soil (ug/mL) |
|---|---|---|
| 4-Chlorophenylphenyl ether | 10 | 330 |
| Fluorene | 10 | 330 |
| 4,6-Dinitro-2-methylphenol | 50 | 1600 |
| 1,2-Diphenylhydrazine | 10 | 330 |
| N-Nitrosodiphenylamine | 10 | 330 |
| 4-Bromophenylphenyl ether | 10 | 330 |
| Hexachlorobenzene | 10 | 330 |
| Pentachlorophenol | 50 | 1600 |
| Phenanthrene | 10 | 330 |
| Anthracene | 10 | 330 |
| Di-n-butyl phthalate | 10 | 330 |
| Fluoranthene | 10 | 330 |
| Pyrene | 10 | 330 |
| Butyl benzyl phthalate | 10 | 330 |
| 3,3'-Dichlorobenzidine | 20 | 660 |
| Benzo(a)anthracene | 10 | 330 |
| bis(2-Ethylhexyl)phthalate | 10 | 330 |
| Chrysene | 10 | 330 |
| Di-n-octyl phthalate | 10 | 330 |
| Benzo(b)fluoranthene | 10 | 330 |
| Benzo(k)fluoranthene | 10 | 330 |
| Benzo(a)pyrene | 10 | 330 |
| Indeno(i,2,3-c,d)pyrene | 10 | 330 |
| Dibenzo(a,h)anthracene | 10 | 330 |
| Benzo(g,h,i)perylene | 10 | 330 |

APPENDIX B Standards Solutions

TABLE B-1 SURROGATE STANDARD SPIKING SOLUTION

| Compound | Concentration in Mix (ug/mL) |
|---|---|
| Nitrobenzene - $D_5$ | 100 |
| 2-Fluorobiphenyl | 100 |
| Terphyenyl - $D_{14}$ | 100 |
| Phenol - $D_5$ | 200 |
| 2-Fluorophenol | 200 |
| 2,4,6-Tribromophenol | 200 |

TABLE B-2 DCS STANDARD SPIKING SOLUTION
(Matrix Spiking Solution)

| Compound | Concentration in Mix (ug/mL) |
|---|---|
| 1,2,4-Trichlorobenzene | 100 |
| Acenaphthene | 100 |
| 2,4-Dinitrotoluene | 100 |
| Pyrene | 100 |
| N-Nitroso-Di-n-propylamine | 100 |
| 1,4-Dichlorobenzene | 100 |
| Pentachlorophenol | 200 |
| Phenol | 200 |
| 2-Chlorophenol | 200 |
| 4-Chloro-3-Methylphenol | 200 |
| 4-Nitrophenol | 200 |

TABLE B-3 GC CALIBRATION STANDARD
(for Screening)

BNA-SCS/IS Working Solution

| Component | Concentration (ug/mL) |
|---|---|
| Dichlorobenzene-D4 | 8 |
| Naphthalene-D8 | 8 |
| Acenaphthene-D10 | 8 |
| Phenanthrene-D10 | 8 |
| Chrysene-D12 | 8 |
| Perylene-D12 | 8 |
| 2-Fluorophenol | 10 |
| Phenol-D5 | 10 |
| Tribromophenol | 10 |
| Nitrobenzene-D5 | 5 |
| 2-Fluorobiphenyl | 5 |
| Terphenyl-D14 | 5 |

TABLE B-4 INTERNAL STANDARD SOLUTION

| Compound | Concentration in Mix (ug/mL) |
|---|---|
| $D_4$-1,4-Dichlorobenzene | 400 |
| $D_8$-Naphthalene | 400 |
| $D_{10}$-Acenaphthene | 400 |
| $D_{10}$-Phenanthrene | 400 |
| $D_{12}$-Chrysene | 400 |
| $D_{12}$-Perylene | 400 |

TABLE B-5 CALIBRATION STANDARD SOLUTIONS

1) MS-BNAHSL-CSB

| Compound | Concentration in Mix (ug/mL) |
|---|---|
| Nitrobenzene-$D_5$ | 50 |
| 2-Fluorobiphenyl | 50 |
| Terphenyl-$D_{14}$ | 50 |
| $D_5$-Phenol | 100 |
| 2-Fluorophenol | 100 |
| 2,4,6-Tribromophenol | 100 |
| N-Nitrosodimethylamine | 50 |
| Aniline | 50 |
| Phenol | 50 |
| bis(2-Chloroethyl)ether | 50 |
| 2-Chlorophenol | 50 |
| 1,3-Dichlorobenzene | 50 |
| 1,4-Dichlorobenzene | 50 |
| Benzyl Alcohol | 50 |
| 1,2-Dichlorobenzene | 50 |

TABLE B-5 CALIBRATION STANDARD SOLUTIONS (Cont.)

1) MS-BNAHSL-CSB

| Compound | Concentration in Mix (ug/mL) |
|---|---|
| 2-Methylphenol | 50 |
| bis(2-Chloroisopropyl)ether | 50 |
| 4-Methylphenol | 50 |
| N-Nitroso-di-n-propylamine | 50 |
| Hexachloroethane | 50 |
| Nitrobenzene | 50 |
| Isophorone | 50 |
| 2-Nitrophenol | 50 |
| 2,4-Dimethylphenol | 50 |
| bis(2-Chloroethoxy)methane | 50 |
| 2,4-Dichlorophenol | 50 |
| Benzoic Acid | 50 |
| 1,2,4-Trichlorobenzene | 50 |
| Naphthalene | 50 |
| 4-Chloroanaline | 50 |
| Hexachlorobutadiene | 50 |
| 4-Chloro-3-Methylphenol | 50 |
| 2-Methylnaphthalene | 50 |
| Hexachlorocyclopentadiene | 50 |
| 2,4,6-Trichlorophenol | 50 |
| 2,4,5-Trichlorophenol | 50 |
| 2-Chloronaphthalene | 50 |
| 2-Nitroanaline | 50 |
| Dimethylphthalate | 50 |
| Acenaphthylene | 50 |
| 2,6-Dinitrotoluene | 50 |
| 3-Nitroaniline | 50 |
| Acenaphthene | 50 |
| 2,4-Dinitrophenol | 50 |
| Dibenzofuran | 50 |

TABLE B-5 CALIBRATION STANDARD SOLUTIONS

1) MS-BNAHSL-CSB

| Compound | Concentration in Mix (ug/mL) |
|---|---|
| 4-Nitrophenol | 50 |
| 2,4-Dinitrotoluene | 50 |
| Diethylphthalate | 50 |
| Fluorene | 50 |
| 4-Chlorophenyl-phenyl Ether | 50 |
| 4-Nitroanaline | 50 |
| 4,6-Dinitro-2-Methylphenol | 50 |
| N-Nitrosodiphenylamine | 50 |
| Azobenzene | 50 |
| 4-Bromophenylphenyl Ether | 50 |
| Hexachlorobenzene | 50 |
| Pentachlorophenol | 50 |
| Phenanthrene | 50 |
| Anthracene | 50 |
| Di-n-butylphthalate | 50 |
| Fluoranthene | 50 |
| Benzidine | 100 |
| Pyrene | 50 |
| Butylbenzylphthalate | 50 |
| Benzo(a)anthracene | 50 |
| 3,3'-Dichlorobenzidine | 100 |
| Chrysene | 50 |
| bis(2-Ethylhexyl)phthalate | 50 |
| Di-n-octyl phthalate | 50 |
| Benzo(b)fluoranthene | 50 |
| Benzo(k)fluoranthene | 50 |
| Benzo(a)pyrene | 50 |
| Indeno(1,2,3-c,d)pyrene | 50 |
| Dibenz(a,h)anthracene | 50 |
| Benzo(g,h,i)perylene | 50 |

The other 4 points in the 5 pt. calibration standard set contain the above list of compounds at 20 ug/mL, 80 ug/mL, 120 ug/mL and 160 ug/mL, with the exception of acid surrogates, benzidine and 3,3'-Dichlorobenzidine which are in each mix at twice the concentration. 1 or 2 uLs of each standard is injected.

APPENDIX C

TABLE C-1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

| Mass | Ion Abundance Criteria |
|---|---|
| 51 | 30.0 - 60.0 percent of mass 198 |
| 68 | less than 2.0 percent of mass 69 |
| 70 | less than 2.0 percent of mass 69 |
| 127 | 40.0 - 60.0 percent of mass 198 |
| 197 | less than 1.0 percent of mass 198 |
| 198 | base peak, 100 percent relative abuncance |
| 199 | 5.0 - 9.0 percent of mass 198 |
| 275 | 10.0 - 30.0 percent of mass 198 |
| 365 | greater than 1.00 percent of mass 198 |
| 441 | present but less than mass 443 |
| 442 | greater than 40.0 percent of mass 198 |
| 443 | 17.0 - 23.0 percent of mass 442 |

TABLE C-2

CALIBRATION CHECK COMPOUNDS

Initial Calibration: The percent relative standard deviation for the each compound listed below should be $\leq$30%.

Continuing Calibration: The percent deviation from the average response factor determined in the initial calibration should be $\leq$25% for each compound listed below.

| Base/Neutral Fraction | Acid Fraction |
|---|---|
| Acenaphthene | 4-Chloro-3-Methylphenol |
| 1,4-Dichlorobenzene | 2,4-Dichlorophenol |
| Hexachlorobutadiene | 2-Nitrophenol |
| N-Nitrosodinphenylamine | Phenol |
| Di-n-octylphthalate | Pentachlorophenol |
| Fluoranthene | 2,4,6-Trichlorophenol |
| Benzo(a)pyrene | |

SYSTEM PERFORMANCE CHECK COMPOUNDS

Initial calibration: The average response factor for each compound listed below should be $>$0.05.

Continuing calibration: The response factor for each compound listed below should be $>$0.05.

| Base/Neutral Fraction | Acid Fraction |
|---|---|
| N-Nitroso-di-n-propylamine | 2,4-Dinitrophenol |
| Hexachlorocyclopentadiene | 4-Nitrophenol |

APPENDIX D

TABLE D-1
SPIKE LEVELS FOR LABORATORY CONTROL SAMPLES

| | Spike Level Aqueous (ug/L) | Spike Level Solid* (ug/Kg) | Concentration in mix (ug/ml) |
|---|---|---|---|
| **DCS** | | | |
| Pentachlorophenol | 100 | 6,670 | 200 |
| Phenol | 100 | 6,670 | 200 |
| 2-Chlorophenol | 100 | 6,670 | 200 |
| 4-Chloro-3-Methylphenol | 100 | 6,670 | 200 |
| 4-Nitrophenol | 100 | 6,670 | 200 |
| 1,2,4-Trichlorobenzene | 50 | 3,330 | 100 |
| Acenaphthene | 50 | 3,330 | 100 |
| 2,4-Dinitrotoluene | 50 | 3,330 | 100 |
| Pyrene | 50 | 3,330 | 100 |
| N-Nitroso-di-n-propylamine | 50 | 3,330 | 100 |
| 1,4-Dichlorobenzene | 50 | 3,330 | 100 |
| **SCS** | | | |
| Phenol-$d_5$ | 200 | 3,330 | 200 |
| 2-Fluorophenol | 200 | 3,330 | 200 |
| 2,4,6-Tribromophenol | 200 | 3,330 | 200 |
| Nitrobenzene-$d_5$ | 100 | 1,670 | 100 |
| 2-fluorobiphenyl | 100 | 1,670 | 100 |
| Terphenyl-$d_{14}$ | 100 | 1,670 | 100 |

*Assumes 30g sample (wet weight)

TABLE D-2

CLP RECOVERY & RPD CONTROL LIMITS

| | Minimum Control Limit | | | |
|---|---|---|---|---|
| | WATER | | SOIL | |
| DCS: | | | | |
| | DCS | RPD | DCS | RPD |
| Pentachlorophenol | 9-103 | 50 | 17-109 | 47 |
| Phenol | 12-89 | 42 | 26-90 | 35 |
| 2-Chlorophenol | 27-123 | 40 | 25-102 | 50 |
| 4-Chloro-3-Methylphenol | 23-97 | 42 | 26-103 | 33 |
| 4-Nitrophenol | 10-80 | 50 | 11-114 | 50 |
| 1,2,4-Trichlorobenzene | 39-98 | 28 | 38-107 | 23 |
| Acenaphthene | 46-118 | 31 | 31-137 | 19 |
| 2,4-Dinitrotoluene | 24-96 | 38 | 28-89 | 47 |
| Pyrene | 26-127 | 31 | 35-142 | 36 |
| N-Nitroso-di-n-propylamine | 41-116 | 38 | 41-126 | 38 |
| 1,4-Dichlorobenzene | 36-97 | 28 | 28-104 | 27 |

| SCS | WATER | SOIL |
|---|---|---|
| Phenol-$d_5$ | 10-94 | 24-113 |
| 2-Fluorophenol | 21-100 | 25-121 |
| 2,4,6-Tribromophenol | 10-123 | 19-122 |
| Nitrobenzene-$d_5$ | 35-114 | 23-120 |
| 2-Fluorobiphenyl | 43-116 | 30-115 |
| Terphenyl-$d_{14}$ | 33-141 | 18-137 |

## APPENDIX A ANALYTICAL TEST REFERENCES

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Subject or Title: 

GC/MS Analysis of Semivolatile Organics

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| LM-RMA-3013 BNA 625 | 1.0 | 12/01/89 |

Supersedes:

## Note About Analytical Test References

The following test references have been written utilizing commands as described in the GC/MS Software Manual. Procedure names and functions are subject to change and therefore most have not been written into this SOP.

See Appendix B for a summary of current procedures.

Analytical Test Reference No. 1

HAZARDOUS SUBSTANCE LIST (HSL)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSLSTD; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for daily standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. For samples, one surrogate each fraction is allowed out if present above 10%.

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard, samples and QC processed using AUTOQUAN and BNAHSLSTD.LL, i.e.

TC filename,, BNAHSLSTD <CR>

2) Pull spectra for samples and blanks.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Preparations laboratory benchsheets.

GC/MS Analysis of Semivolatile Organics

| SOP No.: LM-RMA-3013 BNA 625 | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes:

Analytical Test Reference No. 2

PRIORITY POLLUTANT (PP OR 625)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in 'HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSL; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for daily standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. In samples, one surrogate each fraction is allowed out if present above 10%.

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard, samples and QC processed using AUTOQUAN and BNAHSLSTD.LL, i.e.

TC filename,, BNAHSLSTD

2) Pull spectra for samples and blanks.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to bank of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Preparations Laboratory benchsheet

GC/MS Analysis of Semivolatile Organics

| SOP No.: LM-RMA-3013 BNA 625 | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes:

Analytical Test Reference No. 3

INDUSTRIAL, NON-CONTRACT CLIENTS

INDUSTRIAL CLP (RAS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: A special header format is required for each analysis (i.e., standard, blanks, samples and QC samples). See attached sheet for instruction on header format before analyzing standard.

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

   a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "HEDR"

   c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSLSTD; DUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. Must meet EPA criteria for CCC and SPCC compounds in daily calibration standard and initial calibration

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. QC samples as prepped (e.g. method blanks, matrix spikes and matrix spike dups) with each project

E. Repreps are required for any samples (blanks not included) having 2 or more surrogates either fraction outside limits; 1 surrogate each fraction is allowed outside limits provided recovery is > 10%.

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard processed using AUTOQUAN and BNAHSLSTD.LL

2) Samples and QC processed using AUTOQUAN and BNACLP.LL

3) Pull spectra for samples and QC samples, <u>except</u> no spectra required for spike compounds in QC samples

4) Pull TIC's for 25 largest unknown peaks > 10% peak area of closest uninterfered I.S.

5) Capture target compound data and TIC data for foremaster using procedures SCAP and TICCAP respectively

B) Data Package Includes

1) All raw data including

DFTPP spectrum
DFTPP mass list
DFTPP summary
DFTPP "Form V" (which lists datafiles and time of acquisition)
Daily standard raw data
RFAC ongoing calibration summary
RFAC initial calibration summary
Sample, blank and QC sample raw data

NOTE: All pages must be torn apart at perforation so copies can be made

2) Sample, blank and QC sample chromatograms labeled (stamped) with I.S. names, S.S. names, "TC, TIC and SC" for target compounds, TIC's and spike compounds respectively, and a legend defining any symbols used (i.e. TC, TIC and SC)

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample (No spectra required for QC sample spike compounds)

5) TIC library searches (labeled) and summary.

6) Data generated by capturing target compound and TIC data.

7) Floppy disk on which data is captured

8) Anomaly sheet

9) Spike recovery forms for matrix spikes and matrix spike dups

**GC/MS Analysis of Semivolatile Organics**

| SOP No.: **LM-RMA-3013 BNA 625** | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes:

Analytical Test Reference No. 4

REFINERY (REF)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - SKBNA - CSB @ 50 ug/mL

Analytical Test Reference No. 4

REFINERY (REF)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - SKBNA - CSB @ 50 ug/mL

2) Initial Calibration

a) 3 point curve using 10 ug/mL, 50 ug/mL & 150 ug/mL standards

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL
b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP
2) "DOIT" is used on 4500's to acquire data manually; "HEDR" is used on INCOS 50
3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "AASK"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAREF; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for daily calibration and consult supervisor if outside limits

C. Enseco QC - DCS's and SCS's

D. Document analytical problems on anomaly sheet

E. One surrogate each fraction allowed out for samples if detected above 10%

III. Data

A) Processing

1) Standard and QC samples processed using AUTOQUAN and BNAREFSTD.LL

2) Samples processed using AUTOQUAN and BNAREF.LL

3) Pull spectra for samples and blanks

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Sample preparations laboratory benchsheet

Analytical Test Reference No. 5

APPENDIX IX (AP9)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

   b) MS - AP9BNA - CSB @ 50 ug/mL

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "HEDR"

   c) Step through "AUTO" to input both acquisition information *and processing information*

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAAP9; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for HSL standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. One surrogate each fraction allowed out for samples if detected above 10%

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL

a) AA - Dimethylphenethylamine typically has extremely poor peak shape and needs to be manually quantitated.
b) Hexachlorophene is usually not detected at 50 ug/mL and as a general rule is subtracted from the AP9 STD QUAN list prior to updating the libraries to preserve the existing retention time data for this compound.

3) Samples and QC processed using AUTOQUAN and BNAAP9.LL

4) Famphur degrades in the standard and needs to be searched for by mass chro for all samples.
5) Pull spectra for samples and blanks.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Sample preparations laboratory benchsheets

Analytical Test Reference No. 6

APPENDIX VIII (AP8)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

   b) MS - AP9BNA - CSB @ 50 ug/mL

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extra

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL".

   b) Header information goes in "HEDR".

   c) Step through "AUTO" to input both acquisition information and processing information

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAAP9; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for HSL standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. One surrogate each fraction allowed out for samples (above 10% rec.).

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL

a) AA - Dimethylphenethylamine typically has extremely poor peak shape and needs to be manually quantitated.

b) Hexachlorophene is usually not detected at 50 ug/mL and as a general rule is subtracted from the AP9 STD QUAN list prior to updating the libraries to preserve the existing retention time data for this compound.

3) Samples and QC processed using AUTOQUAN and BNAAP9.LL

4) Famphur degrades in the standard and needs to be searched by mass chro for all samples.

5) Pull spectra for samples and blanks.

6) Pull library "E8" searches on all unknown peaks > I.S. height and quantitate any peaks that match library spectra using a response factor of 1.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Sample preparations laboratory benchsheet.

Analytical Test Reference No. 7

TCLP

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: For convenience TCLP samples should be scheduled on an instrument running AP9 work on same day.

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

b) MS - AP9BNA - CSB @ 50 ug/mL - the AP9 STD is necessary to the TCLP analysis for retention time and response factor data for the following 2 compounds:

Pyridine
2,3,4,6-Tetrachlorophenol

Consult a supervisor prior to injecting AP9 STD if AP9 samples have not been scheduled on the instrument the day TCLP samples are to be analyzed.

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSL; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must hit DFTPP

B. Evaluate CCC and SPCC compounds for HSL standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. One surrogate, >10%, allowed out for each fraction

E. Document analytical problems on anomaly sheet

F. Leach blanks are analyzed as regular samples

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL (See Analytical Test Reference No. 5 (AP9) for processing hints)

3) Samples and QC processed using AUTOQUAN and BNATCLP.LL

4) Pull spectra for samples and SCS's

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Preparations laboratory benchsheet.

**GC/MS Analysis of Semivolatile Organics**

| SOP No.: **LM-RMA-3013 BNA 625** | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes:

Analytical Test Reference No. 8

AFIRP Version 1

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSL; DUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. Must meet EPA criteria for CCC and SPCC compounds for daily standard

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. Enseco QC - DCS's and blanks

E. One surrogate >10% allowed out for each fraction

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard and QC processed using AUTOQUAN and BNAHSLSTD.LL

2) Samples processed using AUTOQUAN and BNAHSLSTD.LL

3) Pull spectra for samples and SCS's

4) Pull TIC's for 20 largest unknown peaks > 10% peak area of closest uninterfered I.S.

B) Data Package Includes

1) DFTPP summary and RFAC summary are required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC, TIC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) TIC library searches and summary attached to back of each sample

6) DCS and SCS summary sheets (and target compound summary for SCS)

7) Anomaly sheet

8) Copies of DCS and blank sample raw data

**GC/MS Analysis of Semivolatile Organics**

| SOP No.: **LM-RMA-3013 BNA 625** | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes:

Analytical Test Reference No. 9

AFIRP Version 2

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

   b) MS - AP9BNA - CSB @ 50 ug/mL

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "HEDR"

   c) Step through "AUTO" to input both acquisition information and processing information

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAAFIR; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must hit DFTPP

B. Must meet EPA criteria for CCC and SPCC compounds for daily standard (HSL)

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. Enseco QC - DCS's and blanks

E. One surrogate >10% allowed out for each fraction

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL

3) Samples and QC processed using AUTOQUAN and BNAAFIR.LL

4) Pull spectra for samples and SCS's

5) Pull TIC's for 20 largest unknown peaks > 10% peak area of closest uninterfered I.S.

6) Search for Dibenz(a,j)acridine by mass chromatogram.

B) Data Package Includes

1) DFTPP summary and RFAC summary are required; DFTPP "Form V" (lists samples and time of acquisition) is required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC, TIC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) TIC library searches and summary attached to back of each sample

6) DCS and SCS raw data

7) Anomaly sheet

**GC/MS Analysis of Semivolatile Organics**

| SOP No.: LM-RMA-3013 BNA 625 | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes:

Analytical Test Reference No. 10

USGS USGS

(CONTRACTUAL)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175RC and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30RC and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - USGSBNA - CSB @ 50 ug/mL

2) Initial Calibration

a) 5 point curve using 20, 50, 80, 120, and 160 ug/mL standards.

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSL; DUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. Must meet EPA criteria for CCC and SPCC compounds in daily calibration standard (when compared to initial calibration curve average response factors)

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. QC samples as prepped - usually a blank and blank spike for each project or analytical lot

E. All surrogate recoveries must fall within recovery limits set by the USGS; if any surrogates are outside limits a reprep of the sample involved is requested.

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard, samples and QC processed using AUTOQUAN and BNAUSGS.LL

2) Pull spectra for samples and QC samples

3) Pull "LCOMPS", i.e., Library Comparison Spectra, for samples and QC samples target compounds

4) When requested pull TIC's for all unknown peaks >10% peak area of closest uninterfered I.S.

B) Data Package Includes

1) All raw data (torn apart at perforations) including

DFTPP spectrum
DFTPP mass list

DFTPP summary
DFTPP "Form V" (which lists datafiles and time of acquisition)
Daily Standard Raw Data -
RFAC ongoing calibration summary
RFAC initial calibration summary
Sample, blank and blank spike raw data

2) Sample, blank and blank spike chromatograms labeled with I.S. names, S.S. names, "TC, TIC and SC" for target compounds, TIC's and spike compounds respectively, and a legend defining any symbols used (i.e. TC, TIC, SC)

3) USGS target compound cover sheets attached to front of each sample

4) Labeled spectra and LCOMP spectra attached to back of each sample

5) TIC library searches (when requested) and summary attached to back of each sample

6) USGS blank summary forms

7) USGS blank spike recovery forms

8) USGS continuing calibration forms (or initial calibration forms if appropriate)

9) USGS surrogate recovery summary form

10) Anomaly sheet

**GC/MS Analysis of Semivolatile Organics**

| SOP No.: **LM-RMA-3013 BNA 625** | Revision No.: 1.0 | Effective Date: 12/01/89 |
|---|---|---|

Supersedes:

Analytical Test Reference No. 11

PAH - part per billion Full Scan

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

5. Then "START" (GC control section)

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) 50 ug/mL PAH standard

2) Initial Calibration

a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

a) MS - PAH - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL".

b) Header information goes in "HEDR".

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAPAHSTD; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. CCC compounds must be within ± 25% of initial calibration curve average

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. QC samples as prepped

E. Samples having 1 or more surrogates outside limits are reanalyzed for verification of original data, if consistent with first analysis surrogate problem is then assessed by the analyst and/or supervisor.

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard samples and QC processed using autoquan

e.g., TC filename,, BNAPAHSTD

2) Pull spectra for samples and QC samples

3) <u>No</u> TIC's

B) Data Package Includes

1) All raw data including

DFTPP spectrum
DFTPP mass list
DFTPP summary
DFTPP "Form V" (which lists datafiles and time of acquisition)
Daily standard raw data
RFAC ongoing calibration summary
RFAC initial calibration summary
Sample, blank and QC sample raw data

NOTE: All pages must be torn apart at perforation so copies can be made

2) Sample, blank and QC sample chromatograms labeled (stamped) with I.S. names, S.S. names, "TC and SC" for target compounds and spike compounds respectively, and a legend defining any symbols used (i.e. TC and SC)

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) Spike recovery forms for matrix spikes and matrix spike duplicates

6) Surrogate recovery forms

7) Method blank summary sheet

8) Anomaly sheet

APPENDIX B

Higher Level Procedures

```
EDR FILES:      HSL AP9 REF PAH PYR ABL SBL ADCS1 ADCS2 SDCS1 SDCS2

IBRARIES:       BNAHSLSTD       HSL ONLY
                BNAAP9STD       AP9 ONLY
                BNAAP9          HSL + AP9
                BNAREFSTD       ALL REF
                BNAREF          REF - SPIKE COMPOUNDS
                BNAPAHSTD       PAH ONLY
                BNAPYRSTD       PYR ONLY
                BNAPYR          HSL + PYR
                BNACLP          HSL - 2 COMPOUNDS
                SPIKE           SPIKE COMPOUNDS
                BNAAFIR         HSL + AP9 - SOME COMPOUNDS

ICS:            IBL=RUN TICP(/SB;E)             IBL1=RUN TICP(/SB;E)
                   =TIC IBL                         =TIC1

                ISAMP=RUN TICP(/SS;E)           ISAMP1=RUN TICP(/SS;E)
                     =TIC ISAMP                       =TIC1

                CBL=RUN TICP(/SB;E)             CBL1=RUN TICP(/SB;E)
                   =TICCLP CBL                      =TICCL1

                CSAMP=RUN TICP(/SS;E)           CSAMP1=RUN TICP(/SS;E)
                     =TICCLP CSAMP                    =TICCL1

JMSHEETS:       [S][MATRIX][LIBR][LEVEL/SPECIAL]

                SA=CHK;SET8 $1;RUN SUM1(/BE;L;H;E)
                SS=CHK;SET8 $1;RUN SUM1(/BF;L;H;E)
                SAPAH=CHK;SET8 $1;RUN SUM1(/PA;L;H;E)
                SAPYR=CHK;SET8 $1;RUN SUM1(/AP;L;H;E)          (INCLUDES HSL)
                SSPYRM=CHK;SET8 $1;RUN SUM1(/SP;X,2;L;H;E)     (INCLUDES HSL)
                SSPYRL=CHK;SET8 $1;RUN SUM1(/SP;L;H;E)         (INDLUDES HSL)
                SSCLP=CHK;SET8 $1;RUN SUM1(/CL;L;H;E)
                SAPYRO=CHK;SET8 $1;RUN SUM1(/SA;L;H;E)         (PYR ONLY)

      PARAMETERS:     PA      PAH SURROGATE RECOVERIES
                      AP      AQUEOUS HSL/PYR SURROGATE RECOVERIES
                      SP      SOIL HSL/PYR SURROGATE RECOVERIES
                      SA      AQUEOUS PYR SURROGATE RECOVERY

ANDARDS:        [LIBRARY][STD]

                HSLSTD=TCIND,,BNAHSLSTD;CCHSL/H
                AP9STD=TCIND,,BNAAP9STD
                REFSTD=TCIND,,BNAREFSTD;CCREF/H
                PAHSTD=TCIND,,BNAPAHSTD;CCPAHB/H
                PYRSTD=TCIND,,BNAPYRSTD;CCPYR/H

:               [MATRIX][BL/DCS]

                ABL=TCIND,,BNAHSLSTD;NDUAL;IBL1;SA
                SBL=TCIND,,BNAHSLSTD;NDUAL;IBL1;SS
                ADCS=TCIND,,SPIKE;SA
                SDCS=TCIND,,SPIKE;SS

DUSTRIAL:       [MATRIX][LIBR][LEVEL/SPECIAL]

                AHSL=TCIND,,BNAHSLSTD;NDUAL;SA
                SHSL=TCIND,,BNAHSLSTD;NDUAL;SS
                AAP9=TCIND,,BNAAP9;NDUAL;SA;FAM;DBAJ
                SAP9=TCIND,,BNAAP9;NDUAL;SS;FAM;DBAJ
                AREF=TCIND,,BNAREF;NDUAL;SA
                SREF=TCIND,,BNAREF;NDUAL;SS
                APAH=TCIND,,BNAPAHSTD;NDUAL;SAPAH
```

```
                APYRO=TCIND,,BNAPYRSTD;NDUAL;SAPYRO      (PYR ONLY)
                AAFIR=TCIND,,BNAAFIR;NDUAL;SA;DBAJ
                SAFIR=TCIND,,BNAAFIR;NDUAL;SS;DBAJ
                ATCLP=TCIND,,BNATCLP;NDUAL;SA

LP:             [MATRIX][LIBR][LEVEL/SPECIAL]

                ACLP=TCCLP,,BNACLP;SDUAL;SA
                SCLP=TCCLP,,BNACLP;SDUAL;SSCLP
                APYR=TCIND,,BNAPYR;SDUAL;SAPYR           (INCLUDES HSL)
                SPYRL=TCIND,,BNAPYR;SDUAL;SSPYRL         (INCLUDES HSL)
                SPYRM=TCIND,,BNAPYR;SDUAL;SSPYRM         (INCLUDES HSL)

ISC:            NEWCOL=QUAN NEWHSL(S;T;E)
                      =QUAN NEWAP9(S;T;E)
                      =QUAN NEWREF(S;T;E)

                CAL=ATAQ1
                   =ACQU/O(G10;E)

                GAS=CALI#3
                   =FIT

                FIA=SET4 S1#1                            (QUANTITATES IS#1)
                   =CHRO(S;#;&;R;A1,1;N1,1;D-50,50;E)

                LIA=SET4 S6#1                            (QUANTITATES IS#6)
                   =CHRO(S;#;&;R;A1,1;N1,1;D-50,50;E)

                QION=SETL BNAHSLSTD     (DISPLAYS QUAN ION FOR ALL ENTRIES
                    =QION2               IN THE SPECIFIED LIBRARY)
                QION2=GETL
                     =CHRO(S;#;&;R;D-50,50)
                     =LOOP

                SCUP #SCAN,,#SCAN
                        UPDATES "FILAMENT ON" AND "FIRST SURROGATE"
                        IN FOLLOWING:   TCIND
                                        TCCLP
                                        TIC
                                        TIC1
                                        TICCLP
                                        TICCL1

                SCL     CHECKS SCUP UPDATED SCANS

                DTB=EDNL TB(-;W;E)

                F0=FILE(H 0:*.TI;E)

                F2=FILE(H 2:*.TI;E)

                F3=FILE(H 3:*.TI;E)

                METHCH=PRIN(T;METHYL CHRYSENE;C;E)
                      =CHRO(D1800,2600;121;239;242;243;H;E)

                FAM=PRIN(T;FAMPHUR;C;E)
                   =CHRO(D1800,2200;93;125;218;H;E)

                DBAJ=PRIN(T;DIBENZ-A,J-ACRIDINE;C;E)
                    =CHRO(D2300,3100;277;278;279;280;H;E)

                DBAH=PRIN(T;DIBENZ-A,H-ACRIDINE;C;E)
                    =CHRO(D2300,3100;277;278;279;280;H;E)
```

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title: Preparation of Chlorinated Phenoxyacid Herbicides in Industrial Aqueous and Solid Samples



| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| LM-RMA-4044 | 2.0 | March 29, 1991 |

Supersedes: None

ENSECO PROPRIETARY INFORMATION STATEMENT

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1. Scope and Application

1.1 Analytes

This method is applicable to EPA SW-846 Method 8150 and Method 615 for the determination of phenoxyacid herbicides in groundwater and solid matrices.

1.2 Reporting Limits

Refer to the individual analysis LM-RMA-4007.

| | |
|---|---|
| Prepared by: Todd Burgesser | Date: March 29, 1991 |
| Management Approval: | Date: 10/8/91 |
| QA Officer Approval: | Date: 10/9/91 |

1.3 Applicable Matrices

This method is applicable to aqueous and solid matrices, not to include drinking water.

1.4 Dynamic Range

Refer to the individual analysis LM-RMA-4007.

1.5 Analysis Time

Preparation time for one batch of 20 samples and required QC [Two Duplicate Control Samples (DCS), and a Single Control Sample (SCS)] is approximately 22 hours for aqueous samples and 30 hours for solid samples.

2. Method Summary

2.1 Waters: a 1 liter sample is acidified and extracted 3 times with diethyl ether.

Solids: A 50 g sample is acidified and extracted 3 times with diethyl ether on a wrist action shaker. The extract is then cleaned in acidified $Na_2SO_4$ solution.

2.2 The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash.

2.3 After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivatizing agent.

2.4 After excess reagent is removed, the esters are analyzed on a gas chromatograph equipped with Electron Capture Detector (ECD).

3. Comments

3.1 Interferences

3.1.1 The most common interference is laboratory contamination which arises from impure reagents, dirty glassware, improper sample transferring, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

3.1.2 All glassware must be washed, rinsed with acetone, baked at 450°C for two hours, and again rinsed with diethyl ether immediately before use.

4. Safety Issues

4.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible.

4.2 All laboratory personnel should be thoroughly familiar with the Laboratory Safety Manual and Hazardous Communication Standard before undertaking any laboratory work.

4.3 Proper protective gear (i.e., safety glasses, gloves, lab coat) should be worn when working with solvents and reagents.

4.4 Respiratory protection (AFR) is required when using the diazomethane generator.

4.5 All sample/extract transfers and operations should take place in a properly vented hood, spills should be cleaned up immediately.

4.6 All waste generated from the test will be placed into appropriate waste containers.

4.7 Material Safety Data Sheets (MSDS) are available for all laboratory standards and reagent chemicals. The appropriate MSDS must be read before handling the chemical(s).

5. Sample collection, preservation and holding times

5.1 Samples/extracts should be refrigerated at 4°C.

5.2 The extraction holding time for aqueous samples is 7 days after collection. The extraction holding time for solid samples is 14 days after collection. The analytical holding time is 40 days after extraction begins.

6. Apparatus

6.1 Separation funnel, 2 liter capacity, 500 mL capacity, and 125 mL capacity, with Teflon stopcock and stopper

6.2 8 oz. wide mouth jar with Teflon-lined cap

6.3 Media bottles, 250 mL or 400 mL or equivalent

6.4 Boiling flask, 250 mL and 500 mL capacity

6.5 Water bath, heated, capable of temperature control (+/-5$^{o}$C)

6.6 Vials, Teflon-lined caps: 40 mL and 12 mL

6.7 Miscellaneous disposable pipets, 5 1/2", 1 mL, 10 mL, or equivalent

6.8 pH paper, wide range

6.9 Boiling chips, Teflon, mesh size 10 - 40

6.10 Nitrogen-evaporator apparatus, (N-evap), connected to $N_2$ gas

6.11 Wrist action shaker: Burrell Model 75 or equivalent

6.12 Funnel, 2 3/4" diameter, fluted, or equivalent

6.13 Diazomethane generation manifold.

7. Reagents and Standards

7.1 Solvents: diethyl ether, hexane, methanol, pesticide grade

7.2 Potassium Hydroxide (KOH): 37% aqueous solution (w/v)

7.3 Hydrochloric acid (HCl): Concentrated

7.4 Sulfuric acid solution $H_2SO_4$: 1:1 (v/v)

7.5 Sodium sulfate, $Na_2SO_4$, granular-baked 4 hours in 400$^{o}$C oven.

7.6 Silicic acid (silica gel), chromatographic grade, 100 mesh

7.7 Duplicate Control Sample (DCS) Standard

7.7.1 Herb DCS Standard

7.7.1.1 2,4-D Acid 5.0 ug/mL

7.7.1.2 2,4,5-T Acid 1.0 ug/mL

7.7.1.3 2,4,5-TP Acid 1.0 ug/mL

7.7.2 Solutions for spiking (DCS) must be from a different source than the calibration standard.

See Standard Prep SOP #LS-RMA-2003.

7.8 Single Control Sample (SCS) Standard

7.8.1 Herb SCS Standard

7.8.1.1 2,4-dichlorophenylacetic acid (DCAA) 5.0 ug/mL

See Standard Prep SOP #LS-RMA-2001

7.9 Methylation Standard

7.9.1 Herb Methylation Standard

7.9.1.1 2,4-D Acid 5.0 ug/mL

7.9.1.2 2,4,5-T Acid 1.0 ug/mL

7.9.1.3 2,4,5-TP Acid 1.0 ug/mL

7.9.1.4 DCAA 5.0 ug/mL

See Standard Prep SOP #LS-RMA-2007

7.10 Carbon filtered water - ASTM Type II water (Milli Q or equivalent).

7.11 2% methanolic KOH

7.12 N-methyl-n-nitroso-p-toluenesulfamide (Diazald): (ACS)

8. Procedure

8.1 Solids

Note: All glassware must be rinsed with diethyl ether before use.

8.1.1 Weigh approximately 50 g of sample into an 8 oz wide mouth jar and record the weight on a prep sheet and in a logbook.

8.1.2 Acidify the sample with 1-2 mL of concentrated hydrochloric acid (HCl) to a pH of less than 2. Allow the sample to stand for 15 minutes with occasional stirring until the pH remains below 2. Add more acid if necessary.

8.1.3 Add 80 mL of diethyl ether to each sample, and add the appropriate surrogate standard to each sample using a 1 mL disposable pipet. Be sure the tip of the pipet is below the solvent level. Add the appropriate spike mix if necessary in the same manner described above.

8.1.4 Tightly cap the jar and shake the sample on the wrist action shaker for 20 minutes. After shaking, remove each sample from the wrist-action shaker and decant the extract into a 400 mL media bottle.

8.1.5 Extract the sample twice more using 80 mL of diethyl ether. After addition of solvent, the mixture should be shaken on the wrist action shaker for 20 minutes. Decant the extract after shaking into the appropriate 400 mL media bottle.

8.1.6 Record the volume of extract recovered on the bench sheet.

8.1.7 Prepare a solution of 5% sodium sulfate ($Na_2SO_4$) in acidified carbon filtered water (CF $H_2O$) as follows: Use a 4 liter beaker and add 200 mL of CF $H_2O$ per sample to the beaker. Then add $Na_2SO_4$ (20 g per 1000 mL of $H_2O$) to the CF $H_2O$. Add the $Na_2SO_4$ slowly and stir the CF $H_2O$ with a glass stirring rod. Continue adding and stirring until all the $Na_2SO_4$ is dissolved. Acidify the solution with 1:1 sulfuric acid ($H_2SO_4$) until the pH remains below 2. Pour 200 mL of the solution into a 500 mL separatory funnel for each sample.

8.1.8 Pour the sample extract into a labeled 500 mL separatory funnel containing 200 mL of the 5% $Na_2SO_4$ in acidified CF $H_2O$.

8.1.9 Gently mix the contents of each separatory funnel for about 1 minute and allow the layers to separate. Collect the aqueous phase in a clean beaker and the extract (top layer) in a 500 mL ground-glass boiling flask. Re-extract the water layer with 25 mL of diethyl ether. Allow the layers to separate and discard the aqueous layer. Collect the extracts in the respective boiling flasks.

8.1.10 Add 30 mL of CF $H_2O$, 5 mL of 37% KOH and a few boiling chips to each flask.

8.1.11 Fit each flask with a 3-ball Snyder column and cover the sides of the flask with aluminum foil. Do not cover the Snyder column or the bottom of the flask. Prewet the snyder column with 1-2 mL of diethyl ether. Evaporate the diethyl ether on a steam bath set at 95°C. Allow to heat for 90 minutes.

8.1.12 Remove the flasks from the steam bath and allow them to cool. Remove the snyder column and rinse the ground-glass joint and the inside of the snyder column with CF $H_2O$ into the boiling flask.

8.1.13 Transfer the aqueous solution to a 125 mL separatory funnel. Rinse the flask with a few mL of CF $H_2O$. Pour this rinsate into the separatory funnel. Extract the basic solution with 40 mL of diethyl ether, allow layers to separate, and drain off the aqueous layer into a clean beaker. Discard the ether layer through the top of the separatory funnel into a waste container. Pour the aqueous layer back into the separatory funnel and extract twice more with 20 mL of diethyl ether. Discard the ether fractions each time.

8.1.14 Add 5 mL of cold, 1:1 $H_2SO_4$ to the contents of each separatory funnel to adjust the pH to less than 2. Add more acid if necessary.

8.1.15 Extract the acidic solutions once with 20 mL of diethyl ether by shaking for two minutes. Allow the layers to separate, drain off the bottom layer into a beaker, and pour the ether layer into a 40 mL VOA vial that contains 1.0 g of $Na_2SO_4$. Repeat the extractions twice more with 10 mL of diethyl ether each time. Pour the ether layer into the 40 mL vial after each extraction.

8.1.16 Put a plug of glass wool in a powder funnel and fill 2/3 full with acidified $Na_2SO_4$, (0.1 mL concentrated $H_2SO_4$ added to 100 g of $Na_2SO_4$). Pour the extract through the $Na_2SO_4$ into a 50 mL receiver tube. Rinse the vial, then the $Na_2SO_4$ with small amounts of diethyl ether back into the receiver tube.

Note: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. If all the $Na_2SO_4$ solidifies in a cake, pour the extract through fresh $Na_2SO_4$ again.

8.1.17 Evaporate the sample extract on a nitrogen evaporator equilibrated at 28°C to an approximate volume of 2-5 mL. Transfer the sample to 12 mL vial calibrated at 10 and 1.0 mL. Rinse the 40 mL vial with 2 mL of diethyl ether and add this rinsate to the 12 mL vial. Continue evaporating the sample extract to 1.0 mL. Remove the vial from the nitrogen-evaporator and allow the extract to cool. Add 0.1 mL (100 uL) methanol to each extract.

Note: The concentration step is critical; losses of target compounds can occur if care is not taken. Rinse the walls of the vial frequently and monitor the temperature closely.

8.1.18 Proceed with esterification using diazomethane (8.4).

8.2 Waters

Note: All glassware must be rinsed with diethyl ether before use.

8.2.1 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle. All others are mixed by shaking the bottle.

8.2.2 If the sample is in a 1 L or smaller bottle, mark the water meniscus on the side of the bottle for later determination of the sample volume. In this case, the volume will be determined as described in 8.2.8. If the sample is in a larger bottle, measure 1 L of sample in a graduated cylinder. Pour the sample into a 2 L separatory funnel. For blanks and quality control samples, pour 1 L of CF $H_2O$ into the separatory funnel.

8.2.3 Check the pH of each sample and note the initial pH on the prep sheet. Adjust the pH of each sample to less than 2 with 1:1 $H_2SO_4$. Note the adjusted pH on the prep sheet.

8.2.4 Add the appropriate surrogate standard to each sample using a 1.0 mL disposable pipet. Be sure that the pipet tip is below the liquid when adding the standard. Add the appropriate spike mix if necessary in the same manner described above.

8.2.5 Add 150 mL of diethyl ether directly into the separatory funnel for each sample. Extract the sample by shaking it for 2 minutes with frequent ventilation.

8.2.6 Allow the layers to separate. If there is an emulsion, break it mechanically or by centrifugation. Note the presence of any emulsion and record the corrective action on the bench sheet.

8.2.7 Drain the aqueous phase (bottom layer) into an appropriate container. Drain the solvent layer into a 250 mL boiling flask (with ground glass joint).

8.2.8 Add 15 mL of CF $H_2O$, 2 mL of 37% KOH and a few boiling chips to each flask.

8.2.9 Pour the sample back into the separatory funnel. Repeat the extraction two more times using 50 mL of diethyl ether each time. Rinse the sample container with each additional diethyl ether aliquot before adding it to the separatory funnel. Determine the original volume by refilling the sample bottle to the meniscus mark with water and transferring to a 1 liter graduated cylinder. Record the sample volume to the nearest 10 mL.

8.2.10 Attach a 3-ball Synder column to the boiling flask and cover the sides of the flask with aluminum foil. Do not cover the Snyder column or the bottom of the flask. Prewet the column with approximately 1 mL of diethyl ether.

8.2.11 Heat each flask on a steam bath set at 95°C for 60 minutes. Remove the flask and check that no diethyl ether remains. Allow the flask to cool. Remove the Snyder column and rinse the ground-glass joint with 1-2 mL of CF $H_2O$ into the boiling flask. Also, rinse the inside of the Snyder column with CF $H_2O$ into the flask.

8.2.12 Transfer the aqueous solution to a 125 mL separatory funnel. Rinse the flask with a few mL of CF $H_2O$ and add this rinsate to the separatory funnel. Extract the basic solutions with 20 mL of diethyl ether and allow the layers to separate. Drain the aqueous (bottom) layer back into the boiling flask and discard the diethyl ether through the top of the separatory funnel into a waste container. Pour the aqueous layer back into the separatory funnel and extract once more with 20 ml diethyl ether. Discard the ether fraction after this second extraction.

8.2.13 Add approximately 5 mL of cold, 1:1 $H_2SO_4$ to the contents of each separatory funnel to adjust the pH to less than 2. Add more acid if necessary.

8.2.14 Extract the acidic solutions once with 20 mL of diethyl ether by shaking for 2 minutes. Allow the layers to separate, drain the bottom layer (aqueous) back into the boiling flask and pour the diethyl ether extract through the top of the funnel into a 40 mL VOA vial that contains 1.0 g of $Na_2SO_4$. Repeat the extraction twice more with 10 mL aliquots of diethyl ether. Pour the ether layer into the 40 mL vial after each extraction.

8.2.15 Put a plug of glass wool in a powder funnel and fill 2/3 full with acidified $Na_2SO_4$, (0.1 mL concentrated $H_2SO_4$ added to 100 g of $Na_2SO_4$). Pour the extract through the $Na_2SO_4$ into a 50 mL receiver tube. Rinse the vial, then the $Na_2SO_4$ with small amounts of diethyl ether back into the receiver tube.

Note: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. If all the $Na_2SO_4$ solidifies in a cake, pour the extract through a fresh $Na_2SO_4$ funnel again.

8.2.16 Evaporate the sample extract on a nitrogen evaporator equilibrated at 28°C to an approximate volume of 2-5 mL. Transfer the sample to 12 mL vial calibrated at 10 and 1.0 mL. Rinse the 40 mL vial with 2 mL of diethyl ether and add this rinsate to the 12 mL vial. Continue evaporating the sample extract to 1.0 mL. Remove the vial from the nitrogen-evaporator and allow the extract to cool. Add 0.1 mL (100 uL) methanol to each extract.

Note: The concentration step is critical; losses of target compounds can occur if care is not taken. Rinse the walls of the vial frequently and monitor the temperature closely.

8.2.17 Proceed with esterification using diazomethane (8.4).

8.3 Leachates

Note: All glassware must be rinsed with diethyl ether before use.

8.3.1 Measure 100 mL of sample into a graduate cylinder. Pour the sample into a 500 mL separatory funnel. For blanks and quality control samples, pour 1 L of CF $H_2O$ into a 2 L separatory funnel.

8.3.2 Check the pH of each sample and note the initial pH on the prep sheet. Adjust the pH of each sample to less than 2 with 1:1 $H_2SO_4$. Note the adjusted pH on the prep sheet.

8.3.3 Add the appropriate surrogate standard to each sample using a 1.0 mL disposable pipet. Be sure that the pipet tip is below the liquid when adding the standard. Add the appropriate spike mix if necessary in the same manner described above.

8.3.4 Add 20 mL of diethyl ether directly into the separatory funnel for each sample. Extract the sample by shaking it for 2 minutes with frequent ventilation.

8.3.5 Allow the layers to separate. If there is an emulsion, break it mechanically or by centrifugation. Note the presence of any emulsion and record the corrective action on the bench sheet and in a logbook.

8.3.6 Drain the aqueous phase (bottom layer) into an appropriate container. Drain the solvent layer into a 250 mL boiling flask (with ground glass joint).

8.3.7 Pour the sample back into the separatory funnel. Repeat the extraction two more times using 20 mL of diethyl ether each time. Rinse the sample container with each additional diethyl ether aliquot before adding it to the separatory funnel.

8.3.8 Add 15 mL CF $H_2O$, 2 mL 37% KOH and boiling chips to each flask.

8.3.9 Attach a 3-ball Synder column to the boiling flask and cover the sides of the flask with aluminum foil. Do not cover the Snyder column or the bottom of the flask. Prewet the column with approximately 1 mL of diethyl ether.

8.3.10 Heat each flask on a steam bath set at 95°C for 60 minutes. Remove the flask and check that no diethyl ether remains. Allow the flask to cool. Remove the Snyder column and rinse the ground-glass joint with 1-2 mL of CF $H_2O$ into the boiling flask. Also, rinse the inside of the Snyder column with CF $H_2O$ into the flask.

8.3.11 Transfer the aqueous solution to a 125 mL separatory funnel. Rinse the flask with a few mL of CF $H_2O$ and add this rinsate to the separatory funnel. Extract the basic solutions with 20 mL of diethyl ether and allow the layers to separate. Drain the aqueous (bottom) layer back into the boiling flask and discard the diethyl ether through the top of the separatory funnel into a waste container. Pour the aqueous layer back into the separatory funnel and extract once more with 20 ml diethyl ether. Discard the ether fraction after this second extraction.

8.3.12 Add approximately 5 mL of cold, 1:1 $H_2SO_4$ to the contents of each separatory funnel to adjust the pH to less than 2. Add more acid if necessary.

8.3.13 Extract the acidic solutions once with 20 mL of diethyl ether by shaking for 2 minutes. Allow the layers to separate, drain the bottom layer (aqueous) back into the boiling flask and pour the diethyl ether extract through the top of the funnel into a 40 mL VOA vial that contains 1.0 g of $Na_2SO_4$. Repeat the extraction twice more with 10 mL aliquots of diethyl ether. Pour the ether layer into the 40 mL vial after each extraction.

8.3.14 Put a plug of glass wool in a powder funnel and fill 2/3 full with acidified $Na_2SO_4$, (0.1 mL concentrated $H_2SO_4$ added to 100 g of $Na_2SO_4$). Pour the extract through the $Na_2SO_4$ into a 50 mL receiver tube. Rinse the vial, then the $Na_2SO_4$ with small amounts of diethyl ether back into the receiver tube.

Note: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. If all the $Na_2SO_4$ solidifies in a cake, pour the extract through fresh $Na_2SO_4$ again.

8.3.15 Evaporate the sample extract on a nitrogen evaporator equilibrated at 28°C to an approximate volume of 2-5 mL. Transfer the sample to 12 mL vial calibrated at 10 and 1.0 mL. Rinse the 40 mL vial with 2 mL of diethyl ether and add this rinsate to the 12 mL vial. Continue evaporating the sample extract to 1.0 mL. Remove the vial from the nitrogen-evaporator and allow the extract to cool. Add 0.1 mL (100 uL) methanol to each extract.

Note: The concentration step is critical; losses of target compounds can occur if care is not taken. Rinse the walls of the vial frequently and monitor the temperature closely.

8.3.16 Proceed with esterification using diazomethane (8.4).

8.4 Diazomethane Esterification

8.4.1 The bubbler method of diazomethane generation is suggested for small batches (10-15) of samples that require esterification. The diazomethane derivatization procedure described below will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts due to the potential hazards associated with its use. The following precautions should be taken:

CAUTION: Diazomethane is a carcinogen and can explode under certain conditions.

8.4.1.1 Use a safety screen

8.4.1.2 Use mechanical pipetting aids.

8.4.1.3 Do not heat above 90°C -- EXPLOSION may result.

8.4.1.4 Avoid grinding surfaces, ground-glass joints, sleeve bearings, glass stirrers, scored glassware -- EXPLOSION may result.

8.4.1.5 Store away from alkali metals -- EXPLOSION may result.

8.4.1.6 Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

8.4.1.7 Wear a respirator.

8.4.2 Assemble the diazomethane bubbler (See Figure 1).

8.4.3 Add 10 mL of diethyl ether to tube #1.

8.4.4 Add 2 mL of 2% methoanolic KOH to tube #2. After the tube is on the bubbler, add 0.1 - 0.2 g diazald to tube 2. Immediately place the tip of the disposable pasteur pipet into the vials containing the sample extracts.

BE SURE THAT ALL SAMPLE EXTRACTS, INCLUDING THE METHYLATION STANDARD, HAVE 0.1 mL (100 uL) OF METHANOL ADDED TO THEM BEFORE ESTERIFICATION.

8.4.5 Apply nitrogen flow (10 mL/min) to bubble diazomethane through the sample extract for a few minutes (or until the yellow color of diazomethane persists). Allow the diazomethane to continue bubbling through the samples for 30 seconds after the yellow color persists. An additional 0.1 - 0.2 g of diazald may be added (after the initial diazald is consumed) to extend the generation of the diazomethane.

8.4.6 After each sample is esterified, remove the disposable pipets from the sample extract vials. Seal the vials and allow the extract to sit in a hood at room temperature for 30 min.

8.4.7 Destroy any unreacted diazomethane by adding 0.1 - 0.2 g of silica gel (100-200 mesh) to the extract vial. Allow the extract to stand until the evolution of nitrogen gas has stopped (approximately 20 min). Adjust the final volume to 10.0 mL with hexane.

8.4.8 The tubes that contained the diazald must be treated to destroy any unreacted diazomethane gas. Fill tubes with silica gel and allow to stand in the back of the hood overnight.

8.4.9 Be sure to clean all equipment after use. Use damp paper towels to swab up any excess diazald powder (DO NOT use a vacuum cleaner). Rinse any excess diazald off equipment with diethyl ether into a waste container. Dispose of the solvent waste properly.

9. QA/QC Requirements

9.1 QC Samples

Two Duplicate Control Samples (DCS) shall be extracted for every set of 20 samples. A single Control Sample (SCS) and method blank shall be extracted with every batch of samples.

10. Calculations

10.1 Record sample volume in liters (L) and weight in grams (g).

11. Reporting Requirements

11.1 Benchsheets and data entry into LIMS.

12. Review Requirements

12.1 Analyst initials and signs benchsheets for Level 1 review

13. Deviations

13.1 2% methanolic KOH replaces carbitol and 37% KOH in the esterification procedure.

13.2 Micro concentration of extracts is accomplished by the use of a nitrogen evaporative concentrator.

13.3 Extraction solvent for solids is diethyl ether.

14. References

14.1 LM-RMA-4007

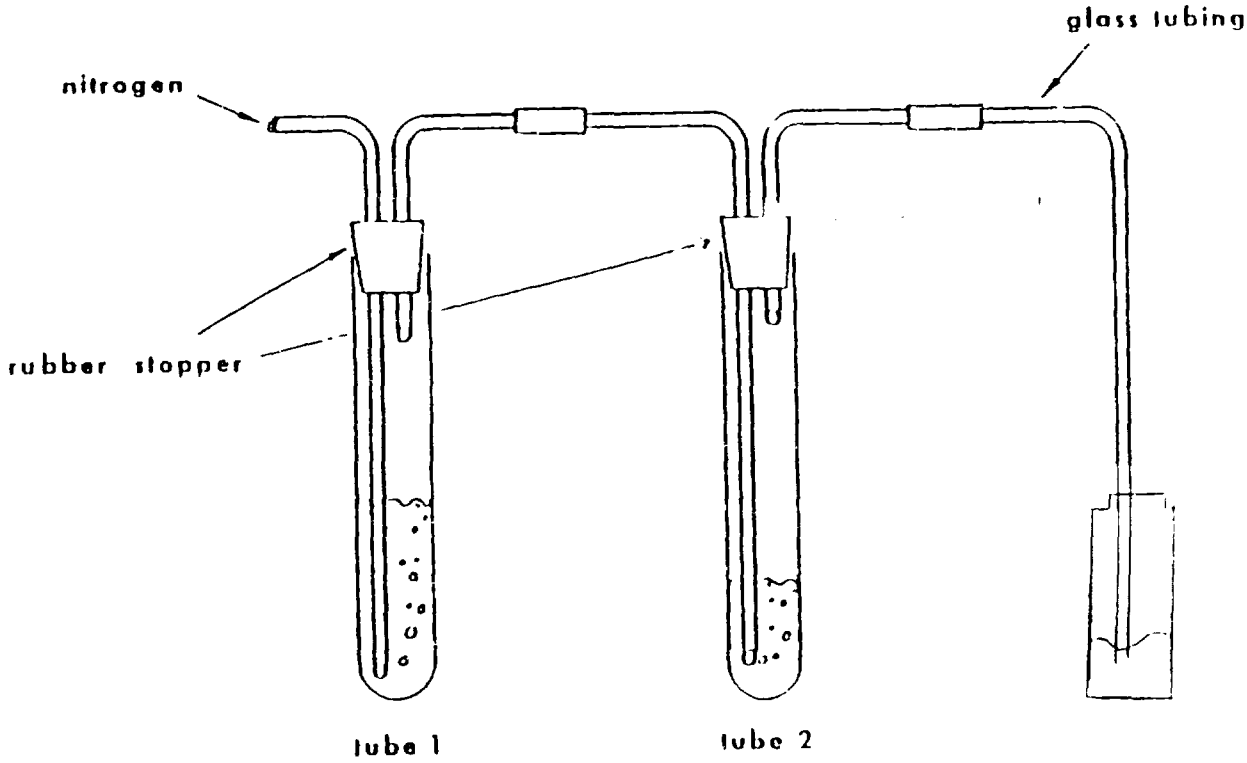
14.2 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," 40 CFR, Part 136.

14.3 "Test Methods for Evaluating Solid Waste," (SW-846) 3rd Edition (1986), Update I (1989). Method Ref. 8150.

14.4 "Enseco QAPP for Environmental Chemical Monitoring", most recent revision.

Figure 1.

Diazomethane generator (bubbler method)



STANDARD
OPERATING
PROCEDURE

Subject or Title: 

**Analysis of Environmental Samples for Chlorinated Phenoxyacid Herbicides (Modified EPA Method 615/8150)**

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| LM-RMA-4007 | 3.0 | December 10, 1991 |

Supersedes: Revision 2.0

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1. Scope and Application

   1.1 Analytes

| Prepared by: | Date: |
|---|---|
| GC Department | December 6, 1991 |
| Management Approval: Gary Walter | Date: 12-26-91 |
| QA Officer Approval: | Date: |

| Parameter | STORET No. | CAS No. |
|---|---|---|
| 2,4-D | 39736 | 94-75-7 |
| Dalapon | | 75-99-0 |
| 2,4-DB | | 94-82-6 |
| Dicamba | | 1918-00-9 |
| Dichlorprop | | 120-36-5 |
| Dinoseb | | 88-85-7 |
| MCPA | | 94-74-6 |
| MCPP | | 93-65-2 |
| 2,4,5-T | 39740 | 93-76-5 |
| 2,4,5-TP (Silvex) | 39760 | 93-72-1 |
| DCAA | | 19719-28-9 |

1.2 Matrices

This method is applicable to the analysis of chlorinated phenoxyacid herbicides in water, soil, solid, leachate, and vegetation samples.

1.3 Linear Range of Instrument

Calibration standards are run at 6 levels and a quadratic calibration curve is generated for each compound. The calibration range normally covers one order of magnitude.

1.4 Analysis time is approximately 30 minutes per sample or standard.

1.5 Sample preparation requires at least three days for completion.

2. Summary of Method

2.1 This method is applicable to the chlorinated phenoxyacid herbicides listed in 1.1, their salts, and esters. The herbicide is hydrolyzed to the acid form, solvent extracted, then converted to the methyl ester and analyzed by electron-capture gas chromatography.

3. Comments

3.1 Helpful Hints

After analyzing herbicides it is necessary to change the liner and cut the front ends of the columns every seven days. After performing maintenance, raise injection port temperature to 260°C, oven to 280°C, and detector to 350°C for a few hours while injecting hexane every two minutes. This procedure effectively cleans the GC system.

4. Safety Issues

4.1 Diazomethane is used for esterification. Not only is diazomethane a toxic carcinogen, but it may explode if improperly generated or used. The following precautions must be followed:

4.1.1 Use only a well ventilated hood - do not breathe vapors.

4.1.2 Use a safety screen.

4.1.3 Use mechanical pipetting aides.

4.1.4 Do not heat above 90°C - EXPLOSION may result.

4.1.5 Avoid grinding surfaces, ground glass joints, sleeve bearings, glass stirrers - EXPLOSION may result.

4.1.6 Store away from alkali metals - EXPLOSION may result.

4.1.7 Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

4.1.8 Wear a respirator.

4.2 Ethyl ether is a flammable solvent with a low flash point. Ethyl ether vapors are heavier than air and may "creep" along a bench top to a source of ignition. Use only in a properly functioning hood.

4.3 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

5. Sample Collection, Preservation and Handling

5.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed; however, the bottle must not be pre-rinsed with sample before collection.

5.2 The samples must be iced or refrigerated at 4°C from the time of collection until extraction.

5.3 All water samples must be extracted within 7 days and all soils within 14 days of collection. All samples must be analyzed within 40 days from the date that extraction begins.

6. Apparatus and Materials

6.1 Sample bottle - Amber borosilicate or flint glass, 1-liter or 1-quart volume, fitted with screw caps lined with TFE-fluorocarbon or aluminum foil. If amber bottles are not available, protect samples from light. The container and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

6.2 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.3 Gas chromatograph - Analytical system complete with gas chromatograph suitable for split/splitless capillary injection and all required accessories including: syringes, analytical columns, gases, detector and a data system or strip-chart recorder to measure peak areas. Enseco-RMAL uses a VG data system to quantify data.

6.3.1 Column 1 - $RT_X$-5, 30 m, 0.25 mm ID, 0.25 um film thickness or equivalent.

6.3.2 Column 2 - $RT_X$-20, 30 m, 0.25 mm ID, 0.25 um film thickness or equivalent.

6.3.3 A "guard column" - 5 m, 0.25 mm with no coating.

6.3.4 A glass "Y" connector - connects both analytical columns to the guard column.

6.3.5 Detectors - electron capture.

7. Reagents and Standards

7.1 Reagent water - Reagent water is defined as a water in which an interferent is not observed at the method detection limit of each parameter of interest.

7.2 Methanol and Hexane - Pesticide quality or equivalent.

7.3 Ethyl ether - (Burdick and Jackson Product No. 107 or equivalent). Nanograde, redistilled in glass if necessary. Ether must be free of peroxides as indicated by EM Quantity test strips. Procedures recommended for removal of peroxides are provided with the test strips. Ethers must be tested daily for peroxide formation during use.

7.4 Calibration standard solutions.

7.4.1 The methyl ester standards used to calibrate the GC system are purchased as a certified solution. Serial dilutions are made to obtain standard solutions at six different levels.

7.4.2 Solutions used by the prep lab for spikes and methylation check standards are prepared from pure standard materials. These standards are all in the acid form and at a concentration of approximately 1.0 ug/uL.

7.4.2.1 Prepare primary standard solutions by accurately weighing about 0.0100 g of pure acids. Dissolve the material in pesticide quality hexane and dilute to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. If compound purity is certified at 99% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8. Procedure

8.1 Method Summary

This method provides extraction and esterification procedures for the analysis of chlorinated phenoxyacid herbicides. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material

is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivitizing agent. After excess reagent is removed, the esters are analyzed by gas chromatography employing an electron capture detector (Goerlitz and Lamar, 1967).

8.2 Sample Preparation and Extraction - See RMAL SOP No. LM-RMA-4044

9. Analysis

9.1 Sample analyses are performed simultaneously on dissimilar analytical columns.

9.1.1 The linear velocity ($\bar{u}$) should be approximately 40 cm/second. A typical temperature program for this analysis is as follows.

| | |
|---|---|
| $T_1$ | 45°C |
| $t_1$ | 1.0 minute |
| $R_1$ | 20 °C/minute |
| $T_2$ | 160°C |
| $t_2$ | 4.0 minutes |
| $R_2$ | 1.3 °C/minute |
| $T_3$ | 180°C |
| $t_3$ | 0.0 minute |
| $R_3$ | 50.0 °C/minute |
| $T_4$ | 280°C |
| $t_4$ | 1.0 minute |

Recommended GC conditions are as follows:

injector 240°C
detector 325°C
purge on 0.60 minute
carrier gas $H_2$
auxiliary gas $N_2$
volume injected 1-2 uL

9.2 Calibration of Instrument

9.2.1 Run six levels of standards to generate a 6-point quadratic calibration curve for each standard. RSE values less than 15% are required to quantify data. A mid-level calibration check

standard is run after every 10 samples and at the end of the run. Insert a hexane blank before each check standard. If the check standards are within 20% difference from the curve the instrument is in control and data may be quantified. If the change in response factors exceeds 20% for selected compounds, consult an experienced analyst. The recommended internal standard, 4,4'-dibromooctafluorobiphenyl, is added to all samples and standards at 0.025 ug/mL. Samples may be quantified using internal standard calculations if the percent difference between the areas of the internal standard in the sample and the standard is $\leq$ 30%.

10. QA/QC Requirements

10.1 QC samples: With each set of samples methylated, a Methylation Standard is run consisting of 2,4-D, Silvex, and 2,4-DB to determine if the methylation step is working properly. There are no recovery limits established for the Methylation Standard. Recoveries of greater than or equal to 80% are desirable.

10.2 For standard product herbicide tests two lab control standards are prepared for every twenty samples and a spike control standard is prepped with every batch. Recoveries for both soils and waters are based on historical data and are periodically updated. The relative percent difference of the two replicates is limited to 20%. 80% of all QC values are required to be within limits before proceeding. A reagent blank is extracted with every group of samples.

10.3 DCAA is used as the surrogate.

10.4 Corrective action: See LP-RMA-0022; Enseco QAPP, Revision 3.3, May, 1989, and M-EQA-0002.

11. Calculations: ug/mL can be read directly from the internal or external standard table.

11.1 For waters:

$$\frac{ug}{mL} \text{ of analyte} \times \frac{\text{final vol (mL)}}{\text{liters extracted}} \times \text{dilution factor} = \frac{ug}{L}$$

11.2 For soils:

$$\frac{ug}{mL} \text{ of analyte} \times \frac{\text{final vol (mL)}}{\text{kg extracted}} \times \text{dilution factor} = \frac{ug}{kg}$$

Reporting units are ug/L for liquids and ug/kg for solids.

11.3 Unless specifically requested by the client, two significant figures are reported. Values below detection limit are reported as not detected (ND).

12. References

12.1 "Method for Chlorinated Phenoxy Acid Herbicides in Industrial Effluents" Federal Register, Vol. 38, No. 75, Pt II.

12.1.1 "Simultaneous Determination of 2,4-D, 2,4,5-T and 2-Methoxy-3,6-Dichlorobenzoic Acid in Soil and Water by GC/ECD" by R. Purkazastha, J. Agric and Food Chem., 74, 22, 3, p. 453-458.

12.2 USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, November, 1986, SW-846, Third Edition, Method 8150.

13. Reporting Limits

The following reporting limits have been established based upon MDL data and the linear range of the detector.

| Target Compound | Water (ug/L) | Soil (mg/kg) | Waste (mg/kg) |
|---|---|---|---|
| Dalapon | 5.0 | 100 | 0.10 |
| Dicamba | 0.50 | 10 | 0.010 |
| MCPP | 250 | 5000 | 5.0 |
| MCPA | 250 | 5000 | 5.0 |
| Dichlorprop | 1.0 | 20 | 0.020 |
| 2,4-D | 2.0 | 40 | 0.040 |
| 2,4,5-TP (Silvex) | 0.50 | 10 | 0.010 |
| 2,4,5-T | 0.50 | 10 | 0.010 |
| 2,4-DB | 5.0 | 100 | 0.10 |
| Dinoseb | 1.0 | 20 | 0.020 |

Subject or Title: Page 1 of 22

Toxicity Characteristic Leaching Procedure
(TCLP)

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| LM-RMA-5002 | Original | September 25, 1990 |

Supersedes: None

## 1. Scope and Application

### 1.1 Analytes

The TCLP is designed to determine the mobility of certain organic and inorganic contaminants. The specific list of analytes may be found in Appendix I.

### 1.2 Reporting limits

The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits. The specific list of analytes and their regulatory limits may be found in Appendix I.

### 1.3 Applicable Matrices

The TCLP is applicable to liquid, solid and multiphase wastes.

### 1.4 Dynamic Range

Consult the individual analysis SOPs (see Section 15.2).

### 1.5 Analysis Time

Consult the individual analysis SOPs (see Section 15.2).

| Prepared by: | Date: |
|---|---|
| Will Pratt | September 18, 1990 |
| Management Approval: | Date: |
| | 9/25/90 |
| QA Officer Approval: | Date: |
| | 9/25/90 |

## 2. Method Summary

The TCLP is designed to generate an aqueous leachate of a waste sample for analysis of various organic and inorganic constituents. This leachate is intended to represent leaching the waste would experience under the condition of co-disposal with municipal waste in a sanitary landfill.

The sample is filtered to separate the solid and liquid portions. The solid portion is leached with an appropriate leaching solution. The resulting solid phase leachate is recombined with the initial liquid filtrate. The resulting TCLP leachate is then prepared and analyzed by appropriate methodology.

Two types of leachate may be generated: one for analysis of Non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals) or one from a Zero Headspace Extractor (ZHE) for analysis of Volatile organic constituents.

## 3. Comments

Various terminology is used in this procedure which may be ambiguous. To clarify, "leachate" is used to refer to the TCLP solution generated from this procedure. This leachate may be analyzed directly for volatile constituents, prepared for non-volatile organics by an appropriate "extraction" or prepared for metals by "digestion".

### 3.1 Interferences

3.1.1 Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized.

3.1.2 All pH probes are to be calibrated daily (see Appendix II for the calibration procedure).

3.1.3 The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching.

3.2 Helpful Hints

3.2.1 If a "Total" analysis of the waste demonstrates that individual contaminants are absent, or present in such low concentrations that the regulatory limits could not possibly be exceeded, the TCLP need not be performed.

3.2.2 The ZHE device has approximately a 500-mL capacity. Based on the need to add an amount of extraction fluid equal to 20 times the mass of the "solid" phase, the ZHE can therefore accommodate a maximum of 25 grams of "solid" (see Section 10.7).

## 4. Safety Issues

4.1 Proper protective gear (i.e. safety glasses, gloves, lab coat, etc.) should be worn at all times.

4.2 Samples should be opened and handled in a properly vented fume hood.

4.3 Concentrated reagents should be handled in a properly vented fume hood. Appropriate caution should be used when handling diluted reagents.

4.4 Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized.

4.5 Consult the Enseco Health and Safety Manual and specific Material Safety Data Sheets (MSDS) for additional information.

## 5. Sample Collection, Preservation, and Holding Times

5.1 Samples being analyzed for organic compounds should be collected and stored in glass, air-tight containers to minimize the loss of volatiles. Samples being analyzed for metals only may be collected in glass or plastic containers.

5.2 Samples should be refrigerated to $4^{o}C$. No preservatives shall be added until AFTER leachate generation (see Sections 9.20 and 10.23).

5.3 Samples are subject to appropriate treatment within the following time periods (days):

| From: / To: | Collection / TCLP Leach | TCLP Leach / Preparation | Preparation / Analysis | Total Elapsed Time |
|---|---|---|---|---|
| Volatiles | 14 | N/A | 14 | 28 |
| Semi-volatiles | 14 | 7 | 40 | 61 |
| Mercury | 28 | N/A | 28 | 56 |
| Other metals | 180 | N/A | 180 | 360 |

6. Apparatus

6.1 Stainless steel pressure filtration apparatus (142 mm dia.), capable of 0-50 psi.

6.2 Vacuum filtration apparatus, capable of 0-50 psi.

6.3 Borosilicate glass fiber filters, 0.6 - 0.8 um (Whatman GF/F 14.2 cm, 0.7 um or equivalent).

6.4 Cellulose acetate filters, 0.65 um (Corning 25932-200 custom or equivalent), for Metals ONLY.

6.5 Zero Headspace Extractor (ZHE), gas-pressure actuated, Millipore YT30090HW or equivalent.

6.6 ZHE rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent.

6.7 Gas-tight syringes, 100 mL capacity, Hamilton 0158330 or equivalent.

6.8 Top loading balance, 0.01 g min/4000 g max capability.

6.9 pH meter and probe, capable of reading to the nearest 0.01 unit.

6.9 pH probes and splitter box.

6.10 Magnetic stirrer/hotplate and stirring bars.

6.11 VOA vials, 20 mL, with caps and septa.

6.12 Glass jars, 1/2 - 1 gallon, with teflon lid-inserts.

6.13 Nalgene plastic bottles, 1 liter.

6.14 Miscellaneous laboratory glassware and equipment.

7. Reagents and Standards

7.1 Reagent water: For Non-volatile constituents, ASTM Type II water (Milli-Q or equivalent). For Volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through 500 g activated carbon).

7.2 Hydrochloric acid, 1 N: Carefully add 85 mL concentrated reagent grade HCl to 915 mL Milli-Q. Cap and shake to mix well.

7.3 Nitric acid, 1 N: Carefully add 64 mL concentrated reagent grade $HNO_3$ to 936 mL Milli-Q. Cap and shake to mix well.

7.4 Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 1 L Milli-Q. Cap and shake until completely dissolved.

7.5 Acetic acid, glacial: Concentrated, reagent grade liquid (HOAc).

7.6 pH buffer solutions: 7, 4, 10. Commercially available.

7.7 Leaching Fluids: The pH of both solutions should be monitored daily and the pH probes are to be calibrated daily (see Appendix II).

7.7.1 Fluid #1: Carefully add 6.25 mL glacial HOAc and 64.55 mL of 1 N NaOH to 500 mL Milli-Q in a 1 gallon glass jar. Dilute to a final volume of 1 L with Milli-Q, cap and shake to mix well. When correctly prepared, the pH of this solution is 4.93 +/- 0.05.

7.7.2 Fluid #2: Carefully add 4.96 mL glacial HOAc to 500 mL Milli-Q in a one gallon glass jar. Dilute to a final volume of 1 L with Milli-Q, cap and shake to mix well. When correctly prepared, the pH of this solution is 2.88 +/- 0.05.

7.8 Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated $HNO_3$ to 500 mL Milli-Q. Cap and shake to mix well.

8. Procedure: Preliminary Evaluations

NOTE: Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot MAY undergo the actual TCLP extraction for Non-volatiles ONLY if it has NOT been oven dried as in Section 8.2.10. If the solid portion IS oven dried, a separate aliquot must be used for the actual leaching procedure.

8.1 Determination of Required Analyses

8.1.1 Determine from the TCLP backlog which samples require the generation of TCLP leachates and which leachates are to be generated (volatiles, etc.).

8.1.2 Consult the holding times for the appropriate tests (Section 5.3) and prioritize such that they ARE NOT EXCEEDED.

8.1.3 Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated according to the following:

| Prep | Analysis | Req'd Volume |
|---|---|---|
| P-TCLP-VOA | Volatiles | 3 x 20 mL |
| P-TCLP-C/EXORG | Semi-volatiles | 400 mL |
| " | Pesticides | 200 mL |
| " | Herbicides | 200 mL |
| P-TCLP-C/M | Metals | 150 mL |

8.2 Determination of Percent Solids

8.2.1 Examine the sample and determine the type of filtration to employ. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e. it is a solid), then proceed to Section 8.3 (Particle-size Reduction). If the sample is mostly non-viscous liquid (water or thin oil), vacuum filtration may be used. If the sample is viscous (sludge or high solids content), use pressure filtration.

8.2.2 Pre-weigh the filter device and the filtrate collection vessel. Record both masses on the benchsheet. Assemble the filtration apparatus (use blunt forceps to handle the filter membrane).

8.2.3 Homogenize the waste and weigh out a 100 g minimum subsample. Quantitatively transfer the sample to the filter apparatus and record the mass of any remaining residue. Calculate and record the mass of sample.

8.2.4 Slowly apply gentle pressure or vacuum of 10 psi. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter in a 2 minute period.

8.2.5 Repeat previous step up to 50 psi max in 10 psi increments. Remove the filtrate collection vessel and determine and record its mass.

8.2.6 Determine and record the mass of the initial filtrate. Retain the filtrate for use in Section 8.5.

8.2.7 Determine and record the "solid" phase mass by subtracting the mass of the initial filtrate from the mass of the subsample. Retain the solid phase for use in Section 8.3.

NOTE: Some samples will contain liquid material that does not filter (e.g. oil). Do not attempt to filter the sample again. The "solid" phase is defined as any material remaining in the filtration apparatus.

8.2.8 Determine and record the % Solids (see Section 12.1):

If the % Solids is slightly greater than or equal to 0.5% and it is believed that some of the mass is due to moisture entrained in the filter, proceed to 8.2.9.

If the % Solids is significantly $>$ 0.5 %, proceed to Section 8.3.

If the % Solids is $<$ 0.5 %, discard the solid phase. No leaching will be necessary; the filtrate will be considered the TCLP leachate. Proceed to Section 8.5.4.

8.2.9 Remove the solid phase and filter from the filtration apparatus.

8.2.10 Dry the filter and solid phase at 100 +/- 20°C until two successive weighings yield the same value within +/- 1%. Record the final weight. Return to 8.2.8.

8.3 Particle-size Reduction

Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e. capable of passing through a 9.5 mm (0.375 inch) standard sieve). Record on the benchsheet.

NOTE: Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick).

8.4 Determination of Appropriate Leaching Fluid

8.4.1 Weigh out a 5.0 g sub-sample of the solid phase into a 250-mL beaker.

8.4.2 Add 96.5 mL of Milli-Q water, cover with a watchglass, and stir for 5 minutes on a hotplate/stirrer.

8.4.3 Measure and record the pH. If the pH is LESS THAN OR EQUAL to 5.0, use Fluid #1 and proceed to Section 8.5.

8.4.4 If the pH is GREATER than 5.0, add 3.5 mL 1 N HCl, cover with a watchglass and while stirring, heat at 50°C for 10 minutes. Remove, cool.

8.4.5 Again, measure and record the pH. If the pH is less than or equal to 5.0, use Fluid #1. If the pH is greater than 5.0, use Fluid #2.

8.5 Determination of Filtrate/Leachate Compatibility

8.5.1 Place 5 mLs of the appropriate leaching fluid (determined in the previous step) into a blood dilution vial.

8.5.2 Add 5 mLs of the initial filtrate, cap and shake. Examine for miscibility and record on the benchsheet.

8.5.3 If the phases are miscible, the initial filtrate and solid phase leachate may be physically recombined upon completion of the leachate generation. If the phases are NOT miscible, the initial filtrate and the solid phase leachate should be prepared and analyzed separately and the results mathematically combined (see Section 12.2).

8.5.4 For samples requiring TCLP Semi-volatiles, Pesticides, Herbicides or Metals analysis, proceed to Section 9.

8.5.5 For samples requiring TCLP Volatiles (ZHE) analysis, proceed to Section 10.

9. Procedure: Non-volatile Constituents: Semi-volatiles, Pesticides, Herbicides, Metals (Bottle leach)

NOTE: The aliquot used in the Preliminary Evaluation MAY be used for this procedure ONLY if it was not oven dried.

NOTE: All masses should be recorded to the nearest 0.01 g.

9.1 Examine the sample and determine the type of filtration to employ. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e. it is a solid), then homogenize the sample, weigh out an appropriate size subsample, record the mass, and proceed to Section 9.10. If the sample is mostly non-viscous liquid (water or thin oil), vacuum filtration may be used. If the sample is viscous (sludge or high solids content), use pressure filtration.

9.2 Pre-weigh the filter device and the filtrate collection vessel. Record both masses on the benchsheet. Assemble the filtration apparatus (use blunt forceps to handle the filter membrane). If evaluating for the mobility of metals, wash the filter with dilute $HNO_3$ and rinse with Milli-Q.

9.3 Determine the optimum sample size to use based on the required analyses (Section 8.1) and the preliminary percent solids data (Section 8.2). A sufficient volume of final leachate should be generated to support all required analyses.

9.4 Prepare one leachate blank PER 20 SAMPLES, PER LEACHATE FLUID, PER LEACH VESSEL TYPE (this yields 3 possibilities: ZHE, Bottle-Fluid #1, Bottle-Fluid #2). This allows for samples to be leached on different days without leaching a blank every day. Samples which are NOT leached (merely filtered) will ALSO be associated with these blanks.

9.5 Homogenize the waste. Weigh out the subsample into the filtration device and record the mass. Quantitatively transfer the sample to the filter apparatus and record the mass of any remaining residue.

9.6 Slowly apply gentle pressure or vacuum of 10 psi. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period.

9.7 Repeat previous step up to 50 psi max in 10 psi increments. Remove the filtrate collection vessel and determine and record its mass.

9.8 Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil and retain for use in Section 9.17.

9.9 Determine and record the "solid" phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

9.10 Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e. capable of passing through a 9.5 mm (0.375 inch) standard sieve). Record on the benchsheet.

NOTE: Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick).

9.11 Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 8.1.3.

9.12 Divide the total volume of solid phase leachate required by 20 to determine the mass of solid phase required for leaching. Round this mass UP to the nearest 5 g.

9.13 Weigh the required mass of solid phase into an appropriate bottle (plastic for Metals only, glass for all others) and slowly add 20 times its mass of appropriate leaching fluid (Section 8.4). (For example, 20 g of sample would require 400 mL of leaching fluid).

9.14 Cap the bottle tightly, secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours.

NOTE: The bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

9.15 Remove the bottle and filter the sample using vacuum or pressure filtration. The entire sample need not be filtered, however, sufficient volume should be generated to support the required analyses. See Section 8.1.3. Cellulose filters may be used for leachates being prepared for metals only.

9.16 If the waste contained no initial filtrate, this solution is defined as the TCLP leachate.

9.17 If the waste DID yield an initial filtrate, consult the benchsheet for initial filtrate/leachate compatibility. If they are compatible, they are to be combined in the correct proportions (see Section 12.2) and mixed well. This combined solution is defined as the TCLP leachate.

9.18 If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 12.3 and the attached example.

9.19 Measure and record the pH of the TCLP leachate. (Do not attempt to measure the pH of oily samples as the probe may be rendered inoperable.)

9.20 Immediately preserve the leachate as follows:

| | |
|---|---|
| Metals | pH $< 2$ w/50% $HNO_3$ for non-oils. |
| Others | Refrigerate to $4^oC$. |

9.21 Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis. Also submit copies of the prep benchsheets with the samples. See Sections 8.1 and 15.2 for additional information.

10. Procedure: Volatile Constituents (ZHE)

NOTE: Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g. metals, pesticides, etc.).

NOTE: All masses should be recorded to the nearest 0.01 g.

10.1 Assemble the ZHE apparatus, close all valves except the gas inlet/outlet valve and pressurize to 50 psi. Allow to stand for 1 hour and check the pressure on the built-in gauge to make sure it is not leaking. If the pressure is NOT 50 psi, consult your supervisor. Disassemble the ZHE leaving the piston in the ZHE body.

10.2 Adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

10.3 Consult the benchsheet and examine the sample. If the sample appears to be different from the preliminary information found on the benchsheet, consult your supervisor. If the sample is < 100% solids (i.e. yielded free liquid upon filtration), proceed to Section 10.6.

10.4 Prepare one leachate blank PER 20 SAMPLES. This information will need to be tracked for the purpose of assigning Prep QC Lot numbers. This allows for samples to be leached on different days without leaching a blank every day.

10.5 Homogenize the waste. If the preliminary evaluations indicated the need for particle size reduction, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e. capable of passing through a 9.5 mm (0.375 inch) standard sieve; do NOT sieve the sample, however). Record on the benchsheet.

NOTE: Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick).

10.6 Place the ZHE apparatus on the balance and tare the balance.

10.7 Determine the appropriate size subsample to weigh using the percent solids information from Section 8.2. For wastes containing < 5.0 % solids, weigh out enough sample to support the volatiles analyses (Section 8.1) since the the liquid portion of the waste after filtration is defined as the TCLP leachate.

10.8 Homogenize and weigh an appropriate size subsample of the waste into the ZHE and record the mass. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

10.9 Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

10.10 Carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure. If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 10.14.

10.11 Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the syringe to the liquid inlet/outlet valve and open the valve.

10.12 Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period. If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe and return to Section 10.11.

10.13 Repeat previous step up to 50 psi max in 10 psi increments. Remove the syringe and record the total filtrate volume. Close the valve and discontinue gas pressure. Transfer the filtrate to VOA vials and label appropriately.

NOTE: If the original waste contained < 5.0 % solids (Section 8.2), this filtrate is defined as the TCLP leachate and you may proceed to Section 10.24. Otherwise, save the vials for recombination as in Section 10.22.

NOTE: The material remaining in the ZHE is defined to be the "solid" phase.

10.14 Based on the information from Sections 8.2 and 10.7, determine the volume of Fluid #1 to load into the ZHE on the "solid" phase.

NOTE: The ZHE prep uses ONLY Fluid #1.

10.15 Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

10.16 Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.

10.17 Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) 2 or 3 times. Reposition the ZHE in the vertical position.

10.18 Pressurize the ZHE to 5-10 psi. Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

10.19 Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours.

10.20 Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 10.1 and repeat the leachate with a new aliquot of sample.

10.21 Attach a syringe and open the liquid inlet/outlet valve to collect the aqueous leachate.

10.22 If the waste contained an initial filtrate (Section 10.12) that is miscible with the solid phase leachate (as determined in Section 8.5), the solid phase leachate may be directly recombined in the correct proportions (see Section 12.2) with the initial filtrate. If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately.

10.23 Following collection, store the TCLP leachate in 3 20-mL VOA vials with minimal headspace at 4°C and prepare for analysis as soon as possible using the appropriate organic extraction procedure (see Section 15.2).

10.24 If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.3.

## 11. QA/QC Requirements

### 11.1 QC Samples

11.1.1 One blank per Fluid type per extraction vessel type (using the same leaching Fluid as used for the samples) shall be prepared for every twenty samples (whether leached or not). The blanks are generated in the same way as the samples.

11.1.2 Duplicate Control Samples (DCSs) are required with each batch of 20 or fewer samples. The DCSs shall be generated AFTER the TCLP leachate at the time of the preparative digestion or extraction. Consult the individual analysis SOPs (see Section 15.2).

11.1.3 Duplicates may be required for project specific QC.

11.1.4 Matrix spikes may be required for project specific QC. They shall be added AFTER the TCLP leachate is generated but BEFORE PRESERVATION.

### 11.2 Acceptance Criteria

11.2.1 Consult the Enseco QAPP and Internal QC SOP (M-EQA-002) for method blank acceptance criteria.

11.2.2 Consult the individual analysis SOPs for DCS recovery criteria.

11.2.3 Acceptance criteria for project specific duplicates have not been established.

11.2.4 Acceptance criteria for project specific matrix spikes have not been established.

11.3 Corrective Actions

11.3.1 Consult the Enseco QAPP and Internal QC SOP (M-EQA-002) for corrective action for blanks and DCSs.

11.3.2 No corrective actions have been defined for duplicates.

11.3.3 Evaluation of matrix spike results and recovery correction of data based on same will not be employed by Enseco but will be the sole responsibility of the client. See Section 15.3.5.

11.3.4 If the ZHE exhibits loss of pressure, repressurize to 50 psi and submerge in water to check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, replace O-rings as necessary. Retest the device. If leakage problems cannot be resolved, the ZHE is unsuitable for use and the manufacturer should be contacted.

## 12. Calculations

12.1 Calculation of % Solids:

$$\frac{\text{Mass, "solid" phase}}{\text{Mass, initial subsample}} * 100$$

12.2 Calculation of volume of initial filtrate phase to recombine with solid phase extract:

$$\frac{\text{Mass, extracted solids}}{\text{Mass, solids}} * \text{Total Volume, initial filtrate}$$

12.3 Mathematical recombination of analytical results:

$$\text{Final Analyte Concentration} = \frac{(V_1 * C_1) + (V_2 * C_2)}{V_1 + V_2}$$

where $V_1$ = total volume of the initial filtrate phase (L),
$C_1$ = analyte concentration in initial filtrate phase (mg/L),
$V_2$ = volume of the theoretical solid phase extract (L),
$C_2$ = analyte concentration in solid phase extract (mg/L),

and where $V_2$ = total solid phase mass * 20.

## 13. Reporting Requirements

### 13.1 Units

Regardless of the nature of the sample, all results are reported in units of mg/L.

### 13.2 Limits, Significant Figures

Consult the appropriate analytical methods (Section 15.2).

### 13.3 LIMS Data Entry

13.3.1 Enter the QC Lot number of the leachate blank in the TCLP leachate prep test.

13.3.2 The usual standards for the remaining data entry apply.

### 13.4 Anomalies

All anomalies observed during the leach procedure must be noted on an anomaly form. Some examples of such anomalies are:

13.4.1 Sample was monolithic - subsample was obtained by (crushing, cutting, grinding, sawing, etc.).

13.4.2 Insufficient sample - less than the required 100 g minimum was available.

13.4.3 Multiple phases - "X" phases were present.

13.4.4 Sample was oil - single phase.

13.4.5 Sample contained liquid which did not filter under test conditions.

13.5 Data Package

The original TCLP data package will contain the following information: Cover sheet, Special Instructions (only when applicable), Leachate benchsheet, Blank tracking form and any additional information. This will be filed normally and a copy of the data package will be submitted to each analytical group (as appropriate) with each batch of leachates generated.

## 14. Review Requirements

14.1 Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation form was properly documented and routed.

14.2 If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$\text{Total} >= \text{TCLP} * \frac{\{(\text{Solids mass (g)} * 20) + \text{Filtrate volume}\}}{1000 * \text{sample mass used (kg)}}$$

NOTE: When the sample is 100% Solids, this equation reduces to:

$$\text{Total} >= \text{TCLP} * 20$$

## 15. References

15.1 Method Source: Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

15.2 Related Documents

15.2.1 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

15.2.2 Technical Background Document and Response To Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

15.2.3 LM-RMA-2002: [TCLP] Metals: Acid Digestion

15.2.4 LM-RMA-2005: Mercury

15.2.5 LM-RMA-2006: GFAA (Se)

15.2.6 LM-RMA-2001: ICP

15.2.7 LM-RMA-3001: VOAs

15.2.8 LM-RMA-3013: BNAs

15.2.9 LM-RMA-4003: Pesticides

15.2.10 LM-RMA-4007: Herbicides

15.3 Deviations from Source Method and Rationale

15.3.1 Section 8: Preliminary Evaluations. Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction". No reason is given there or in the background document. It is assumed that the reasoning is that solid phase degradation may occur upon heating to determine % dry solids. In order to reduce the time involved, and based on said assumption, the sample used for the preliminary evaluation may be used for the actual TCLP leach PROVIDED THE SAMPLE WAS NOT OVEN DRIED during the % solids determination.

15.3.2 Section 8.2.4: Preliminary Determination of Percent Solids. The wording of Section 7.1.1.7 of the source method has been modified. Filtration proceeds until "...no SIGNIFICANT additional liquid has passed through the filter..." to allow for judgement in determining the total time required for filtration.

15.3.3 Section 8.5: Preliminary Determination of Filtrate/Leachate Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed and incorporated into the Preliminary Determinations section.

15.3.4 Section 9.4: Leachate Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." Enseco-RMAL has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e. Bottle or ZHE) per leach fluid used.

15.3.5 Section 9.15: Filtration. Section 7.2.12 of the source method requires the use of glass fiber filters for the filtration of the Non-volatiles TCLP leachates. Cellulose filter material has been deemed acceptable by Enseco for the filtration of metals only.

15.3.6 Section 10.21: Volatiles Filtrate Collection. Section 7.3.9 of the source method requires that the mass of ZHE filtrate be determined. Enseco-RMAL currently uses graduated syringes for collection and therefore determines ZHE filtrate volume.

15.3.7 Section 11.1.4: QA/QC - Matrix Spikes. Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Because of the diverse nature of samples that Enseco-RMAL receives, the logical conclusion of this requirement would be to spike every sample. Further, Section 8.2.3 of the source method states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The standard Enseco QAPP is designed to address the performance monitoring of analytical methodology through the LCS program. As a result, Enseco-RMAL will perform matrix spikes only as requested by the client and will NOT perform calculations for recovery OR correct for alleged analytical bias. The use and interpretation of the matrix spike results shall be the sole responsibility of the client.

# APPENDIX I

## Toxicity Characteristic Analytes and Regulatory Levels

(Final Rule)

| Contaminant | mg/L |
|---|---|
| Arsenic | 5.0 |
| Barium | 100.0 |
| Benzene | 0.5 |
| Cadmium | 1.0 |
| Carbon tetrachloride | 0.5 |
| Chlordane | 0.03 |
| Chlorobenzene | 100.0 |
| Chloroform | 6.0 |
| Chromium | 5.0 |
| Cresols | 200.0 |
| 2,4-D | 10.0 |
| 1,4-Dichlorobenzene | 7.5 |
| 1,2-Dichloroethane | 0.5 |
| 2,4-Dinitrotoluene | 0.13 |
| 1,1-Dichloroethylene | 0.7 |
| Endrin | 0.02 |
| Heptachlor | 0.008 |
| Hexachlorobenzene | 0.13 |
| Hexachlorobutadiene | 0.5 |
| Hexachloroethane | 3.0 |
| Lead | 5.0 |
| Lindane | 0.4 |
| Mercury | 0.2 |
| Methoxychlor | 10.0 |
| Methyl ethyl ketone | 200.0 |
| Nitrobenzene | 2.0 |
| Pentachlorophenol | 100.0 |
| Pyridine | 5.0 |
| Selenium | 1.0 |
| Silver | 5.0 |
| Tetrachloroethylene | 0.7 |
| Toxaphene | 0.5 |
| Trichloroethylene | 0.5 |
| 2,4,5-Trichlorophenol | 400.0 |
| 2,4,6-Trichlorophenol | 2.0 |
| 2,4,5-TP (Silvex) | 1.0 |
| Vinyl chloride | 0.2 |

## APPENDIX II

### pH Meter and Probe Calibration Procedure

Corning 255 Ion meter

1) Depress the MODE button until the meter is in the pH mode.

2) Depress the CAL key.

3) Depress the Right arrow (2 pt calibration).

4) Rinse the electrode with DI water, then with the 7 buffer.

5) Place the electrode in the 7 buffer. Depress the READ button.

6) Allow the reading to stabilize. Depress the READ button again.

7) Rinse the electrode with DI water, then with the 4 buffer.

8) Place the electrode in the 4 buffer. Depress the READ button.

9) Allow the reading to stabilize. Depress the READ button again.

10) The screen will read SAMPLE. Rinse the electrode with DI water and place the electrode in the 10 buffer. Allow the reading to stabilize. It should read between 9.5 and 10.5.

11) Rinse the electrode with DI water and store in either DI water or the 7 buffer. The meter is now calibrated and ready for use.

NOTE: If the 10 buffer does NOT read between 9.5 and 10.5, recalibrate as above. If the second attempt is unsuccessful, consult your supervisor.

NOTE: DO NOT allow the electrode to dry out. DO NOT immerse the electrode in oily samples. Consult your supervisor.

HAUSER

HAUSER CHEMICAL RESEARCH, INC.

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A Division

VISCOSITY-KINEMATIC Standart test method ASTM D445-86
Annual book of ASTM standards section 5.01.

SCOPE: This test method covers the determination of the kinematic viscosity of liquid petroleum products both transparent and opaque by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer.

DEFINITONS: Kinematic viscosity -- a measure of the resistive flow of a fluid under gravity, the pressure head being proportional to the density, ρ, of the fluid: for gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density,ρ. For any particulear viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, $\nu = \eta/\rho$, where $\eta$ is the dynamic viscosity coefficient.

PROCEDURE: follow general procedure sections 8 and 9.

CALCULATION/RESULTS:

kinematic viscosity = cSt = Ct

where:

C = calibration constant
t = flow time

NOTES:

1. Use ASTM thermometers for viscometer baths (table 2). Maintain temperature +/- 0.02 F.
2. Use viscometers as described (table 1)
3. Between determinations thoroughly clean viscometers with appropriate solvents, and periodically clean with chromic acid. (section 12)
4. Viscosity standards: used as confirmatory checks on the procedure in the laboratory, and for redetermination of calibration constant.(ASTM D446).
5. Recalibration: based on use. Recalibrate every 10 runs or when standard varies by more than +/- 0.35%.
6. Run duplicate tests on 10% of samples.

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Designation: D 445 - 88

An American National Stand
British Standard

Designation: 71/84

# Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[1]

This standard is issued under the fixed designation D 445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval. This is also a standard of the Institute of Petroleum issued under the fixed designation IP 71. The final number indicates the year of last revision.

*This test method has been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures.*

*This standard has been approved for use by agencies of the Department of Defense and replaces Method 305.6 of Federal Test Method Standard 791b. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.*

## 1. Scope

1.1 This test method covers the determination of the kinematic viscosity of liquid petroleum products (Note 1), both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid.

NOTE 1—*For the measurement of the viscosity of bitumens, see also* Test Method D 2170 and Test Method D 2171.

1.2 This test method is intended primarily for application to liquids for which the shear stress and shear rates are proportional.

1.2.1 This test method depends on the behavior of the sample, and ideally the coefficient of viscosity should be independent of the rate of shear (this is commonly called Newtonian flow behavior). If, however, the coefficient of viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of differe . capillary diameters.

1.3 This test method also includes the determination of the kinematic viscosity of fuel oils which often exhibit non-Newtonian properties.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 446 Specifications and Operating Instructions for Gl Capillary Kinematic Viscometers[2]

D 2170 Test Method for Kinematic Viscosity of Aspha (Bitumens)[3]

D 2171 Test Method for Viscosity of Asphalts by Vacuu Capillary Viscometer[3]

E 1 Specification for ASTM Thermometers[4]

E 77 Method for Inspection and Verification of Liqui in-Glass Thermometers[5]

## 3. Terminology

3.1 *Definitions:*

3.1.1 *kinematic viscosity*—a measure of the resistive fl of a fluid under gravity, the pressure head being proportio to the density, $\rho$, of the fluid: for gravity flow under a giv hydrostatic head, the pressure head of a liquid is propo tional to its density, $\rho$. For any particular viscometer, t time of flow of a fixed volume of fluid is directly propo tional to its kinematic viscosity, $\nu = \eta/\rho$, where $\eta$ is t dynamic viscosity coefficient. The kinematic viscosity coef cient has the dimension $L^2/T$, where $L$ is a length, and $T$ is time. The cgs unit of kinematic viscosity is one centimet squared per second and is called one stokes (symbol St). Tl SI unit of kinematic viscosity is one metre squared p second and is equivalent to $10^4$ St. Frequently, t centistokes (symbol cSt) is used (1 cSt = $10^{-2}$ St = 1 mm$^2$/s

3.1.2 *density*—the mass per unit volume of the fluid. Tl dimension of density is $M/L^3$, where $M$ is a mass. The c unit of density ($\rho$) is one gram per millilitre, and the SI u of density is one kilogram per cubic metre.

3.1.3 *dynamic viscosity* (coefficient of)—the ratio betwe the applied shear stress and rate of shear. This coefficient, is thus a measure of the resistance to flow of the fluid; it commonly called the viscosity of the liquid. The dimensio of the coefficient of dynamic viscosity is $M/LT = FT/L$

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 445 - 37 T. Last previous edition D 445 - 86[ε1].

In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 04.03.

[4] *Annual Book of ASTM Standards*, Vols 05.01 and 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.01.

TABLE 1 Viscometer Types

| Viscometer Identification | Range, cSt (mm²/s)[A] |
|---|---|
| A Gravity Types for Transparent Liquids: | |
| 1 Cannon-Fenske Routine[C] | 0.5[B] to 20 000 |
| 2 Zeitfuchs[C] | 0.8 to 5 000 |
| 3 SIL[C] | 0.8 to 10 000 |
| 4 Cannon-Manning Semi-micro[B] | 0.4 to 20 000 |
| 5 BS/IP U-tube[B] | 0.9[B] to 10 000 |
| 6 BS/IP U-tube Miniature[B] | 0.2 to 100 |
| 7 Ostwald | 0.8[B] to 17 000 |
| B Suspended-Level Types for Transparent Liquids: | |
| 1 Ubbelohde[C] | 0.3[B] to 100 000 |
| 2 Fitz-Simons[C] | 0.8 to 1 200 |
| 3 Atlantic[B] | 0.75[B] to 5 000 |
| 4 Cannon-Ubbelohde, Cannon-Ubbelohde Dilution[C] | 0.5[B] to 100 000 |
| 5 Cannon-Ubbelohde Semi-micro[C] | 0.4 to 20 000 |
| 6 BS/IP Suspended Level[B] | 3.5[B] to 100 000 |
| 7 BS/IP Suspended Level, Shortened Form[B] | 1.05[B] to 10 000 |
| 8 BS/IP Miniature Suspended Level[B] | 0.6 to 8 000 |
| C Reverse-Flow Types for Transparent and Opaque Liquids: | |
| 1 Zeitfuchs Cross-Arm[C] | 0.8 to 100 000 |
| 2 Cannon-Fenske Opaque[C] | 0.4 to 20 000 |
| 3 Lantz-Zeitfuchs[B] | 60 to 100 000 |
| 4 BS/IP U-tube Reverse Flow[B] | 0.6 to 300 000 |

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Table 4.

[B] In each of these series, the minimum flow time for the viscometers with the upper constant exceeds 200 s.

[C] Specifications and operating instructions for these viscometers have been examined in Specifications and Operating Instructions D 446.

[D] Specifications for these are given in Appendixes to IP 71.

depending on whether the dimension of viscosity is based on the $M$-$L$-$T$ system or the $F$-$L$-$T$ system (where $F$ represents a force). The cgs unit of dynamic viscosity is one gram per centimetre per second = one dyne-second per centimetre squared and is called one poise (symbol P). Frequently, the centipoise (symbol cP) is used (1 cP = $10^{-2}$ P). The SI unit of dynamic viscosity is one pascal-second; for convenience its submultiple of millipascal-second is frequently used (1 mPa·s = 1 cP).

NOTE 1—Dynamic viscosity also denotes a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence; it is hoped that this dual use of the same term will not be confusing.

## 4. Summary of Test Method

4.1 The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

## 5. Significance and Use

5.1 Many petroleum products, as well as nonpetroleum materials, are used as lubricants for bearings, gears, compressor cylinders, hydraulic equipment, etc. The proper operation of the equipment depends upon the proper kinematic viscosity or viscosity (sometimes called dynamic viscosity) of the liquid. Thus, the accurate measurement of kinematic viscosity and viscosity is essential to many product specifications.

5.2 The kinematic viscosity of many petroleum fuels is important for their proper use, for example, flow of fuels through pipe lines, injection nozzles and orifices, and the determination of the temperature range for proper operation of the fuel in burners.

## 6. Apparatus

6.1 *Viscometers* of the glass capillary type, calibrated and capable of measuring kinematic viscosity within the limits of precision given in Section 15 are acceptable. Viscometers listed in Table 1 meet these requirements.

6.1.1 Automated assemblies that measure kinematic viscosity within the limits of precision given in Section 15 are acceptable alternatives; kinematic viscosities less than 10 cSt (mm²/s) and flow times less than 200 s may require a kinetic energy correction (see Specification D 446).

6.2 *Viscometer Holders* to enable the viscometer to be suspended in a similar position as when calibrated. The proper alignment of vertical parts may be confirmed by using a plumb line.

6.3 *Viscometer Thermostat and Bath*—Any transparent liquid or vapor bath may be used, provided that it is of sufficient depth that at no time during the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 The temperature control must be such that for the range from 15 to 100°C (60 to 212°F) the temperature of the bath medium does not vary by more than 0.01°C (0.02°F) over the length of the viscometers, or between the position of each viscometer, or at the location of the thermometer. For

TABLE 2 Kinematic Viscosity Test Thermometers[A]

| Test Temperature[B] | Scale Error[B] | Thermometer Number | |
|---|---|---|---|
| °F | °C | ASTM[C] | IP[D] |
| −45 | −63.9 | 74F, C† | 90F, C |
| −60 to −65 | −51 to −55 | 42F | 62F, C |
| −46 | −40 | 73F, C | −66F, C |
| −15 | −26.1 | 126F, C | 71F, C |
| | −20 | 127C | 30C |
| 0 | −17.8 | 72F, C† | 67F, C |
| 32 | 0 | 129F, C | 39F, C |
| 68 and 70 | 20 and 21.1 | 44F, C | 23F, C |
| 77 | 25 | 40F, C | 30F, C |
| 86 | 30 | 118F, C | |
| 100 | 37.8 | 28F, C | 31F, C |
| | 40 | 120C | 32C |
| 122 | 50 | 46F, C | 68F, C |
| 130 | 54.4 | 29F, C† | 34F, C |
| 140 | 60 | 47F, C | 35F, C |
| | 80 | | 100C |
| 160 | 69.9 | 48F, C | 90F, C |
| 200 | 93.3 | 129F, C | 36F, C |
| 210 and 212 | 98.9 and 100 | 30F | 38F, C |
| | 100 | 121C | |
| 275 | 135 | 110C, C | |

[A] The smallest graduation of the Fahrenheit thermometers is 0.1°F and for the Celsius thermometers is 0.05°C except for ASTM 46F and IP 65F for which it is 0.2°F.

[B] Scale error for the Fahrenheit thermometers is not to exceed ±0.2°F (except for ASTM 110F which is ±0.5°F); for the Celsius thermometers it is ±0.1°C. These scale errors are required to apply only at the given test temperature.

[C] Complete construction detail is given in Specification E 1.

[D] Complete construction detail is given in Part I of IP Standards for Petroleum and Its Products.

† Stationary corrected.

TABLE 3 Values of the Several ASTM Viscosity Oil Standards

| Viscosity Standard Conforming to ASTM Standards[A] | Approximate Kinematic Viscosity, cSt (mm²/s) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | At −40°C (−40°F) | At 20°C (68°F) | At 25°C (77°F) | At[B] 37.78°C (100°F) | At 40°C (104°F) | At 50°C (122°F) | At[B] 98.89°C (210°F) | At 100°C (212°F) |
| S-3 | 10 | 4.6 | 4.0 | 3.0 | 2.9 | | 1.2 | 1.2 |
| S-6 | | 11 | 8.8 | 6.0 | 5.7 | | 1.8 | 1.8 |
| S-20 | | 44 | 34 | 20 | 18 | | 4.0 | 3.9 |
| S-60 | | 170 | 120 | 60 | 54 | | 7.4 | 7.2 |
| S-200 | | 840 | 400 | 200 | 180 | | 17 | 17 |
| S-600 | | 2400 | 1600 | 600 | 520 | 280 | 35 | 33 |
| S-2000 | | 8700 | 5500 | 2000 | 1700 | | 75 | 70 |
| S-6000 | | 37 000 | 25 000 | 6000 | 6700 | | | |
| S-30 000 | | | 81 000 | 27 000 | 22 000 | 11 300 | | |

[A] The actual values for the standards listed above are established and already reaffirmed by cooperative tests. In 1965 tests were made using 16 different types of viscometers in 26 laboratories located in 13 countries.

[B] Standardizations at 37.78°C and 98.89°C may be discontinued January 1, 1967.

temperatures outside this range, the variation must not exceed 0.03°C (0.05°F).

6.4 *Temperature-Measuring Device*—Standardized liquid-in-glass thermometers (Table 2) of an accuracy after correction of 0.02°C (0.04°F) can be used, or any other thermometric device of equal or better accuracy. If standardized liquid-in-glass thermometers are used, it is recommended (but not required) that two thermometers be used; they must agree within 0.04°C (0.07°F).

6.5 *Timing Device*—Any timing device may be used provided that the readings can be taken with a discrimination of 0.2 s or better, and that it has an accuracy within ±0.07 % when tested over intervals of 15 min.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

## 7. Reagents and Materials

7.1 *Appropriate Solvent*, completely miscible with the sample, for example, petroleum ether.

7.2 *Appropriate Volatile Solvent*, completely miscible with the solvent described in 7.1.

7.3 *Chromic Acid Solution* for cleaning glassware.

## 8. Calibration

8.1 *Viscometers*—Use only calibrated viscometers with constants measured and provided to the nearest 0.1 % of their value.

8.2 *Thermometers*—Routine liquid-in-glass thermometers should be checked to the nearest 0.01°C (0.02°F) by direct comparison with a suitable calibrated thermometer.

TABLE 4 Minimum Flow Times

NOTE—All sizes of all viscometers listed in Specification D 446 are designed for a flow-time in excess of 200 s, except as listed below. The minimum flow times for the six "BS/IP" viscometers listed in Table 1 are given in the appendices to IP71.

| Viscometer Identification | ASTM Size | Minimum Flow-Time, s |
|---|---|---|
| Cannon-Fenske routine | 25 | 250 |
| Ubbelohde | 0 | 200 |
| Atlantic | OC | 250 |
| Cannon-Ubbelohde, Cannon-Ubbelohde micro | 25 | 200 |

8.2.1 Kinematic viscosity test thermometers shall be standardized at total immersion which means immersion to the top of the mercury column, with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. Do not submerge the expansion bulb at the top of the thermometer.

8.2.2 It is essential that the ice point of standardized thermometers be determined periodically and that the official corrections be adjusted to conform to the change in ice point.

8.3 *Timers*—Standard time signals available in some countries may be used for checking accuracy of timing devices.

8.4 *Viscosity Standards*[A] (Table 3)—These may be used as confirmatory checks on the procedure in the laboratory. If the measured kinematic viscosity does not agree within ±0.35 % of the certified value, each step in the procedure should be rechecked, including thermometer and viscometer calibration, to locate the source of error. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a countervailancing combination of the possible sources of error.

8.4.1 *Viscosity Oil Standards*, ASTM, having the approximate kinematic viscosity shown in Table 3 are available. Certified kinematic viscosity values are compared by annual cooperative tests by a number of laboratories. The current values are supplied with each portion.

## 9. General Procedure for Kinematic Viscosity

9.1 The specific details of operation vary for the different types of viscometers listed in Table 1. The operating instructions for the different types of viscometers are given in Specification D 446.

9.2 Maintain the bath at the test temperature within the limits given in 6.3.1 taking account of the precautions given in Appendix X1 and of the correction supplied on the certificates of calibration.

9.2.1 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used. The thermometers should be held in an upright position under the same

[A] The ASTM Viscosity Oil Standards are available in 1-qt (0.47 L) containers. Purchase orders should be addressed to the Cannon Instrument Co., P.O. Box 16, State College, PA. 16801. Shipments will be made as specified or by best method.

TABLE 1 Viscometer Types

| Viscometer Identification | Range, cSt ($mm^2/s$)[A] |
|---|---|
| 1. Ostwald Types for Transparent Liquids: | |
| 1. Cannon Fenske Routine[C] | 0.5[B] to 20 000 |
| 2. Zeitfuchs[C] | 0.6 to 3 000 |
| 3. SIL[C] | 0.6 to 10 000 |
| 4. Cannon-Manning Semi-micro[C] | 0.4 to 20 000 |
| 5. BS/IP U-tube[D] | 0.9[B] to 10 000 |
| 6. BS/IP U-tube Miniature[D] | 0.2 to 100 |
| 7. Pinkevitch | 0.6[B] to 17 000 |
| 2. Suspended-Level Types for Transparent Liquids: | |
| 1. Ubbelohde[C] | 0.3[B] to 100 000 |
| 2. Fitz-Simons[C] | 0.6 to 1 200 |
| 3. Atlantic[C] | 0.75[B] to 5 000 |
| 4. Cannon-Ubbelohde, Cannon-Ubbelohde Dilution[C] | 0.6[B] to 100 000 |
| 5. Cannon-Ubbelohde Semi-micro[C] | 0.4 to 20 000 |
| 6. BS/IP Suspended Level[D] | 3.5[B] to 100 000 |
| 7. BS/IP Suspended Level, Shortened Form[D] | 1.05[B] to 10 000 |
| 8. BS/IP Miniature Suspended Level[D] | 0.6 to 3 000 |
| 3. Reverse-Flow Types for Transparent and Opaque Liquids: | |
| 1. Zeitfuchs Cross-Arm[C] | 0.6 to 100 000 |
| 2. Cannon-Fenske Opaque[C] | 0.4 to 20 000 |
| 3. Lantz-Zeitfuchs[C] | 60 to 100 000 |
| 4. BS/IP U-tube Reverse Flow[D] | 0.6 to 300 000 |

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Table 4.

[B] In each of these series, the minimum flow time for the viscometers with the lowest constant exceeds 200 s.

[C] Specifications and operating instructions for these viscometers have been assembled in Specifications and Operating Instructions D 446.

[D] Specifications for these are given in Appendices to IP 71.

depending on whether the dimension of viscosity is based on the $M$-$L$-$T$ system or the $F$-$L$-$T$ system (where $F$ represents a force). The cgs unit of dynamic viscosity is one gram per centimetre per second = one dyne-second per centimetre squared and is called one poise (symbol P). Frequently, the centipoise (symbol cP) is used (1 cP = $10^{-2}$ P). The SI unit of dynamic viscosity is one pascal-second; for convenience its submultiple of millipascal-second is frequently used (1 mPa·s = 1 cP).

NOTE 2—Dynamic viscosity also denotes a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence; it is hoped that this dual use of the same term will not be confusing.

## 4. Summary of Test Method

4.1 The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

## 5. Significance and Use

5.1 Many petroleum products, as well as nonpetroleum materials, are used as lubricants for bearings, gears, compressor cylinders, hydraulic equipment, etc. The proper operation of the equipment depends upon the proper kinematic viscosity or viscosity (sometimes called dynamic viscosity) of the liquid. Thus, the accurate measurement of kinematic viscosity and viscosity is essential to many product specifications.

5.2 The kinematic viscosity of many petroleum fuels is important for their proper use, for example, flow of fuels through pipe lines, injection nozzles and orifices, and the determination of the temperature range for proper operation of the fuel in burners.

## 6. Apparatus

6.1 *Viscometers* of the glass capillary type, calibrated and capable of measuring kinematic viscosity within the limits of precision given in Section 15 are acceptable. Viscometers listed in Table 1 meet these requirements.

6.1.1 Automated assemblies that measure kinematic viscosity within the limits of precision given in Section 15 are acceptable alternatives; kinematic viscosities less than 10 cSt ($mm^2/s$) and flow times less than 200 s may require a kinetic energy correction (see Specification D 446).

6.2 *Viscometer Holders* to enable the viscometer to be suspended in a similar position as when calibrated. The proper alignment of vertical parts may be confirmed by using a plumb line.

6.3 *Viscometer Thermostat and Bath*—Any transparent liquid or vapor bath may be used, provided that it is of sufficient depth that at no time during the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 The temperature control must be such that for the range from 15 to 100°C (60 to 212°F) the temperature of the bath medium does not vary by more than 0.01°C (0.02°F) over the length of the viscometers, or between the position of each viscometer, or at the location of the thermometer. For

TABLE 2 Kinematic Viscosity Test Thermometers[A]

| Test Temperature[B] | | Scale Error[B] | Thermometer Number | |
|---|---|---|---|---|
| °F | °C | | ASTM[C] | IP[D] |
| −55 | −53.9 | | 74F, C† | 69F, C |
| −60 to −35 | −51 to −35 | | 43F | 65F, C |
| −40 | −40 | | 73F, C | 68F, C |
| −15 | −26.1 | | 128F, C | 71F, C |
| | −20 | | 127C | 99C |
| 0 | −17.8 | | 72F, C† | 67F, C |
| 32 | 0 | | 121F, C | 33F, C |
| 68 and 70 | 20 and 21.1 | | 44F, C | 29F, C |
| 77 | 25 | | 45F, C | 30F, C |
| 86 | 30 | | 116F, C | |
| 100 | 37.8 | | 21F, C | 31F, C |
| | 40 | | 120C | 92C |
| 122 | 50 | | 44F, C | 66F, C |
| 130 | 54.4 | | 28F, C† | 34F, C |
| 140 | 60 | | 47F, C | 35F, C |
| | 80 | | | 100C |
| 180 | 82.2 | | 48F, C | 90F, C |
| 200 | 93.3 | | 122F, C | 36F, C |
| 210 and 212 | 98.9 and 100 | | 30F | 32F, C |
| | 100 | | 121C | |
| 276 | 135 | | 110F, C | |

[A] The smallest graduation of the Fahrenheit thermometers is 0.1°F and for the Celsius thermometers is 0.05°C except for ASTM 43F and IP 65F for which it is 0.2°F.

[B] Scale error for the Fahrenheit thermometers is not to exceed ±0.2°F (except for ASTM 110F which is ±0.3°F); for the Celsius thermometers it is ±0.1°C. These scale errors are required to apply only at the given test temperature.

[C] Complete construction detail is given in Specification E 1.

[D] Complete construction detail is given in Part I of IP Standards for Petroleum and its Products.

† Editorially corrected.

TABLE 3 Values of the Several ASTM Viscosity Oil Standards

| Viscosity Standard Conforming to ASTM Standards[A] | Approximate Kinematic Viscosity, cSt (mm²/s) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | At −40°C (−40°F) | At 20°C (68°F) | At 25°C (77°F) | At[B] 37.78°C (100°F) | At 40°C (104°F) | At 50°C (122°F) | At[B] 98.89°C (210°F) | At 100°C (212°F) |
| S-3 | 30 | 4.6 | 4.0 | 3.0 | 2.9 | | 1.2 | 1.2 |
| S-6 | | 11 | 8.9 | 6.0 | 5.7 | | 1.8 | 1.8 |
| S-20 | | 44 | 34 | 20 | 18 | | 4.0 | 3.9 |
| S-60 | | 170 | 120 | 60 | 54 | | 7.4 | 7.2 |
| S-200 | | 640 | 450 | 200 | 180 | | 17 | 17 |
| S-600 | | 2400 | 1600 | 600 | 520 | 280 | 33 | 32 |
| S-2000 | | 8700 | 5600 | 2000 | 1700 | | 78 | 75 |
| S-8000 | | 37 000 | 23 000 | 8000 | 6700 | | | |
| S-30 000 | | | 81 000 | 27 000 | 23 000 | 11 000 | | |

[A] The actual values for the standards listed above are established and annually reaffirmed by cooperative tests. In 1985 tests were made using 16 different types of viscometers in 28 laboratories located in 13 countries.

[B] Standardizations at 37.78°C and 98.89°C may be discontinued January 1, 1987.

temperatures outside this range, the variation must not exceed 0.03°C (0.05°F).

6.4 *Temperature-Measuring Device*—Standardized liquid-in-glass thermometers (Table 2) of an accuracy after correction of 0.02°C (0.04°F) can be used, or any other thermometric device of equal or better accuracy. If standardized liquid-in-glass thermometers are used, it is recommended (but not required) that two thermometers be used; they must agree within 0.04°C (0.07°F).

6.5 *Timing Device*—Any timing device may be used provided that the readings can be taken with a discrimination of 0.2 s or better, and that it has an accuracy within ±0.07 % when tested over intervals of 15 min.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

## 7. Reagents and Materials

7.1 *Appropriate Solvent*, completely miscible with the sample, for example, petroleum ether.

7.2 *Appropriate Volatile Solvent*, completely miscible with the solvent described in 7.1.

7.3 *Chromic Acid Solution* for cleaning glassware.

## 8. Calibration

8.1 *Viscometers*—Use only calibrated viscometers with constants measured and provided to the nearest 0.1 % of their value.

8.2 *Thermometers*—Routine liquid-in-glass thermometers should be checked to the nearest 0.01°C (0.02°F) by direct comparison with a suitable calibrated thermometer.

TABLE 4 Minimum Flow Times

NOTE—All sizes of all viscometers listed in Specification D 446 are designed for a flow-time in excess of 200 s, except as listed below. The minimum flow times for the six "BS/IP" viscometers listed in Table 1 are given in the appendices to IP71.

| Viscometer Identification | ASTM Size | Minimum Flow-Time, s |
|---|---|---|
| Cannon-Fenske routine | 25 | 250 |
| Ubbelohde | 0 | 300 |
| Atlantic | 0C | 250 |
| Cannon-Ubbelohde, Cannon-Ubbelohde dilution | 25 | 250 |

8.2.1 Kinematic viscosity test thermometers shall be standardized at total immersion which means immersion to the top of the mercury column, with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. Do not submerge the expansion bulb at the top of the thermometer.

8.2.2 It is essential that the ice point of standardized thermometers be determined periodically and that the official corrections be adjusted to conform to the change in ice point.

8.3 *Timers*—Standard time signals available in some countries may be used for checking accuracy of timing devices.

8.4 *Viscosity Standards*[6] (Table 3)—These may be used as confirmatory checks on the procedure in the laboratory. If the measured kinematic viscosity does not agree within ±0.35 % of the certified value, each step in the procedure should be rechecked, including thermometer and viscometer calibration, to locate the source of error. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

8.4.1 *Viscosity Oil Standards*, ASTM, having the approximate kinematic viscosity shown in Table 3 are available.[6] Certified kinematic viscosity values are compared by annual cooperative tests by a number of laboratories. The current values are supplied with each portion.

## 9. General Procedure for Kinematic Viscosity

9.1 The specific details of operation vary for the different types of viscometers listed in Table 1. The operating instructions for the different types of viscometers are given in Specification D 446.

9.2 Maintain the bath at the test temperature within the limits given in 6.3.1 taking account of the precautions given in Appendix X1 and of the correction supplied on the certificates of calibration.

9.2.1 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used. The thermometers should be held in an upright position under the same

[6] The ASTM Viscosity Oil Standards are available in 1-pt (0.47 L) containers. Purchase orders should be addressed to the Cannon Instrument Co., P.O. Box 16, State College, PA. 16804. Shipment will be made as specified or by best means.

conditions of immersion as when calibrated. They should be viewed with a lens assembly giving about five times magnification and which should be arranged to eliminate parallax errors.

9.3 Select a clean dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time should not be less than 200 s, or as noted in Table 4.

9.3.1 When the test temperature is below the dew point, affix loosely packed drying tubes to the open ends of the viscometer. The drying tubes must fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument. Carefully flush the moist room air from the viscometer by applying vacuum to one of the drying tubes. Finally, before placing the viscometer in the bath, draw up the sample into the working capillary and timing bulb and allow to drain back as an additional safeguard against moisture condensing or freezing on the walls.

9.3.2 Viscometers used for silicone fluids, fluoro-carbons, and other liquids which are difficult to remove by the use of a cleaning agent, should be reserved for the exclusive use of those fluids except when calibrating. Such viscometers should be subjected to calibration checks at frequent intervals. The solvent washings from these viscometers should not be used for the cleaning of other viscometers.

## 10. Procedure for Transparent Liquids

10.1 In general, the viscometers used for transparent liquids are of the type listed in Table 1, A and B.

10.2 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample contains solid particles, filter during charging through a 200 mesh (75-μm) filter.

10.2.1 With certain products that exhibit "gel-like" behavior, take care that measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar results will be obtained in viscometers of different capillary diameters.

10.2.2 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because the time varies for the different instruments and temperatures, establish a safe equilibrium time by trial (30 min should be sufficient). One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

10.2.3 Where the design of the viscometer requires it, adjust the volume of the sample to the mark.

10.3 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 5 mm ahead of the first timing mark. With the sample flowing freely, measure, in seconds to within 0.2 s (see 6.5), the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum (see 9.3) select a viscometer with a capillary of smaller diameter and repeat the operation.

10.3.1 Repeat the procedure described in 10.3 to make a second measurement of flow time.

10.3.2 If two measurements agree within 0.2 %, use the average for calculating the kinematic viscosity to be reported. If the measurements do not agree, repeat the determination after thorough cleaning and drying of the viscometers and filtering of the sample.

## 11. Procedure for Opaque Liquids

11.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table 1, C.

11.2 For steam refined cylinder oils and black lubricating oils, proceed to 11.4 ensuring a thoroughly representative sample is used. The viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure shall be followed to minimize this.

11.2.1 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

11.2.2 Thoroughly stir the sample with a nonmetallic rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

11.2.3 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

NOTE 3—With samples of a very waxy nature or oils of high viscosity, it may be necessary to increase the heating temperature to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

11.3 Immediately after completing 11.2.3, pour a sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

11.3.1 Immerse the flask in a bath of boiling water for 30 min.

NOTE 4: Caution—Vigorous boil-over may occur when opaque liquids that contain high levels of water are heated to high temperatures.

11.3.2 Remove the flask from the water, stopper tightly and shake for 1 min.

11.4 Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 200 mesh (75-μm) filter into two viscometers previously placed in the bath. For samples subjected to heat treatment, use preheated filter to prevent the sample coagulating during the filtration. Viscometers that are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample to ensure the sample will not be cooled below test temperature.

11.4.1 After 10 min, adjust the volume of the sample to coincide with the filling marks as in the viscometer specification. For example, for the cross-arm viscometer for opaque liquids, this is described in the annex of Specification D 446. The Cannon-Fenske Opaque Viscometer design does not require further adjustment of sample volume. Allow the viscometers to reach test temperature. Because this time will vary for the different instruments and for different test temperatures, establish a safe equilibrium time by trial (30 min is normally sufficient). One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

11.5 With the sample flowing freely, measure in seconds to within 0.2 s (see 6.5), the time required for the advancing ring of contact to pass from the first timing mark to the second.

11.5.1 In the case of samples requiring heat treatment described in 11.2 through 11.3.2, complete the determinations within 1 h of completing 11.3.2.

11.6 Calculate the mean kinematic viscosity $\nu$ in centistokes (millimetres squared per second) from the two determinations. For fuel oils, the two determinations should not differ by more than 1.7 % of $\nu$ at 50°C and 1.1 % of ($\nu$ + 8) at 80 and 100°C. If the two determinations exceed these limits, repeat the operation from 11.2.1. For other opaque liquids which may be non-Newtonian, the precision has not been determined.

11.6.1 Report the mean of the two determinations as the kinematic viscosity.

## 12. Cleaning of Viscometer

12.1 Between successive determinations, clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

12.2 Periodically clean the viscometer with chromic acid cleaning solution for at least twelve hours to remove residual traces of organic deposits; nonchromium-containing, strongly-oxidizing acid cleaning solutions[7] may be substituted so as to avoid disposal problems of chromium-containing solutions. Rinse thoroughly with distilled water followed by acetone, and dry with clean, dry air. Inorganic deposits may be removed by hydrochloric acid treatment before use of cleaning acid, particularly if barium salts are suspected. The use of alkaline cleaning solutions is not recommended as this can enlarge the working capillary and necessitate recalibration.

## 13. Procedure for Dynamic Viscosity

13.1 Determine the kinematic viscosity as described in Section 10 or 11.

13.2 Determine the density of the sample, to the nearest 0.001 g/mL at the same temperature as the viscosity, in accordance with any applicable method.

## 14. Calculation and Report

14.1 Calculate the kinematic viscosity, $\nu$, from the measured flow time, $t$, and the instrument constant, $C$, by means of the following equation:

$$\nu = Ct$$

where:

$\nu$ = kinematic viscosity, cSt (mm²/s)
$C$ = calibration constant of the viscometer, cSt/s, and
$t$ = flow time, s.

14.2 Calculate the viscosity, $\eta$, from the calculated kinematic viscosity, $\nu$, and the density, $\rho$, by means of the following equation:

$$\eta = \rho\nu$$

where:

$\eta$ = dynamic viscosity, cP (mPa·s)
$\rho$ = density, g/mL, at the same temperature used in measuring the flow time $t$, and
$\nu$ = kinematic viscosity, cSt (mm²/s)

14.3 Report test results for both the kinematic and dynamic viscosity rounded to the nearest one part in a thousand of the value measured or calculated, respectively.

## 15. Precision and Bias

15.1 The precision of this test method as obtained by statistical examination of interlaboratory test results of clean, transparent oils tested over the range from 15 to 100°C (59 to 212°F) using the procedure described in Section 10 is as follows:

15.1.1 *Repeatability*—The difference between successive test results, obtained by the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 0.35 % of their mean only in one case in twenty. Differences greater than this should be considered suspect.

15.1.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in normal and correct operation of this test method, exceed 0.70 % of their mean only in one case in twenty. Differences greater than this should be considered suspect.

15.1.3 The precision data[8] in 15.1.1 and 15.1.2 were obtained using five mineral oils covering the kinematic viscosity range from 3 to 1200 cSt (mm²/s) at temperatures from 38 and 99°C.

15.2 The precision[9] of this test method as obtained by statistical examination of interlaboratory test results for residual fuel oils of 30 to 1300 cSt (mm²/s) at 50°C, and of 8 to 170 cSt (mm²/s) at 80 and 100°C, using the procedure described in Section 11 is as follows:

15.2.1 *Repeatability*—The difference between successive test results, obtained by the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 1.5 % of their mean for results at 50°C and 1.3 % of their mean plus 8 cSt (mm²/s) [that is, 1.3 % of (mean + 8 cSt (mm²/s))] for results at 80 and 100°C only in one case in twenty. Differences greater than this should be considered suspect.

15.2.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in normal and correct operation of the test method, exceed 7.4 % of their mean for results at 50°C and 4.0 % of their mean plus 8 cSt (mm²/s) [that is, 4.0 % of (mean + 8 cSt (mm²/s))] for results at 80 and 100°C only in one case in twenty. Differences greater

[7] A commercial source for a non-chromium containing cleaning solution is Codax Laboratories Inc., 480 Canal Street, New York, NY 10013.

[8] Supporting data are available from ASTM Headquarters. Request RR:D02-1132.

[9] Supporting data are available from ASTM Headquarters. Request RR:D02-1198.

than this should be considered suspect.

15.3 *Bias*—No justifiable statement can be made on the bias of the procedure in Test Method D 445 for measuring kinematic viscosity because all determinations are relative to a calibration fluid.

## APPENDIX

### (Nonmandatory Information)

### X1. ICE POINT DETERMINATION

X1.1 To achieve an accuracy of ±0.02°C for calibrated kinematic viscosity thermometers, it is required that a check at the ice point be made. It is recommended that the interval of checking be every six months; for a new thermometer, check monthly for the first six months.

X1.2 A detailed procedure for the measurement of the ice point is described in Method E 77. The suggestions in the following sections of this appendix are given specifically for the mercury-in-glass "kinematic viscosity" thermometers described in Table 2, and may not apply to other thermometers.

X1.2.1 The ice point reading of kinematic viscosity thermometers shall be taken 5 min after being at test temperature for not less than 3 min. The ice point reading shall be expressed to the nearest 0.01°C or 0.02°F.

X1.2.2 Select clear pieces of ice, preferably made from pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the crushed ice and add sufficient distilled and preferably precooled water to form a slush, but not enough to float the ice. Insert the thermometer packing the ice gently about the stem, to a depth sufficient to cover the 0°C (32°F) graduation. As the ice melts, drain off some of the water and add more crushed ice.

X1.2.3 Raise the thermometer a few millimetres after at least 3 min have elapsed, tap the stem gently, and observe the reading. Successive readings taken at least 1 min apart should agree within one tenth of a division.

X1.2.4 Alternatively, some of the ice may be heaped around the stem above the ice point and a deep narrow channel formed to permit observation of the meniscus which is thus kept well below the general level of the ice. Observations may then be made as described above without, however, raising the thermometer.

X1.2.5 Record the readings and compare with previous readings. If the readings are found to be higher or lower than the reading corresponding to a previous calibration, readings at all other temperatures will be correspondingly increased or decreased.

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# APPENDIX B

## GENERAL STANDARD OPERATING PROCEDURE FOR SKINNER AND SHERMAN LABORATORY

# INTERNAL QUALITY CONTROL

## 13.1 PERFORMANCE CHECKS OF RADIATION SCREENING EQUIPMENT

Performance checks are made to assure the continuing capability of the screening instruments. Procedures include efficiency checks, and background determinations. The procedure and frequency of each check is optimized for each detector system to provide assurance of the detector's performance. Documentation of the checks and the results are kept for all procedures. The Radiation Safety Officer is responsible for all performance checks. Detailed procedures and schedules are outlined in the "Radiation Protection Plan" of the NRC license application.

## 13.2 ENVIRONMENTAL LABORATORY SERVICES

### 13.2.1 INTERNAL QUALITY CONTROL CHECKS

The Supervisors or Program Data Managers are responsible to ensure that analytical results are produced internally within acceptable limits.

#### 13.2.1.1 PRECISION

Replicate standards and/or samples are used to estimate the precision of each analytical test procedure for a known matrix. Data control limits are established to satisfy the requirements of specific measurement projects, based on prior knowledge of the measurement system and method validation studies or state certification limits.

#### 13.2.1.2 ACCURACY

Certified standards and/or spiked samples are used to estimate analyte recovery for each test procedure for a known matrix.

13.2.1.3 DUPLICATE AND SPIKES

Duplicate sample and/or matrix spike and matrix spike duplicate samples are analyzed with each set of samples, every 20 samples, 5 percent, or every 10 samples, whichever is appropriate.

13.2.1.4 COMPLETENESS

Data control limits are based on the mean precision and accuracy values determined in the laboratories or based on EPA Contract Laboratory Protocol requirements. When practical, acceptance limits are set at the mean values plus or minus three standard deviations. Samples outside the control limits are reanalyzed during a successive testing period or flagged where appropriate.

13.2.1.5 REPRESENTATIVENESS

Whenever possible, samples are collected and aliquotted so that the analysis results are representative of the media and conditions being measured.

13.2.1.6 COMPARABILITY

Unless otherwise specified, all data are calculated and reported in units consistent with other organizations reporting similar data to allow comparison of data bases.

**Table 13.1 PERFORMANCE OBJECTIVES FOR GENERAL QC PARAMETERS**

| QC PARAMETER | PERFORMANCE OBJECTIVES |
|---|---|
| **Laboratory and Trip Blanks.** Detect contamination during shipping, handling and preparation. | Results less than contract required detection limit. Once per batch per matrix and concentration level (high, medium, low). |
| **Initial and Continuing Calibration Blanks.** Determine background levels. | Results less than contract or client required detection limit. Every 10 samples for inorganics and wet chemistries or every 12 hours for organics. |
| **Spike Samples and Duplicate Samples.** Samples spiked during preparation that determine accuracy, precision and detect matrix effects. | If unspecified, recoveries should be 75-125% of the theoretical value. Once per batch per matrix per concentration level. |
| **Duplicate Samples.** Preparation and analysis performed to determine precision. | Relative percent difference should be less than 20% unless otherwise specified. Once per batch per matrix per concentration level. |
| **Initial Calibration Verifications.** Independent source standard analyzed to validate instrument calibration. | Recoveries between 90-110%. May also be analyzed at the end of sample analysis run. |
| **Continuing Calibration Verifications.** Confirm the calibration throughout the sample analyses. | Recoveries between 90-110%. If unspecified, every 10 samples for inorganic and wet chemistries, and every 12 hours for organics. |
| **Surrogate Standards and Internal Standards.** Estimate recoveries and account for sample variations and matrix effects. | As specified by contract, client or Standard Operating Procedure. |
| **External/Blind Performance Evalution Samples.** Assure laboratory wide compliance with QC objectives. | Recoveries within published acceptance levels. Performed twice per test method per year. |
| **Laboratory Control Standards and Method Spikes.** Determine method interferences and accuracy. | Recoveries between 75-125% of theoretical. Once per batch unless otherwise specified. |

13.2.3 SAMPLE CUSTODY

All samples are assigned a unique internal laboratory identification number, marked directly on the container to denote customer number and sample number. The laboratory shall designate a Sample Custodian and a back-up Sample Custodian for chain of custody samples. Lockable refrigerators, freezers, and cabinets are available for controlled samples. Internal sample custody forms are used for tracking all these samples through the analytical process.

13.2.4 STANDARD PREPARATIONS

All standards are prepared from certified solutions or reagent materials that are checked against certified standards. A complete record, including preparation date, individual preparing solutions, and the person who prepared the standard is maintained in a standards preparation logbook. Laboratory personnel are instructed to label standard's container.

13.2.5 COLLABORATIVE TESTING

In addition to the internal QC program, the laboratory participates in collaborative testing or interlaboratory comparison programs. This includes participation in numerous round robin, interlaboratory studies, and Quality Control testing programs. At a minimum, the laboratory actively participates in the U.S. EPA InterLaboratory Quality Control Program for Water Supply/Water Pollution.

## 13.3 DATA VERIFICATION

Data reliability is based on method specific quality control limits worked into data processing systems at the bench level. Calculated precision and accuracy control limits are used to flag outliers. Overall data compatibility is performed by checking for proper reporting units and minimum and maximum reporting ranges. The laboratory Supervisor reviews and initials the input data. All raw data, including the final results, are stored for later review. Calculations performed manually are reviewed by a second person and initialed as checked.

### 13.3.1 ELECTRONIC DELIVERABLES VERIFICATION

Supervisors and/or Program Managers are responsible for assuring that electronic deliverables accurately reflect the hardcopy deliverables.

## 13.4 DATA SECURITY

Computer Software Systems used in Data Handling and information and reporting use password-based security levels for all who have access to the computer network and data base. Data and information is archived on a daily and weekly basis.

## 13.5 TECHNICAL COMPLAINTS

Technical Complaints are addressed by the Program Manager or staff member with the most expertise in the field of complaint with support from the Technical Director. Upon receipt of the technical complaint, the Program Manager assigns the data inquiry a number and initiates a Data Inquiry Form. The form will contain the work order number, client, and concern. The Supervisor will receive this form attached to the work order file for review. The Supervisor will then determine if the complaint is valid, fill out the Data Inquiry Form and return it to the Program Manager for client call back. If the complaint is determined to be valid, then the cause for the complaint will be corrected as soon as feasible. A copy of the Data Inquiry Form will be stored in the case file. The original will be maintained by the QA Officer.

## PREVENTIVE MAINTENANCE AND INSTRUMENTATION REDUNDANCY

### 9.1 POLICY

Preventive maintenance is performed as required on analytical instrumentation to prevent down time of the measuring systems.

### 9.2 MAINTENANCE

Maintenance procedures are developed by the Supervisors for use where procedures are not provided in the manufacturer supplied operator's manual. Records of instrument set-up, maintenance, and repair are maintained. The Supervisors and operating personnel are responsible for complying with the instrument maintenance schedule.

### 9.3 SPARE PARTS

Supervisors and operators are responsible to assure that an adequate inventory of spare parts and consumables are requisitioned and maintained for the instrumentation in their area(s) in order to prevent downtime or compromised running conditions.

### 9.4 INSTRUMENTATION REDUNDANCY

Back-up instrumentation, autosamplers, and computer software systems used to perform routine processes are maintained where necessary.

Redundancy exists for the following instrumentation and equipment: Laboratory Information Management System (LIMS) Database and hardware, LIMS Network Fileserver and Hardware, Gas Chromatography-Mass Spectrometry, Gas Chromatography, High Performance Liquid Chromatography, Wet Chemistry Autoanalyzer, Inductively Coupled Argon Plasma Spectrophotometer, Graphite Furnace Atomic Absorption Spectrophotometer, Cold-Vapour Mercury Analyzer, Ion Selective Electrodes, Refrigerators, Ovens, and Distillation, Digestion and Extraction equipment.

# CORRECTIVE ACTION

## 16.1 POLICY

In order to maintain and assure acceptable quality levels for services provided by TMA/Skinner & Sherman, a feed-back system shall be established to assure that conditions adverse to quality shall be promptly identified and corrected. Corrective actions shall be determined and initiated as indicated below.

## 16.2 CORRECTIVE ACTION

16.2.1 The QA Officer shall initiate investigation and corrective action by using a Corrective Action Request Form in any of the following situations:

16.2.1.1 When an audit reveals circumstances that may adversely affect quality as determined by the QA Officer.

16.2.1.2 When the results of an intercomparison study program are out of control.

16.2.1.3 When procedural or technical problems arise and the QA Officer or supervisors determine that they may significantly affect quality.

## 16.3 RESPONSIBILITY

All personnel are responsible to communicate any evidence of unacceptable quality performance to the Supervisor and/or the QA Officer, or laboratory management.

16.3.1 The Supervisor or designee shall be responsible for investigating conditions adverse to quality, determining the assignable cause and recommending the actions necessary for their correction.

16.3.2 The General Manager or designee shall provide pertinent information regarding cause of adverse conditions and then determine the specific corrective action necessary to preclude recurrence.

16.3.3 The QA Officer shall review the Corrective Action trends and other routine Quality Control data for evidence of unacceptable quality.

16.3.4 All items requiring corrective action shall be clearly identified in the Corrective Action Request Form for subsequent follow-up and close-out actions. Completed copies of the Corrective Action Request Form shall be kept by the QA Officer.

"Internal Sample and Extract Custody"
TMA/Skinner & Sherman Laboratories, Inc.

Approval: Joan Lacava — Joan Lacava, General Manager
Lisa Snow — Lisa Snow, QA Officer

==========

## 1. Scope and Applicability:

This SOP describes practices employed by TMA/Skinner & Sherman concerning the procedures and the documentation of sample and extract transfers for controlled samples. The procedure starts upon transmittal of the Internal (SAM) Chain of Custody and ends with sample purge.

## 2. Reference:

2.1 USEPA CONTRACT LABORATORY PROGRAM STATEMENT OF WORK, Document Number ILM01.0 (SOW 3/90).

2.2 TMA/Skinner & Sherman Quality Assurance Manual

## 3. Procedures:

3.1 Internal Storage

3.1.1 The sample custodian checks that each Internal Chain of Custody has the appropriate storage locations (Section 3.1.1) and sample identifications.

3.1.2 The corresponding Extract, Distillate, Digestate Chain of Custodies (COC's) are delivered to or posted at the area where the samples are originally stored or where the extracts, digestates or distillates are originated by the sample custodian.

3.1.3 The raw sample Internal COC's remain at their original post until the whole batch of samples have been analyzed and are relocated by the sample custodian, then the COC is moved into a COC folder maintained in sample control with the documented move.

3.1.4 For EPA/CLP samples copies of the Internal COC's are included in the data package. The Sample Custodian copies the Internal COC's and includes the copy(s) in the case file folder.

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3.1.5 When the samples are moved for disposal, this is documented on the Internal COC which is then archived in the library. Raw sample disposal is documented on the raw sample COC and the Extract, Distillate, and Digestate sample disposal is documented on their COC(s).

3.1.6 Raw Sample storage is maintained in-house for a minimum of 30 days for commercial and 60 days for CLP or CLP-type projects after reporting the data.

3.1.7 EPA/CLP organic sample splits are refrigerated for 60 days after reporting and commercial sample splits are refrigerated for at least 30 days after reporting.

3.1.8 Samples are Removed and returned in batches are documented on an internal custody form. Only one sample delivery group or batch is entered per internal custody form.

3.1.9 The analyst who signs out for the sample(s) is responsible for the custody of the sample, and is responsible for returning the sample as soon as is practical after taking the aliquot(s).

3.2 Internal Chain of Custody Locations

3.2.1 CLP, CLP-TYPE Raw Inorganics Samples - For CLP raw Inorganics samples the <u>IP</u> internal COC is posted on the clipboard to the right of the Large (Arctic) Walk-in and is used for the <u>all</u> CLP Inorganic tests.

3.2.2 CLP, CLP-TYPE Digestates and Distillates - The <u>IC</u> internal COC is delivered to the Inorganic Prep Lab and used for internal transfers of the AA and ICP digestates. The WC internal COC is delivered to the Cyanide Distillation Lab and is used for the movement of CLP cyanide distillates.

3.2.3 Raw Commercial Organics and Inorganics - For Commercial samples that are stored in either the (REVCO) 2-Door refrigerator or the (VOLLRATH) small Walk-in, the internal COC is the overall one that has <u>no</u> <u>department code</u> specified. It is posted to the right of the REVCO 2-Door.

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3.2.4 Volatiles (VOA) Raw and Extract Samples - All VOA internal COC's are posted on the clipboard to the right of the Low-level VOA refrigerator.

3.2.5 PCB/Pesticide and Semi-Volatile (BNA) Extracts - The GC and GCMS internal chain of custodies are delivered to the Organic Prep lab to document the custody of the organic PCB/Pesticide and BNA extracts, respectively, for CLP and CLP-type protocols.

3.3 Sample and Container Integrity

Broken containers are discarded of appropriately. Samples that are received broken are brought to the attention of the client representative and the sample is transferred to a duplicate container, unless otherwise requested by the client. This is documented on the chain of custody. For EPA/CLP samples call SMO.

3.4 Internal Custody Form

The TMA/Skinner & Sherman internal custody form is the CHAIN OF CUSTODY (Fig. 1) generated in the transmittal of SAM paperwork.

When the Workorder paperwork is transmitted the following departmental CHAIN OF CUSTODY forms are posted at the appropriate stations as noted above.

Fill-in the RELEASED BY/TRANSFERRED TO/RECEIVED BY section as such:

| RELEASED BY | DATE | TRANSFERRED TO | DATE | RECEIVED BY | DATE |
|---|---|---|---|---|---|

The Sample Custodian uses the top set of lines to log the samples into the storage area or refrigerator --

The second line under RELEASED BY - the sample custodian's name (initials) with date and then the first prep chemist to remove the samples after log-in.

Samples are removed in batches with the container in which they are stored. Chemists remove return the whole bucket or box of sample for the test at hand.

"Internal Sample and Extract Custody"
TMA/Skinner & Sherman Laboratories, Inc.

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Fig. 1 CHAIN OF CUSTODY

| RELEASED BY | DATE | TRANSFERRED TO | DATE | RECEIVED BY | DATE |
|---|---|---|---|---|---|
| "Jeff DePaolo stored 6 soils/3 waters in WA on 3/21/91" | | | | | |
| JD | 3/21/91 | Prep Lab | 3/21/91 | HE | 3/21/91 |
| HE | 3/21/91 | Arctic Walk-in | 3/21/91 | JD | 3/21/91 |
| JD | 3/25/91 | Cyanide Lab | 3/25/91 | BB | 3/25/91 |
| BB | 3/25/91 | Arctic Walk-in | 3/25/91 | JD | 3/25/91 |
| JD | 3/28/91 | HG Lab | 3/28/91 | LD | 3/28/91 |
| LD | 3/28/91 | Arctic Walk-in | 3/28/91 | JD | 3/28/91 |
| JD | 4/25/91 | Cabinet 8 | 4/25/91 | JD | 4/25/91 |
| JD | 6/26/91 | Closet | 6/26/91 | JD | 6/26/91 |
| JD | 6/26/91 | Disposal | 6/26/91 | RL | 6/26/91 |

3.5 Storage System

3.5.1 The location of sample storage is typed into the space under "STORED" in the SAM log-in of each sample split. The sample custodian uses the storage abbreviations listed for the location where the samples will be stored after log-in, and during analysis.

STORAGE DESIGNATIONS

COMM - VOLLRATH WALK-IN (6'x 6 x 6')
CLP - ARCTIC WALK-IN (7'x 10'x 7.8')
2-Door - REVCO DOUBLE DOOR
VOA - EQUATHERM REFRIGERATOR #2, VOLATILES
524 - EQUATHERM REFRIGERATOR #4, LOW-LEVEL VOLATILES
ORGS#3 - WESTINGHOUSE
GC #1 - REFRIGERATOR #1 FOR GC SAMPLES
METLAB - DIGESTATES FOR TOTAL METALS
CNLAB - CYANIDE DISTILLATES
DOCK - LOADING DOCK CLOSET SHELVES

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3.5.2 Once analysis is complete for Inorganics raw samples any left-over sample is transferred by the sample custodian to the Office 10 cabinets as listed below. The sample organization for these cabinets is based sample types and volumes, however samples that arrived in the same months are typically stored together.

OFFICE 10 (CABINET ROOM):

C1 - LOCKABLE CABINET
C2 - " "
C3 - " "
C4 - " "
C5 - " "
C6 - " "
C7 - " "
C8 - " "
C9 - " "
C10 - BLACK CABINET NON-ROUTINE CABINETS

# APPENDIX C

# GENERAL STANDARD OPERATING PROCEDURES FOR ROCKY MOUNTAIN ANALYTICAL LABORATORY

Enseco

STANDARD OPERATING PROCEDURE

INTERNAL QC CHECKS

LABORATORY PERFORMANCE QC

SOP NO.: M-EQA-002

REVISION NO.: 2.0

2240 Dabney Road
Richmond, Virginia 23230
804/359-9900 Fax 804/353-1860

Enseco

***MEMORANDUM***

TO: QA Directors

FROM: Peggy Sleevi PSS

DATE: August 2, 1989

SUBJECT: M-EQA-002 Revision 2.0, Ammendment for Oil and Grease DCS

Based on the DCS data for the Oil and Grease Gravimetric method, submitted to the corporate QA group for review, the DCS component must be changed to Paraffin Oil (Baker B356 28 9388-01) to replace the Reference Oil cited on page A4-3 of SOP No. M-EQA-0002. The paraffin oil from Baker is currently in use at Cal and East. The Reference Oil cited in method 413.2 for O&G-IR (mixture of hexadecane, isooctane and chlorobenzene) will continue to be the required spiking component for the IR method. For clarity, a replacement page for Page A4-3 is attached. The other typographical errors that occurred on this page have also been addressed along with a clarification on the DCS for the aromatic method as requested by RMAL. A complete revision of this document will be addressed within the next quarter.

Please distribute this corrected page to all employees who have a copy of this document.

cc: Kathy Carlberg

Enseco Incorporated
2240 Dabney Road
Richmond, Virginia 23230
804/358-1900 Fax 804/353-1860

## Oil and Grease

| DCS | Method | QC Category Aqueous | QC Category Solid | Spike Level Aqueous (mg/L) | Spike Level Solid* (mg/Kg) |
|---|---|---|---|---|---|
| Control Mixture (1) | Aromatic | O&G-AR-A | O&G-AR-S | 50 | 1000 |
| Paraffin Oil | Gravimetric | O&G-G-A | O&G-G-S | 50 | 1000 |
| Reference Oil (2) | IR | O&G-IR-A | O&G-IR-S | 5+ | 50+ |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

(1) As specifed in LM-RMA-1044 and LM-RMA-1045
(2) As specified in Method 413.2, section 6.4.1

* Assumes 50 g sample (wet weight).

+ Spike level may be adjusted to account for variation in instrument sensitivity. Alternate spike level must be within 5 times the reporting limit.

x Revision of the DCS component for Aromatic and Gravimetric methods and correction of typographical error for solid QC categories made August 2, 1989 by P. Sleevi for immediate incorporation in SOP No. M-EQA-0002, Rev. 2.0.

Peggy Sleevi 5-2-89

Enseco

***MEMORANDUM***

TO: QA Directors

FROM: Peggy Sleevi

DATE: July 5, 1989

SUBJECT: Revision to M-EQA-002, Rev. 2.0 (Solid Matrix)

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SOP M-EQA-002, Rev 2.0, Internal QC Checks--Laboratory Performance QC, requires the use of Ottawa sand as the solid matrix for Duplicate Control Samples (DCS) and Single Control Samples (SCS). Technical problems with background levels of various metals and poor precision have been observed when using Ottawa Sand as the solid matrix in metals analyses. The EPA Solid Matrix, available from USEPA EMSL-Las Vegas, is to be substituted for Ottawa sand when the solid matrix is required for metals analyses.

We will continue to use Ottawa sand as the solid matrix for organic analyses.

A typographical error appears for the solid matrix QC category for the Oil and Grease gravimetric and IR methods. The correct QC categories for the solid matrix for these methods are O&G-G-S and O&G-IR-S, respectively.

Please inform laboratory staff of these changes and retain this memorandum as documentation of these changes to M-EQA-002, Rev. 2.0.

cc: Kathy Carlberg

Enseco Incorporated
2240 Dabney Road
Richmond, Virginia 23230
804/359-1900 Fax. 804/353-1860

STANDAR
OPERATI...
PROCEDURE

| Subject or Title: Internal QC Checks--Laboratory Performance QC | | Page ____ of __ |
|---|---|---|
| SOP No.: M-EQA-002 | Revision No.: 2.0 | Effective Date May 15, 1989 |

Supersedes: M-EQA-002, Revision No. 1, June 28, 1988

## 1.0 PURPOSE

1.1 To describe the essential elements of Enseco's Internal QC Program related to Laboratory Performance QC.

1.2 Laboratory Performance QC consists of internal QC check samples which are used to determine whether the laboratory is "in control" during data generation. These check samples, called Laboratory Control Samples (LCS), are generated using a standard control matrix and are used to measure laboratory performance independent of sample matrix effects.

1.3 There are three types of LCS. Duplicate Control Samples (DCS), Surrogate Control Samples (SCS), and Method Blanks.

1.4 Surrogates are added to samples and QC samples for some organic methods. The monitoring of surrogate recoveries in QC samples is an element of Laboratory Performance QC. However, the monitoring of surrogate recoveries in samples is not used to control laboratory operations and thus is not an element of Laboratory Performance Q Surrogate recoveries in samples are used to measure the effect of the matrix or method performance and are thus an element of Matrix Specific QC (See M-EQA-003).

| Prepared by: Peggy Sleevi | Date: May 15, 1989 |
|---|---|
| Management Approval: Peggy Sleevi for Kathy Lambert | Date: 5-19-89 |
| QA Approval: Peggy Sleevi | Date: 5-19-89 |

## 2.0 POLICIES

2.1 An essential element of Quality Control is to measure laboratory performance of a method. This is done using precision and accuracy data gathered from spiking a consistent, reproducible control matrix with a prescribed list of target analytes at known prescribed concentrations. A control matrix is used so that these measurements reflect laboratory performance and not matrix effects.

2.2 Quality control data, including recovery and method blank data, must be generated with every batch of samples processed.

2.3 QC information is generated and evaluated at the bench. The prime responsibility for implementation of the QC Program lies with the analyst. The QA Department serves in an audit function and addresses special QA needs such as QA Project Plans. QC information needed for standard reports is generated, documented, and entered into LIMS by the analyst.

## 3.0 SAFETY ISSUES

3.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.

3.2 A Material Safety Data Sheet (MSDS) is available for each laboratory standard and reagent chemical. The appropriate MSDS must be reviewed by the employee before handling the chemical(s).

3.3 All laboratory personnel should be thoroughly familiar with the laboratory Safety Manual before undertaking any laboratory work.

## 4.0 PROCEDURE

4.1 <u>Types of Laboratory Performance QC Samples</u>.

4.1.1 Duplicate Control Sample (DCS): A <u>DCS</u> consists of a standard control matrix which is spiked with a group of target compounds representative of the method analytes.

A DCS pair is analyzed for every 20 samples processed by a method. The DCS pair is used to monitor both the precision and accuracy of the analytical method on an ongoing basis, independent of matrix effects.

4.1.2 Single Control Sample (SCS): A SCS consists of a standard control matrix which is spiked with surrogate compounds appropriate to the method. The surrogates are spiked into the method blank, which serves as both the method blank and the SCS. In cases where no surrogate is available for a method e.g., metals analyses, the components of the SCS are ident to the DCS components and the SCS is analyzed in addition to the method blank.

A SCS is analyzed with each batch of samples processed to ensure that QC data are generated with every sample processed by Enseco. In the case where the SCS components are ident to the DCS components, e.g. metals analyses, the SCS are analyzed with every batch of samples which does not include the DCS pair.

4.1.3 Method Blank: A method blank consists of reagents specific to the method which are taken through the entire analytical process to assess the level of contamination which exists in the analytical system and which might lead to the reporting of elevated concentrations or false positives. Method blanks for aqueous samples include, in addition to reagents, an aliquot of analyte-free water equal in volume to the sample volume required in the method SOP. A method blank is analyzed with every batch of samples processed.

4.1.4 Surrogate Spikes of Samples: Certain methods, typically organic methods, specify that surrogates must be added to each sample and QC sample processed by the method. These surrogates are added routinely as specified in Appendix A.

Surrogates in QC samples are used to measure lab performance However, surrogate recoveries in samples are not used to determine whether the laboratory was in control during data generation. Surrogate recoveries in samples are a measure of the effect of the matrix on method performance and are therefore an element of Matrix Specific QC (see SOP No. M-E 003).

4.2 Control Limits for Laboratory QC Samples.

4.2.1 DCS. DCS are monitored for accuracy (average recovery of each analyte in the DCS pair) and precision (Relative Percent Difference (RPD)) between each analyte in the DCS pair. Control limits for accuracy are based on the average recovery of each pair of DCS generated for a QC lot as described below.

4.2.1.1 Accuracy is determined by calculating the percent recovery of each spike component in the DCS using the following formula.

$$\text{DCS (\% Recovery)} = \frac{X}{T} \times 100$$

where: X = the observed value of the measurement.
T = the "true" value

The average recovery is calculated as follows.

$$\text{Average DCS Recovery} = \frac{DCS_1 - DCS_2}{2}$$

where: $DCS_1$ = first DCS recovery value
$DCS_2$ = second DCS recovery value

The control limits for accuracy are based on the average recovery of the DCS pairs. These limits are calculated from the historical average recovery of the average DCS values ± 3 standard deviation units.

Control limits are updated every six months using the most recent nine months of DCS data. CLP limits are used until sufficient data are generated to determine laboratory limits. If the calculated limits exceed CLP limits, CLP limits are implemented.

4.2.1.2 Precision is monitored by calculating the Relative Percent Difference (RPD) of the percent recoveries of each spike component in the DCS using the following formula:

$$RPD = \frac{|DCS_1 - DCS_2|}{(DCS_1 - DCS_2)/2} \times 100$$

where: RPD = Relative Percent Difference
$DCS_1$ = first DCS recovery value
$DCS_2$ = second DCS recovery value

The control limits for precision for the DCS are calculated based on the historical RPD and range from zero (no difference between DCS results) to the average RPD plus 3 standard deviation units.

Control limits are updated every six months using the most recent nine months of DCS data. CLP limits are used until sufficient data are generated to determine laboratory limits. If the calculated limits <u>exceed</u> CLP limits, CLP limits are implemented.

4.2.2 SCS. SCS are monitored for accuracy (percent recovery) of spiked analytes.

Accuracy is determined by calculating the percent recovery of each spike component in the SCS.

$$\text{Percent Recovery} = \frac{X}{T} \times 100$$

where: X = the observed value of the measurement.
T = the "true" value

The control limits for accuracy for the SCS are calculated based on the historical average recovery of all SCS values $\pm$ 3 standard deviation units.

Control limits are updated every six months using the most recent nine months of SCS data. CLP limits are used until sufficient data are generated to determine laboratory limits. If the calculated limits <u>exceed</u> CLP limits, CLP limits are implemented.

4.2.4 Surrogate Spikes of Samples: Enseco uses the recoveries of surrogate spikes added to method blanks (SCS) to monitor laboratory performance. Surrogate recoveries in samples are not used to make decisions on whether the laboratory was "in control" during data generation; sample surrogate recoveries are a measure of the effect of the matrix on the method performance and are, therefore, an element of Matrix Specific QC (see SOP No. M-EQA-003).

4.3 Generation of Laboratory QC Samples.

4.3.1 Volatile Organics (VOA):

4.3.1.1 DCS. A pair of DCS are analyzed for every 20 samples. DCS components and spike concentrations for VOA methods are given in Appendix A, Part 1.

For aqueous samples, the DCS components are spiked into organic-free water. For solid samples, organic-free Ottawa sand is used as the matrix.

4.3.1.2 SCS. A SCS is analyzed with every analytical batch. An analytical batch for VOA's is defined as VOA's analyzed within a 12-hour period. SCS components and spike concentrations are given in Appendix A, Part 1.

For aqueous samples, the SCS components are spiked into organic-free water. For solid samples, the SCS components are spiked into the extraction solvent when the method blank and the SCS are incorporated as one sample. For methods that do not have surrogates, the SCS, analyzed in addition to the method blank, is spiked with the components in the DCS. When the SCS and method blank are analyzed as separate samples, the SCS will incorporate Ottawa sand as the solid matrix.

4.3.1.3 Method Blank. For aqueous samples, an aliquot of organic-free water serves as the method blank for VOA analyses. For solid samples, a reagent blank is used as the method blank. Ottawa sand is never incorporated into the method blank.

4.2.3 Method Blanks. Control limits for method blanks are as follows:

4.2.3.1 For organic analyses, the concentration of target analytes in the method blank must be below the reporting limit for that analyte in order for the method blank to be considered acceptable. An exception is made for common laboratory contaminants [methylene chloride, acetone, 2-butanone, toluene and bis(2-ethylhexyl)phthalate] which may be present in the method blank at up to 5 times the reporting limit and still be considered acceptable.

4.2.3.2 For metals analyses, the concentration of the target analytes in the method blank must be below two times the reporting limit. If the method blank value for a target analyte lies below the reporting limit, the reporting limit for that analyte in the associated samples is unaffected. If the method blank value lies between the reporting limit and two times the reporting limit, the reporting limit for that analyte in the associated samples is raised to the level found in the method blank. A method blank containing analyte(s) above two times the reporting limit is considered unacceptable unless the lowest concentration of the analyte in the associated samples is at least ten times the method blank concentration (as per CLP protocol).

4.2.3.3 For conventional inorganic tests, the method SOP directs how the method blank is treated. Generally, a reagent blank is used both to zero the equipment and as one of the calibration standards. If a preparation step is required for the analysis, then a prep blank is also analyzed to determine the extent of contamination or background interference. In most cases, the concentration found in the prep blank is subtracted from the concentration found in any associated sample prior to calculating the final result. Method blanks have no application or significance for some conventional inorganic parameters (e.g. pH).

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for VOA's is defined as VOA's analyzed within a 12-hour period.

For most VOA methods, at least one designated surrogate is spiked into the method blank. These surrogates also serve as the SCS components for these methods, and therefore, the method blank and the SCS are one and the same (see Appendix A, Part 1).

4.3.1.4 Surrogates Spiked into Samples. Surrogates compounds are required to be spiked into samples and QC samples for most VOA methods. Surrogates designated by the methods and spike concentrations to use are given in Appendix A, Part 1. These surrogates should be spiked into all samples and QC samples.

4.3.2 Extractable Organics:

4.3.2.1 DCS. A pair of DCS are analyzed for every 20 samples. DCS components and spike concentrations are given in Appendix A, Part 2. For aqueous samples, DCS components are spiked into organic-free water. For solid samples, the DCS components are spiked into the organic-free Ottawa sand used as the control matrix.

4.3.2.2 SCS. A SCS is analyzed with every analytical batch. An analytical batch for extractable organics is defined as samples extracted or prepared at the same time. SCS components and spike concentrations are given in Appendix A, Part 2.

For aqueous samples, the SCS components are spiked into organic-free water. For solid samples, the SCS components are spiked into the extraction solvent when the method blank and the SCS are incorporated as one sample. For methods that do not have surrogates, the SCS, analyzed in addition to the method blank, is spiked with the components in the DCS. When the SCS and method blank are analyzed as separate samples, the SCS will incorporate Ottawa sand as the solid matrix.

4.3.2.3 Method Blank. For aqueous samples, an aliquot of organic-free water taken through the entire analytical process serves as the method blank. For solid samples, a reagent blank is used as the method blank. Ottawa sand is never incorporated into the method blank.

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for extractable organics is defined as samples extracted or prepared at the same time.

For many extractable organic methods, at least one designated surrogate is spiked into the method blank. These surrogates also serve as the SCS components. For these methods, the method blank and the SCS are one and the same (see Appendix A, Part 2).

4.3.2.4 Surrogates Spiked into Samples. Surrogate compounds are required to be spiked into samples and QC samples for many extractable organic methods. Surrogates designated by the methods and spike concentrations to use are given in Appendix A, Part 2. These surrogates should be spiked into all samples and QC samples.

4.3.3 Metals:

4.3.3.1 DCS. A pair of DCS are analyzed for every 20 samples. Separate QC Categories have been established by matrix for Dissolved, Total, and Total Recoverable Metals. DCS components and spike concentrations for metals are given in Appendix A, Part 3. For aqueous samples, the DCS components are spiked into deionized water and taken through the procedure. For solid samples, the DCS components are spiked into ~~Ottawa sand~~ EPA Solid Matrix and taken through the procedure.

4.3.3.2 SCS. The SCS has the same components as the DCS. The SCS is analyzed with every analytical batch which does not include the DCS. An analytical batch for

metals is defined as samples which are digested at one time (total and total recoverable metals) or samples analyzed within a 24-hour period (dissolved metals). For solid samples, the SCS components are spiked into ~~Ottawa sand~~. EPA Solid Matrix

4.3.3.3 Method Blank. For both aqueous and solid samples, an aliquot of deionized water taken through the entire analytical process serves as the method blank. Ottawa sand is never incorporated into the method blank for solid samples.

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for metals is defined as samples which are digested at one time (total and total recoverable metals) or samples analyzed within a 24-hour period (dissolved metals).

For metals analyses, the method blank is analyzed <u>in addition</u> to the DCS and SCS (see Appendix A, Part 3).

4.3.3.4 Surrogates Spiked into Samples. Not applicable to metals analyses.

4.3.4 Conventional Inorganics:

4.3.4.1 DCS. A pair of DCS are analyzed for every 20 samples. DCS components and spike concentrations are given in Appendix A, Part 4. For aqueous and solid samples, the DCS components are spiked into deionized water. No solid matrix is incorporated into the DCS for conventional inorganics.

4.3.4.2 SCS. The SCS has the same components as the DCS. The SCS is analyzed with every analytical batch which does not include a pair of DCS. An analytical batch for conventional inorganics is defined as samples which are prepared or analyzed within a 24-hour period.

4.3.4.3 Method Blank. For both aqueous and solid samples, an aliquot of deionized water taken through the entire analytical process serves as the method blank for conventional inorganic analyses. No solid matrix is incorporated in the method blank for solid samples.

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for conventional inorganics is defined as samples which are prepared or analyzed within a 24-hour period.

For conventional inorganic analyses, the method blank is analyzed <u>in addition</u> to the DCS and SCS (see Appendix A, Part 4).

4.3.4.4 Surrogates Spiked into Samples. Not applicable to Conventional Inorganic analyses.

4.4 <u>Tracking of Laboratory QC Samples</u>.

4.4.1 All samples analyzed are assigned a QC lot number. This lot number is used to store and track the QC information associated with a particular sample or group of samples. QC data are identified with the following components:

a) the QC category,
b) the QC type,
c) the QC lot number, and
d) the QC run number.

4.4.2 The QC Category and QC Type: Each test in LIMS is assigned to a QC category which specifies the QC type. The QC type is DCS, SCS, or Method Blank. Associated with each QC type is the component list for the QC category. Variations of the same test (e.g., VOA-HSL list, VOA-PP list, and VOA-Refinery list) may be assigned to the same QC category. QC categories are assigned to the major analytical groups as follows:

4.4.2.1 Volatile Organics (VOA). QC categories are assigned based on the detection system used. Separate QC categories are assigned for aqueous and solid matrices. Separate QC categories are assigned for

low-level and medium-level methods. An instrument specific identifier can be assigned to track VOA QC as instrument specific QC. QC categories and the components and spike levels for each QC type for volatile organics are specified in Appendix A1.

4.4.2.2 Extractable Organics. QC categories are assigned based on prep technique/detection system combination. Separate QC categories are assigned for aqueous and solid matrices. Separate QC categories are assigned for low-level and medium-level methods. QC categories and the components and spike levels for each QC type for extractable organics are specified in Appendix A2.

4.4.2.3 Metals. QC categories are assigned based on prep technique/detection system combination. Separate categories are assigned to aqueous and solid matrices. The categories for aqueous matrix are Dissolved Metals, Total Metals, and Total Recoverable Metals. The QC category Total Recoverable Metals is assigned for the solid matrix. QC categories and the components and spike levels for each QC type for metals are specified in Appendix A3.

4.4.2.4 Conventional Inorganics. QC categories are assigned based on the analytical method. Aqueous and solid matrices are typically tracked in the same QC category. Separate QC categories are assigned when there are significant differences in the sample prep. QC categories and the components and spike levels for each QC type for conventional inorganics are specified in Appendix A4.

4.4.3 The QC Lot: Each DCS pair which is analyzed within a QC category is assigned a unique QC lot number. This number follows the format YYMMMDD [letter] (e.g., 88MAR19A).

where: YY is the year.
MMM is the month, abbreviated as three letters.
DD is the day that the lot number is assigned, and
[letter] is a single letter (from A - Z) designating distinct QC lots for the day.

This QC lot number is assigned to the DCS pair and all QC and environmental samples associated with these DCS. Typically, the same QC lot number is assigned to the DCS pair and the next 20 samples analyzed which have the same QC category.

The QC lot is assigned at the first step of the prep/analysis and tracks with the samples through analysis, data entry, and reporting. The QC lot is associated with the samples in LIMS during the data entry process.

4.4.4 The QC Run: Each analytical batch which is processed is assigned a QC run number which is used to track and store SCS and Blank results. The QC run is associated with a specific QC category and QC lot. The QC run number follows the format YYMMMDD [letter] (i.e., 88MAR15B)

where: YY is the year,
MMM is the month, as a three letter abbreviation,
DD is the day that the lot number is assigned, and
[letter] is a single letter (from A - Z) designating distinct QC runs for the day.

This QC run number is assigned to all samples analyzed within the analytical batch (QC samples plus environmental samples) and is also associated with a specific QC lot number.

The QC run is assigned at the first step of the prep/analysis and tracks with the samples through analysis, data entry, and reporting. The QC run is associated with the samples in LIMS during the data entry process.

Example:

Day 1 (March 4, 1988): 5 water samples analyzed for VOA-HSL. 20 samples have been analyzed since the last DCS were analyzed.

Day 2 (March 6, 1988): 7 water samples analyzed for VOA-PP.

Day 3 (March 7, 1988): 5 water samples analyzed for VOA-HSL.

Day 4 (March 9, 1988): 5 water samples analyzed for VOA-PP.

VOA-HSL and VOA-PP are both in the same QC category: 624-A. The lot numbers would be assigned as follows:

Day 1: QC category 624-A
QC lot 88MAR04A
QC run 88MAR04A

These numbers are assigned to the DCS, the Method Blank, and the 6 samples analyzed on Day 1. A method blank must be analyzed with every batch. With this test, the method blank also contains surrogates and, therefore, also serves as an SCS, although an SCS is not required to be analyzed since DCS are included with the batch.

Day 2: QC category 624-A
QC lot 88MAR04A
QC run 88MAR06A

These numbers are assigned to the 7 samples and the Method Blank/SCS analyzed on Day 2. DCS are not required because 20 samples have not been analyzed since the last set of DCS. A combination Method Blank/SCS is analyzed with the set to provide QC information for that day's analysis. A method blank must be analyzed with every batch. With this test, the method blank also contains surrogates and, therefore, also serves as the SCS.

Day 3: QC category 624-A
QC lot 88MAR04A
QC run 88MAR07A

These numbers are assigned to the 5 samples and the Method Blank/SCS analyzed on Day 3. The rationale is exactly the same as outlined in Day 2.

Day 4: QC category 624-A
QC lot 88MAR09A
QC run 88MAR09A

These numbers are assigned to the DCS, the Method Blank/SCS and the 5 samples analyzed on Day 4. DCS are required because 20 samples have been analyzed since the last DCS pair were run.

4.5 Scheduling of Samples.

4.5.1 Samples should be scheduled for analysis such that all environmental samples and QC samples with the same QC run number are carried through the entire analytical process together.

4.5.2 In instances where it is impossible to analyze all samples with the same QC run number together, the QC samples should be analyzed with the first set of samples analyzed from that batch. QC samples, including method blanks, should be analyzed only once. (EPA-CLP and NJ Tier I work require the method blanks be analyzed on each of the instruments used t generate data for samples associated with the method blank. In these cases, method blanks may be analyzed multiple times. However, samples should be scheduled to minimize the number of method blank analyses required).

4.5.3 Samples should be scheduled for analyses according to the following priorities:

a) Meet holding times
b) Meet promised due date
c) Keep samples within a QC run together.

4.6 Analysis of Samples.

4.6.1 The QC program is structured so that decisions about the acceptability of the data can be made by the analyst at the time the data are generated.

4.6.2 Sample analysis should be conducted according to the following protocol:

4.6.2.1 Prepare the instrument for analysis by calibrating the instrument and verifying that the calibration is within acceptable guidelines (see SOP's related to specific method and equipment.)

4.6.2.2 Analyze all QC samples (method blanks, DCS and/or SCS) in the QC run. Determine if QC results are within acceptable limits. If data are outside control limits DO NOT PROCEED. CONTACT YOUR SUPERVISOR. Proceed with step (4.6.2.4). Analysts using autosamplers must verify that QC results are acceptable before leaving the instrument for unattended operation.

4.6.2.3 If QC samples are acceptable, proceed with analysis of other samples associated with the QC run.

4.6.2.4 If QC results are unacceptable, determine the source of the problem. Reevaluate standards, instrumentation, sample prep, etc. Each analyst should have a checklist to aid in identifying problems. Determine whether the analysis of samples should proceed using the following criteria:

a. Validate instrument operational setting, sensitivity, and linearity. If a problem is detected and corrected, reanalyze standard(s) and reanalyze QC samples. If QC data are within control limits, proceed with the analysis of the other samples in the QC run. If QC samples are still outside limits, continue to troubleshoot or go to Step b.

b. Validate that analytical standards are good. If a problem with the standards is detected, use a different standard, recalibrate, and reanalyze QC samples. If QC data are within control limits, proceed with analysis of the other samples in the QC run. If QC data are still outside of the limits, go to Step c.

c. Validate that sample prep was performed correctly. Check prep sheets for any anomalies. If no anomalies are found, reprep the samples. If samples cannot be reprepped due to lack of sample, analyze the other samples in the QC run and report data with a qualifier indicating that the Laboratory QC performed with the sample was out of

normal control limits. If sufficient sample remains, but holding times have expired, reprep samples unless otherwise required by the client.

If problems with the prep are discovered based on examination of prep sheets, notify the supervisor. The supervisor decides whether to reprep the samples based on the following criteria:

1. If the problems identified with the prep of the QC sample clearly affected only the QC sample and none of the other samples in the run (example: QC sample taken to dryness in the KD), a decision should be made not to reprep the samples, and the sample results can be reported without qualifiers.

2. If the problems identified could have potential affected all of the samples in the QC run, the samples must be reprepped. If the samples cann be reprepped for some reason, (i.e., inadequate sample volume) the sample results must be reported with a qualifier.

3. The entire episode, including reasons to support the final decision, must be documented on an anomaly form. The anomaly form is submitted t the QA Department. It is also forwarded, with the results, to the Program Administrator.

4.7 Calculation of QC Results.

4.7.1 DCS: The percent recovery of all spike components is calculated using the equation given in 4.2.1.1.

The precision of the DCS pairs is calculated by determining the RPD for each spike component using the equation given in 4.2.1.2.

4.7.2 SCS: The percent recovery of all spike components in the SCS is measured using the equation given in 4.2.2.

4.7.3 Method Blanks: The concentrations of target analytes are measured using quantitative techniques described in the me SOP.

4.7.4 Surrogate Spikes of Samples: The percent recovery of all surrogate spikes is calculated using the equation given in 4.2.2.

4.8 Interpretation of QC Results

4.8.1 Data acceptability is based upon the results of Laboratory Control Samples (DCS, SCS, and Method Blanks) unless otherwise negotiated with the client. Data acceptability based on criteria other than DCS, SCS, and Method Blanks falls under Matrix Specific QC (see SOP No. M-EQA-003).

4.8.2 The QC data from the DCS and SCS associated with a particular QC lot are compared to established control limits. QC data must fall within established control limits in order for the laboratory to be considered "in control" when samples from that QC lot were analyzed.

4.8.2.1 For single analyte tests, all DCS recovery and precision data and all SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Method blank values must be acceptable.

4.8.2.2 For organic multi-analyte tests, at least 80% of the average of each pair of DCS recovery data, and at least 80% of the DCS precision data, and at least 80% of the SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Method blank values must be acceptable. The 80% rule is tabulated below.

a.

| # components in DCS | maximum numbers of values permitted outside control limits: average recovery | precision |
|---|---|---|
| 1 - 4 | 0 | 0 |
| 5 - 9 | 1 | 1 |
| 10 - 14 | 2 | 2 |
| 15 - 19 | 3 | 3 |
| 20 - 24 | 4 | 4 |

b.

| # components in SCS | maximum number of values permitted outside control limits |
|---|---|
| 1 - 4 | 0 |
| 5 - 9 | 1 |
| 10 - 14 | 2 |
| 15 - 19 | 3 |
| 20 - 24 | 4 |

4.8.2.3 For metals, all analytes requested by the client in the DCS associated with the samples must be within the control limits. Method blank values must be acceptable.

The DCS component lists for metals are divided into two categories:

a) Components which have been requested by the client to be analyzed in the samples associated with the DCS or SCS. The DCS or SCS counterpart of all analytes requested by the client must be within control limits in order for the laboratory to be considered "in control".

b) Components which are contained in the DCS or SCS but are not requested by the client. A maximum of three (3) analytes falling into this category may be outside control limits and the laboratory judged to be "in control". An anomaly form must be completed for these analytes to prevent analytical problems from going unresolved for a prolonged period of time.

Example: Two (2) projects are associated with the same DCS. Project A requires analyses of Ba, Ca, Cu, and V. Project B requires Cu, Ni, Na, Fe, and Zn.

In order for the lab to be "in control" and for analyses to proceed, the DCS values for Ba, Ca, Cu, V, Ni, Na, Fe, and Zn must be within control limits.

Of the remaining 13 metals in the DCS, at least 10 must also be within control limits.

4.8.3 Analytical data which are generated with DCS, SCS, and method blanks which fall within established control limits based on the criteria in 4.8.2, are judged to be in control. Data which are generated with DCS, SCS, or method blanks which fall outside of acceptance criteria are considered suspect and are reanalyzed or reported with qualifiers.

4.8.4 Sample results are accepted or rejected on the basis of the SCS, associated DCS and method blanks. If surrogates in the samples are outside of the control limits and the SCS is in control, the sample results are accepted and the surrogate recoveries are assumed to be affected by the matrix. In these cases, sample processing should be assessed to ensure that obvious problems with sample preparation and analysis (e.g. concentration of the sample to dryness, leaking septum) have been ruled out.

For certain projects, clients may specifically request sample reruns if the surrogates are outside specified limits in the samples (Lab limits or CLP limits). This is considered to be an element of Matrix Specific QC. Enseco policy duplicates the EPA-CLP program: a sample with surrogate recoveries outside the limits will be reanalyzed with the understanding that if, on reanalysis, surrogates fall within the limits, the lab reports the new data at no charge to the client. If, however, the second analysis duplicates the results from the first analysis, indicating a matrix effect, the client is charged for the second analysis. The same policy applies to matrix spike data (see SOP No. M-EQA-003).

4.9 <u>Reporting of Laboratory Specific QC</u>.

4.9.1 DCS, SCS, and method blank data are entered into LIMS with sample data. <u>ALL</u> QC data must be entered into LIMS, even if it fails the data acceptability criteria and samples are reanalyzed with new QC samples. The Blank QC Type contains method blank information for components typically found in a method blank. These components are lab-specific, but at a minimum, the components specified under Blank in Appendix A should be included in the Blank QC Type. Other compounds are

assumed to be ND (not detected). If the method blank is found to contain compounds other than those listed in the Blank QC type, they must be documented on the anomaly form and reported on the method blank data sheet.

4.9.2 Sample data must be reported with associated DCS, SCS, and method blank data. Sample data for organics and metals are not corrected for levels of analytes observed in the method blank. For conventional inorganic tests, the method SOP directs how the blank is treated. Method blank information is reported for all components in the analyte list for all organic and metals tests. Method blanks are not reported for most conventional inorganic tests, however, method blanks will be reported for Ion Chromatography, Cyanide, Phenol, TKN, Total Phosphorous, Fluoride by Distillation, Ammonia by Distillation, Chromium VI in soil, and Formaldehyde. QC data are reported in the QC section of the final report.

4.9.3 Report formats are shown in Appendix B.

5.0 RESPONSIBILITIES

5.1 The Analyst. The primary responsibility for implementation of the QC Program lies with the analyst. QC information needed for standard reports is generated, documented, and entered into LIMS by the analyst.

5.2 Management. The supervisors and managers who direct the analytical work are directly responsible for ensuring that all employees reporting to them are complying with the QC procedures set forth in this SOP.

5.3 QA Department. The QA Department is responsible for auditing the laboratory to ensure that the QC procedures set forth in this SOP are followed routinely. They are responsible for reviewing anomaly forms and initiating investigations of procedures and corrective actions, as appropriate.

5.4 Program Administrators/Technical Directors. Program Administrators and Technical Directors have the responsibility to convey to their client the quality of the data based on information contained on anomaly forms and in the LIMS QC database.

## 6.0 COMMENTS

As LIMS becomes more sophisticated, we will be tracking data based on both control limits (± 3 std. dev. units) and warning limits (± 2 std. dev. units). Enseco laboratories now have the ability to generate control charts using the QC data stored in LIMS. It is critical that ALL QC data, both good and bad, be entered into LIMS. The Method Blank database will be refined to allow entry of compounds other than common contaminants in an upcoming revision. The SOP will be updated at that time to reflect these changes.

## 7.0 DEFINITIONS

7.1 Quality Control (QC). The routine application of specific, well-documented procedures which ensures the generation of data of known and accepted quality which fulfill the objectives of the Quality Assurance Program.

7.2 Laboratory Control Sample (LCS). A well-characterized laboratory generated sample which is used to monitor the precision and accuracy of the analytical process or to assess the degree of laboratory contamination.

7.2 Duplicate Control Sample (DCS) A standard, control matrix which is spiked with a group of target compounds representative of the method analytes and is used to measure, on an ongoing basis, the precision and accuracy of a method, independent of matrix effects.

7.3. Single Control Sample (SCS). A standard, control matrix spiked with surrogate compounds appropriate to the method being used. In cases where no surrogate is available, the components of the DCS are used in the SCS. The SCS is used to monitor recovery with each analytical batch.

7.4 Method Blank. Reagents specific to a method which are carried through every aspect of the procedure to access the level of contamination which exists in the analytical system.

7.5 Analytical Batch. A group of samples considered to have been exposed to the same analytical conditions by virtue of the time frame in which they were analyzed.

7.6 Ottawa Sand. This material is pure silica sand of particle size 20-30 mesh, specially prepared to conform to ASTM C190 and AASHTO T-132. (Ottawa is a trademark of Bellrose Silica). The laboratory must verify by analysis of the sand using the appropriate method that each lot of sand used is organic- and metal-free at the levels required for spiking target analytes as described in this SOP. Ottawa sand is available from Fisher Scientific.

# APPENDIX A

## Part 1

## Laboratory Specific QC for Volatile Organics

## Method 601/8010
## Halogenated Volatile Organics

| QC Category | Aqueous | Solid |
|---|---|---|
| | 601-A | 8010-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| 1,1-Dichloroethane | 5.0 | 500 |
| Chloroform | 5.0 | 500 |
| Bromodichloromethane | 10.0 | 1,000 |
| Trichloroethene | 5.0 | 500 |
| Chlorobenzene | 5.0 | 500 |
| SCS | | |
| Bromochloromethane | 30 | 3,000 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

**Method 602/8020**
**Aromatic Volatile Organics**

| QC Category | Aqueous | Solid |
|---|---|---|
| | 602-A | 8020-S |

| | Spike Level | |
|---|---|---|
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Benzene | 5.0 | 500 |
| Toluene | 5.0 | 500 |
| 1,3-Dichlorobenzene | 5.0 | 500 |
| Ethyl benzene | 5.0 | 500 |
| Total Xylenes** | 5.0 | 500 |
| SCS | | |
| a,a,a,-Trifluorotoluene | 30 | 3,000 |

Surrogates added to sample

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

** Spiked as o-xylene; reported as Total Xylenes.

## Method 603/8030
## Acrolein/Acrylonitrile

| QC Category | Aqueous | Solid |
|---|---|---|
| | 603-A | 8030-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Acrolein | 25 | 2,500 |
| Acrylonitrile | 25 | 2,500 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

**Method 624/8240**
**Volatile Organics--GC/MS**

| QC Category | Aqueous | Low Level Solid | Medium Level Solid |
|---|---|---|---|
| | 624-A | 8240-SL | 8240-S |

| DCS | Aqueous (ug/L) | Spike Level Low Level Solid+ (ug/Kg) | Medium Level Solid* (ug/Kg) |
|---|---|---|---|
| 1,1-Dichloroethene | 50 | 50 | 5,000 |
| Trichloroethene | 50 | 50 | 5,000 |
| Chlorobenzene | 50 | 50 | 5,000 |
| Toluene | 50 | 50 | 5,000 |
| Benzene | 50 | 50 | 5,000 |
| **SCS** | | | |
| 1,2-Dichloroethane-$d_4$ | 50 | 50 | 5,000 |
| 4-Bromofluorobenzene (BFB) | 50 | 50 | 5,000 |
| Toluene-$d_8$ | 50 | 50 | 5,000 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

+ Assumes 5 g sample.
* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

## Method 502.2

| QC Category | 5022-A |
|---|---|
| | Spike Level (ug/L) |
| DCS* | |
| Benzene | 5 |
| Vinyl Chloride | 2 |
| Carbon tetrachloride | 5 |
| 1,2-Dichloroethane | 5 |
| Trichloroethene | 5 |
| 1,1-Dichloroethene | 7 |
| 1,1,1-Trichloroethane | 10 |
| 1,4-Dichlorobenzene | 10 |
| Tetrachloroethene | 5 |
| Xylene (each isomer) | 10 |
| Chlorobenzene | 10 |
| Ethylbenzene | 10 |
| 1,3-Dichloropropene | 5 |
| 1,1,2,2-Tetrachloroethane | 5 |
| 1,1,2-Trichloroethane | 5 |
| Total THMs | |
| Chloroform | 10 |
| Bromoform | 10 |
| Bromodichloromethane | 10 |
| Dibromochloromethane | 10 |
| trans-1,2-Dichloroethene | 10 |
| 1,3-Dichlorobenzene | 10 |
| Methylene chloride | 10 |
| cis-1,2-Dichloroethene | 10 |
| 1,2-Dichlorobenzene | 10 |
| Dibromomethane | 10 |
| 1,1-Dichloropropane | 10 |
| Styrene | 10 |
| Chloromethane | 10 |
| Bromomethane | 10 |
| 1,2,3-Trichloropropane | 10 |
| 1,1,1,2-Tetrachloroethane | 10 |
| Chloroethane | 10 |
| 2,2-Dichloropropane | 10 |
| o-Chlorotoluene | 10 |
| p-Chlorotoluene | 10 |
| Bromobenzene | 10 |

* Any additional analytes which are added to the component list must be added to the DCS to comply with method requirements that ALL components analyzed for are contained in the QC check sample. The DCS serves as the QC check sample.

## Method 502.2 Continued

QC Category 5022-A

| | Spike Level (ug/L) |
| --- | --- |
| SCS | |
| 1-Chloro-4-fluorobenzene | 5 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

## Method 524.2

| QC Category | 5242-A |
|---|---|
| | Spike Level (ug/L) |
| DCS* | |
| Benzene | 5 |
| Vinyl Chloride | 2 |
| Carbon tetrachloride | 5 |
| 1,2-Dichloroethane | 5 |
| Trichloroethene | 5 |
| 1,1-Dichloroethene | 7 |
| 1,1,1-Trichloroethane | 10 |
| 1,4-Dichlorobenzene | 10 |
| Tetrachloroethene | 5 |
| Xylene (each isomer) | 10 |
| Chlorobenzene | 10 |
| Ethylbenzene | 10 |
| 1,3-Dichloropropene | 5 |
| 1,1,2,2-Tetrachloroethane | 5 |
| 1,1,2-Trichloroethane | 5 |
| Total THMs | |
| Chloroform | 10 |
| Bromoform | 10 |
| Bromodichloromethane | 10 |
| Dibromochloromethane | 10 |
| trans-1,2-Dichloroethene | 10 |
| 1,3-Dichlorobenzene | 10 |
| Methylene chloride | 10 |
| cis-1,2-Dichloroethene | 10 |
| 1,2-Dichlorobenzene | 10 |
| Dibromomethane | 10 |
| 1,1-Dichloropropane | 10 |
| Styrene | 10 |
| Chloromethane | 10 |
| Bromomethane | 10 |
| 1,2,3-Trichloropropane | 10 |
| 1,1,1,2-Tetrachloroethane | 10 |
| Chloroethane | 10 |
| 2,2-Dichloropropane | 10 |
| o-Chlorotoluene | 10 |
| p-Chlorotoluene | 10 |
| Bromobenzene | 10 |

* Any additional analytes which are added to the component list must be added to the DCS to comply with method requirements that ALL components analyzed for are contained in the QC check sample. The DCS serves as the QC check sample.

## Method 524.2

QC Category 5242-A

| SCS | Spike Level (ug/L) |
|---|---|
| 1,2-Dichloroethane-$d_4$ | 10 |
| 4-Bromofluorobenzene (BFB) | 10 |
| Toluene-$d_8$ | 10 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

## Trihalomethanes

QC Category THM-A

DCS

| | Spike Level (ug/L) |
|---|---|
| Chloroform | 2 ug/L |
| Bromoform | 2 ug/L |
| Dibromochloromethane | 2 ug/L |
| Bromodichloromethane | 2 ug/L |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

# APPENDIX A

## Part 2

## Laboratory Specific QC for Extractable Organics

Method 604/8040
Phenols

| QC Category | Aqueous | Solid |
|---|---|---|
| | 604-A | 8040-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| 2-Nitrophenol | 20 | 666 |
| Phenol | 20 | 666 |
| 2,4-Dinitrophenol | 60 | 2,000 |
| Pentachlorophenol | 100 | 3,330 |
| SCS | | |
| 2,4,6-Tribromophenol | 100 | 3,330 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

**Method 606/8060**
**Phthalate Esters**

| QC Category | Aqueous | Solid |
|---|---|---|
| | 606-A | 8060-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Diethylphthalate | 10 | 2500 |
| Butylbenzylphthalate | 1 | 250 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

Method 608/8080
Organochlorine Pesticides

| QC Category | Aqueous | Low Level Solid | Low Level Solid with GPC | Medium Level Solid |
|---|---|---|---|---|
| | 608-A | 8080-S | 8080-GPC-S | 8080-MED-S |

Spike Level

| DCS | Aqueous (ug/L) | Low Level Solid (ug/Kg) | Low Level Solid with GPC (ug/Kg) | Medium Level Solid (ug/Kg) |
|---|---|---|---|---|
| Lindane | 0.2 | 27 | 54 | 2000 |
| Heptachlor | 0.2 | 27 | 54 | 2000 |
| Aldrin | 0.2 | 27 | 54 | 2000 |
| Dieldrin | 0.5 | 67 | 134 | 5000 |
| Endrin | 0.5 | 67 | 134 | 5000 |
| p,p'-DDT | 0.5 | 67 | 134 | 5000 |
| **SCS** | | | | |
| Dibutylchlorendate | 1 | 67 | 134 | 1000 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

**Method 608/8080**
**PCB only**

| QC Category | Aqueous | Solid |
|---|---|---|
| | PCB-A | PCB-S |

| | Spike Level | |
|---|---|---|
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Aroclor 1254 | 5 | 167 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

## PCB in Oil

| QC Category | PCBO-W |
|---|---|
| | Spike Level (ug/Kg)* |
| DCS | |
| Aroclor 1254 | 5,000 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample

**Method 8100**
**PNA's by GC/FID**

| QC Category | Aqueous | Solid |
|---|---|---|
| | 610-GC-A | 8100-S |

| | Spike Level | |
|---|---|---|
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Acenaphthene | 50 | 2,500 |
| Pyrene | 50 | 2,500 |
| Benzo(a)pyrene | 50 | 2,500 |
| SCS | | |
| Orthoterphenyl | 20 | 1,000 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 20 g sample (wet weight).

## Method 612/8120
## Chlorinated Hydrocarbons

| QC Category | Aqueous | Solid |
|---|---|---|
| | 612-A | 8120-S |

| | Spike Level | |
|---|---|---|
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| 1,2,4-Trichlorobenzene | 1.0 | 33.3 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

**Method 614/8140**
**Organophosphate Pesticides**

| QC Category | Aqueous | Solid |
|---|---|---|
| | 614-A | 8140-S |

| DCS | Spike Level Aqueous (ug/L) | Spike Level Solid* (ug/Kg) |
|---|---|---|
| Phorate | 10 | 330 |
| Diazinon | 10 | 330 |
| Malathion | 10 | 330 |
| Parathion | 10 | 330 |
| Ethion | 10 | 330 |
| Methyl Parathion | 10 | 330 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

## Method 615/8150
## Phenoxy Acid Herbicides

| QC Category | Aqueous | Solid |
|---|---|---|
| | 615-A | 8150-S |

| | Spike Level | |
|---|---|---|
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| 2,4-D | 5.0 | 170 |
| 2,4,5-TP (Silvex) | 1.0 | 33 |
| 2,4,5-T | 1.0 | 33 |
| SCS | | |
| 2,4-Dichlorophenylacetic acid (DCAA) | 5 | 100 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 50 g sample (wet weight)

**Method 625/8270**
**Semivolatile Organics--GC/MS**

| QC Category | Aqueous | Low Level Solid | Low Level Solid with GPC | Medium Level Solid |
|---|---|---|---|---|
| | 625-A | 8270-S | 8270-GPC-S | 8270-MED-S |

Spike Level

| DCS | Aqueous (ug/L) | Low Level Solid* (ug/Kg) | Low Level Solid with GPC* (ug/Kg) | Medium Level Solid- (ug/Kg) |
|---|---|---|---|---|
| Pentachlorophenol | 200 | 6,670 | 13,300 | 200,000 |
| Phenol | 200 | 6,670 | 13,300 | 200,000 |
| 2-Chlorophenol | 200 | 6,670 | 13,300 | 200,000 |
| 4-Chloro-3-cresol | 200 | 6,670 | 13,300 | 200,000 |
| 4-Nitrophenol | 200 | 6,670 | 13,300 | 200,000 |
| 1,2,4-Trichlorobenzene | 100 | 3,330 | 6670 | 100,000 |
| Acenaphthene | 100 | 3,330 | 6670 | 100,000 |
| 2,4-Dinitrotoluene | 100 | 3,330 | 6670 | 100,000 |
| Pyrene | 100 | 3,330 | 6670 | 100,000 |
| N-Nitroso-di-N-propylamine | 100 | 3,330 | 6670 | 100,000 |
| 1,4-Dichlorobenzene | 100 | 3,330 | 6670 | 100,000 |
| **SCS** | | | | |
| Phenol-$d_5$ | 200 | 3,330 | 6670 | 200,000 |
| 2-Fluorophenol | 200 | 3,330 | 6670 | 200,000 |
| 2,4,6-Tribromophenol | 200 | 3,330 | 6670 | 200,000 |
| Nitrobenzene-$d_5$ | 100 | 1,670 | 3330 | 200,000 |
| 2-fluorobiphenyl | 100 | 1,670 | 3330 | 200,000 |
| Terphenyl-$d_{14}$ | 100 | 1,670 | 3330 | 200,000 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight) - Assumes 1 g sample (wet weight)

**Method 8310**
**PNA's by HPLC**

| QC Category | Aqueous | Solid |
|---|---|---|
| | 610-A | 8310-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Naphthalene | 5.0 | 1000 |
| Fluorene | 1.0 | 200 |
| Pyrene | 1.0 | 200 |
| Benzo(a)pyrene | 0.5 | 100 |
| Indeno (1,2,3-cd) pyrene | 0.5 | 100 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

## PCP & Tetrachlorophenol

| QC Category | Aqueous | Solid |
|---|---|---|
| | PCP-A | PCP-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Pentachlorophenol | 5 | 167 |
| 2,3,4,6-Tetrachloropnenol | 5 | 167 |
| SCS | | |
| 2,4,6-Tribromophenol | 5 | 167 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

## Marine Petroleum Hydrocarbons

| QC Category | Aqueous | Solid |
|---|---|---|
| | MHYD-A | MHYD-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Fuel Oil #2 | 1,000 | 50,000 |
| SCS | | |
| Orthoterphenyl | 20 | 1,000 |

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 20 g sample (wet weight)

**Method 504**
**DBCP and EDB**

| QC Category | 504-A |
|---|---|
| DCS | Spike Level Aqueous (ug/L) |
| DBCP | 0.25 |
| EDB | 0.25 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

## Method 619
## Triazines--HPLC

| QC Category | Aqueous | Solid |
|---|---|---|
| | 619-LC-A | 8190-LC-S |

| DCS | Spike Level Aqueous (ug/L) | Spike Level Solid* (ug/Kg) |
|---|---|---|
| Simazine | 10 | 333 |
| Cyanazine | 10 | 333 |
| Atrazine | 10 | 333 |
| Prometon | 10 | 333 |
| Propazine | 10 | 333 |
| Prometryn | 10 | 333 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

**Method 619**
Triazines--GC

| QC Category | Aqueous | Solid |
|---|---|---|
| | 619-GC-A | 8190-GC-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Simazine | 10 | 333 |
| Cyanazine | 10 | 333 |
| Atrazine | 10 | 333 |
| Prometon | 10 | 333 |
| Propazine | 10 | 333 |
| Prometryn | 10 | 333 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

## Method 632
## Carbamates--HPLC

| QC Category | Aqueous | Solid |
|---|---|---|
| | 632-A | 632-S |
| | Spike Level | |
| DCS | Aqueous (ug/L) | Solid* (ug/Kg) |
| Oxamyl | 50 | 1,665 |
| Baygon | 100 | 3,330 |
| Furadan | 100 | 3,330 |
| Carbaryl | 50 | 1,665 |
| Diuron | 5 | 167 |

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

## Dioxins & Furans

| QC Category | Aqueous | Solid |
|---|---|---|
| | DXNFUR-A | DXNFUR-S |
| | Spike Level | |
| DCS | Aqueous (ng)/sample | Solid (ng)/sample |
| 2,3,7,8-TCDF | 10 | 10 |
| 2,3,4,7,8-PECDF | 10 | 10 |
| 1,2,3,4,7,8-HXCDF | 10 | 10 |
| 1,2,3,4,6,7,8-HPCDF | 10 | 10 |
| 1,2,3,4,5,6,7,8-OCDF | 50 | 50 |
| 2,3,7,8-TCDD | 10 | 10 |
| 1,2,3,7,8-PECDD | 10 | 10 |
| 1,2,3,4,7,8-HXCDD | 10 | 10 |
| 1,2,3,4,6,7,8-HPCDD | 10 | 10 |
| 1,2,3,4,5,6,7,8-OCDD | 50 | 50 |

SCS

Same as DCS

Surrogates added to samples

| | | |
|---|---|---|
| 37 Cl-2,3,7,8-TCDD | 10 | 10 |

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

## TCDF & TCDD

| QC Category | Aqueous | Solid |
|---|---|---|
| | TCDD-A | TCDD-S |
| | Spike Level | |
| DCS | Aqueous (ng)/sample | Solid (ng)/sample |
| 2,3,7,8-TCDF | 10 | 10 |
| 2,3,7,8-TCDD | 10 | 10 |
| SCS | | |
| Same as DCS | | |
| Surrogates added to samples | | |
| 37 Cl-2,3,7,8-TCDD | 10 | 10 |

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

## High Resolution Dioxins & Furans

| QC Category | Aqueous | Solid |
|---|---|---|
| | DXNFUR-HR-A | DXNFUR-HR-S |
| | Spike Level | |
| DCS | Aqueous (ng)/sample | Solid (ng)/sample |
| 2,3,7,8-TCDF | 0.20 | 0.20 |
| 1,2,3,7,8-PECDF | 0.50 | 0.50 |
| 1,2,3,4,7,8-HXCDF | 0.50 | 0.50 |
| 1,2,3,4,6,7,8-HPCDF | 0.50 | 0.50 |
| 1,2,3,4,5,6,7,8-OCDF | 1.0 | 1.0 |
| 2,3,7,8-TCDD | 0.20 | 0.20 |
| 1,2,3,7,8-PECDD | 0.50 | 0.50 |
| 1,2,3,4,7,8-HXCDD | 0.50 | 0.50 |
| 1,2,3,4,6,7,8-HPCDD | 0.50 | 0.50 |
| 1,2,3,4,5,6,7,8-OCDD | 1.0 | 1.0 |

SCS

Same as DCS

Surrogates added to samples

| | | |
|---|---|---|
| 37 Cl-2,3,7,8-TCDD | 1.0 | 1.0 |

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

## High Resolution TCDF & TCDD

| QC Category | Aqueous | Solid |
|---|---|---|
| | TCDD-HR-A | TCDD-HR-S |
| | Spike Level | |
| DCS | Aqueous (ng)/sample | Solid (ng)/sample |
| 2,3,7,8-TCDF | 0.20 | 0.20 |
| 2,3,7,8-TCDD | 0.20 | 0.20 |
| SCS | | |
| Same as DCS | | |
| Surrogates added to samples | | |
| 37 Cl-2,3,7,8-TCDD | 1.0 | 1.0 |

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

# APPENDIX A

## Part 3

## Laboratory Specific QC for Metals

## ICP Metals

| QC Category | Aqueous | Solid |
|---|---|---|
| | ICP-AD<br>ICP-AT<br>ICP-AR | ICP-S |

| DCS | Spike Level Aqueous (mg/L) | Spike Level Solid* (mg/Kg) |
|---|---|---|
| Aluminum | 2.0 | 200 |
| Antimony | 0.5 | 50 |
| Arsenic | 0.5 | 50 |
| Barium | 2.0 | 200 |
| Beryllium | 0.05 | 5.0 |
| Cadmium | 0.05 | 5.0 |
| Calcium | 100 | 10,000 |
| Chromium | 0.2 | 20 |
| Cobalt | 0.5 | 50 |
| Copper | 0.25 | 25 |
| Iron | 1.0 | 100 |
| Lead | 0.5 | 50 |
| Magnesium | 50 | 5,000 |
| Manganese | 0.5 | 50 |
| Nickel | 0.5 | 50 |
| Potassium | 50 | 5,000 |
| Silver | 0.05 | 5.0 |
| Sodium | 100 | 10,000 |
| Vanadium | 0.5 | 50 |
| Zinc | 0.5 | 50 |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample (wet weight)

## Graphite Furnace Metals--Mix 1

| DCS | QC Category: Aqueous | QC Category: Solid | Spike Level: Aqueous (mg/L) | Spike Level: Solid* (mg/Kg) |
|---|---|---|---|---|
| Arsenic | AS-FAA-AD<br>AS-FAA-AT<br>AS-FAA-AR | AS-FAA-S | 0.04 | 4 |
| Lead | PB-FAA-AD<br>PB-FAA-AT<br>PB-FAA-AR | PB-FAA-S | 0.02 | 2 |
| Selenium | SE-FAA-AD<br>SE-FAA-AT<br>SE-FAA-AR | SE-FAA-S | 0.01 | 1 |
| Thallium | TL-FAA-AD<br>TL-FAA-AT<br>TL-FAA-AR | TL-FAA-S | 0.05 | 5 |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample (wet weight)

## Graphite Furnace Metals--Mix 2

| | QC Category | | Spike Level | |
|---|---|---|---|---|
| DCS | Aqueous | Solid | Aqueous (mg/L) | Solid* (mg/Kg) |
| Cadmium | CD-FAA-AD<br>CD-FAA-AT<br>CD-FAA-AR | CD-FAA-S | 0.002 | 0.2 |
| Silver | AG-FAA-AD<br>AG-FAA-AT<br>AG-FAA-AR | AG-FAA-S | 0.002 | 0.2 |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample (wet weight)

## Mercury by Cold Vapor--AA

| QC Category | Aqueous | Solid |
|---|---|---|
| | HG-CVAA-AT | HG-CVAA-S |
| | Spike Level | |
| DCS | Aqueous (mg/L) | Solid* (mg/Kg) |
| Mercury | 0.001 | 0.5 |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 0.2 g sample (wet weight)

# APPENDIX A

## Part 4

## Laboratory QC for Conventional Inorganics

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| M-EQA-002 | 2.0 | May 15, 1989 |

## Conventional Inorganics

| Test/DCS Component | QC Category Aqueous | QC Category Solid | Spike Level* (ppm unless noted) |
|---|---|---|---|
| Ammonia as N | NH3-A | | 2.0 |
| Ammonia as N, Distilled | NH3-DIST-A | | 2.0 |
| Hardness as $CaCO_3$, Tit. | HARD-A | | 300 |
| Iodide | I-A | | 0.50 |
| Fluoride | F-A | | 10 |
| Fluoride, Distilled | F-DIST-A | | 10 |
| MBAS | MBAS-A | | 0.50 |
| Cyanide, Total | CN-A | | 0.10 |
| Cyanide, Free | CNW-A | | 0.10 |
| Dissolved Cyanide | CN-A | | 0.10 |
| Dissolved Sulfide | S-A | | 0.50 |
| Chemical Oxygen Demand | COD-A | COD-S | 125 |
| Chloride | CL-A | | 20 |
| Biochemical Oxygen Demand | BOD-A | | 200 |
| Bromide | BR-A | | 0.50 |
| Hexavalent Chromium | CR6-A | | 0.05 |
| Hexavalent Chromium | | CR6-S | 5 |
| Nitrate as N, Colorimetric | NO3-A | | 2.0 |
| Nitrite as N, Colorimetric | NO2-A | | 0.10 |
| Orthophosphate as P | PO4-A | | 0.25 |
| Phenolics | PHEN-A | | 0.20 |
| Purgeable Organic Carbon | POC-A | | 10 |
| Purgeable Organic Halogen | POX-A | | 100 ppb |
| Reactive Cyanide | | CNR-S | 100 |
| Reactive Sulfide | | SR-S | 100 |
| Residual Chlorine | CL2-A | | 2.0 |
| Sulfate | SO4-A | | 100 |
| Thiocyanate | SCN-A | | 3.0 |
| Thiosulfate | S2O3-A | | 3.0 |
| Total Alkalinity as $CaCO_3$ | ALK-A | | 190 |
| Total Dissolved Solids | TDS-A | | 1,400 |
| Total Kjeldahl Nitrogen as N | TKN-A | | 3.0 |
| Total Organic Carbon | TOC-A | TOC-S | 25 |
| Total Organic Halogen | TOX-A | TOX-S | 100 ppb |
| Total Phosphorus as P | TPHOS-A | | 0.25 |
| Total Solids | TS-A | | 1,400 |
| Total Sulfide | S-A | | 0.5 |
| Total Sulfide | | S-S | 100 |
| Total Suspended Solids | TSS-A | | 45 |
| pH | PH-A | | 9.0 units |
| Specific Conductance @ 25 C | COND-A | | 1,860 umhos/cm |
| Turbidity | TURB-A | | 1.5 NTU |

* Aqueous and solid samples spiked identically unless otherwise specified in a method SOP. Spike levels are nominal and may vary if spike solutions are obtained from outside vendors. Spike levels should be within the same order of magnitude as those specified.

## Ion Chromatography

| DCS | QC Category | Spike Level Aqueous (mg/L) |
|---|---|---|
| Fluoride | F-IC-A | 5 |
| Chloride | CL-IC-A | 100 |
| Nitrite as N | NO2-IC-A | 2 |
| Bromide | BR-IC-A | 20 |
| Nitrate as N | NO3-IC-A | 20 |
| Sulfate | SO4-IC-A | 200 |
| Orthophosphate as P | PO4-IC-A | 20 |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

**Method 413.1,2**
**Oil and Grease**

| DCS | Method | QC Category Aqueous | QC Category Solid | Spike Level Aqueous (mg/L) | Spike Level Solid* (mg/Kg) |
|---|---|---|---|---|---|
| Aromatic Ref. Oil (1) | Aromatic | O&G-AR-A | O&G-AR-S | 50 | 1000 |
| Reference Oil (2) | Gravimetric | O&G-G-A | O&G-G-~~A~~S | 50 | 1000 |
| Reference Oil (3) | IR | O&G-IR-A | O&G-IR-~~A~~S | 5 | 50+ |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

(1) As specifed in LP-RMA-1044 and LP-RMA-1045
(2) As specified in Method 413.1
(3) As specified in Method 413.2

* Assumes 50 g sample (wet weight).

+ Spike level may be adjusted to account for variation in instrument sensitivity. Alternate spike level must be within 5 times the reporting limit.

## Method 418.1
## Total Petroleum Hydrocarbons by IR

| QC Category | Aqueous | Solid |
|---|---|---|
| | TPH-IR-A | TPH-IR-S |
| | Spike Level | |
| DCS | Aqueous (mg/L) | Solid* (mg/Kg) |
| Reference Oil (per Method 418.1) | 5.0 | 50+ |

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 50 g sample (wet weight)

+ Spike level may be adjusted to account for variation in instrument sensitivity. Alternate spike level must be within 5 times the reporting limit.

# APPENDIX B

## Report Formats

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| M-EQA-002 | 2.0 | May 15, 1989 |

QC LOT ASSIGNMENT REPORT
Volatile Organics by GC

| Laboratory Sample Number | QC Matrix | QC Category | QC Lot Number (DCS) | QC Run Number (SCS/BLANK) |
|---|---|---|---|---|
| 003338-0001-SA | AQUEOUS | 601-A | 12 APR 89-A | 12 APR 89-A |
| 003338-0001-SA | AQUEOUS | 602-A | 12 APR 89-A | 12 APR 89-A |
| 003338-0001-MS | AQUEOUS | 601-A | 12 APR 89-A | 12 APR 89-A |
| 003338-0001-MS | AQUEOUS | 602-A | 12 APR 89-A | 12 APR 89-A |

DUPLICATE CONTROL SAMPLE REPORT
Volatile Organics by GC

| Analyte | Concentration Spiked | Measured DCS1 | Measured DCS2 | Measured AVG | Accuracy Average(%) DCS | Accuracy Average(%) Limits | Precision (RPD) DCS | Precision (RPD) Limits |
|---|---|---|---|---|---|---|---|---|
| Category: 601-A | | | | | | | | |
| Matrix: AQUEOUS | | | | | | | | |
| QC Lot: 12 APR 89-A | | | | | | | | |
| Concentration Units: ug/L | | | | | | | | |
| 1,1-Dichloroethane | 5.0 | 5.76 | 5.95 | 5.86 | 117 | 60-140 | 3.4 | 20 |
| Chloroform | 5.0 | 5.20 | 5.28 | 5.24 | 105 | 60-140 | 1.9 | 20 |
| Bromodichloromethane | 10 | 9.75 | 9.83 | 9.79 | 98 | 60-140 | 0.0 | 20 |
| Trichloroethene(-ylene) | 5.0 | 5.13 | 5.03 | 5.08 | 102 | 60-140 | 2.0 | 20 |
| Chlorobenzene | 5.0 | 5.03 | 5.02 | 5.02 | 100 | 60-140 | 1.0 | 20 |
| Category: 602-A | | | | | | | | |
| Matrix: AQUEOUS | | | | | | | | |
| QC Lot: 12 APR 89-A | | | | | | | | |
| Concentration Units: ug/L | | | | | | | | |
| Benzene | 5.0 | 5.30 | 5.50 | 5.40 | 108 | 77-123 | 3.7 | 20 |
| Toluene | 5.0 | 5.50 | ND | NC | NC | 77-123 | NC | 20 |
| Chlorobenzene | 5.0 | 5.50 | 6.00 | 5.75 | 115 | 77-123 | 8.7 | 20 |
| Ethyl benzene | 5.0 | 4.00 | 4.56 | 4.28 | 85 | 77-123 | 13 | 20 |
| o-Xylene | 5.0 | 3.00 | 3.00 | 3.00 | 60 | 77-123 | 0.0 | 20 |
| 1,3-Dichlorobenzene | 5.0 | 5.00 | 5.00 | 5.00 | 100 | 77-123 | 0.0 | 20 |

ND = Not detected.
NC = Not calculated; calculation not appropriate.
NA = Not applicable.

Calculations are performed before rounding to avoid round-off errors in calculated results.

SINGLE CONTROL SAMPLE REPORT
Volatile Organics by GC

| Analyte | Concentration Spiked | Concentration Measured | Accuracy(%) SCS | Accuracy(%) Limits |
|---|---|---|---|---|
| Category: 601-A | | | | |
| Matrix: AQUEOUS | | | | |
| QC Lot: 12 APR 89-A QC Run: 12 APR 89-A | | | | |
| Concentration Units: ug/L | | | | |
| Bromochloromethane | 30.0 | 31.4 | 105 | 20-160 |
| Category: 602-A | | | | |
| Matrix: AQUEOUS | | | | |
| QC Lot: 12 APR 89-A QC Run: 12 APR 89-A | | | | |
| Concentration Units: ug/L | | | | |
| a,a,a-Trifluorotoluene | 30.0 | 30.0 | 100 | 20-160 |

Calculations are performed before rounding to avoid round-off errors in calculated results.

METHOD BLANK REPORT
Volatile Organics by GC

Test: 601-A
Matrix: AQUEOUS
QC Lot: 12 APR 89-A QC Run: 12 APR 89-A

| Analyte | Result | Units | Reporting Limit |
|---|---|---|---|
| Chloromethane (Methyl chloride) | ND | ug/L | 5.0 |
| Bromomethane | ND | ug/L | 5.0 |
| Vinyl chloride | ND | ug/L | 1.0 |
| Chloroethane | ND | ug/L | 5.0 |
| Methylene chloride | ND | ug/L | 5.0 |
| 1,1-Dichloroethene | ND | ug/L | 0.50 |
| 1,1-Dichloroethane | ND | ug/L | 0.50 |
| 1,2-Dichloroethene (total) | ND | ug/L | 0.50 |
| 1,1,2-Trichloro-2,2,1-trifluoroethane | ND | ug/L | 1.0 |
| 1,2-Dichloroethane | ND | ug/L | 1.0 |
| 1,1,1-Trichloroethane | ND | ug/L | 0.50 |
| Carbon tetrachloride | ND | ug/L | 0.50 |
| Bromodichloromethane | ND | ug/L | 1.0 |
| 1,2-Dichloropropane | ND | ug/L | 1.0 |
| trans-1,3-Dichloropropene | ND | ug/L | 1.0 |
| Trichloroethene(-ylene) | ND | ug/L | 0.50 |
| Dibromochloromethane | ND | ug/L | 1.0 |
| cis-1,3-Dichloropropene | ND | ug/L | 2.0 |
| 1,1,2-Trichloroethane | ND | ug/L | 1.0 |
| EDB (1,2-Dibromoethane) | ND | ug/L | 2.0 |
| Bromoform | ND | ug/L | 5.0 |
| 1,1,2,2-Tetrachloroethane | ND | ug/L | 1.0 |
| Tetrachloroethene | ND | ug/L | 0.50 |

METHOD BLANK REPORT
Volatile Organics by GC

| Analyte | Result | Units | Reporting Limit |
|---|---|---|---|
| Test: 602-AP | | | |
| Matrix: AQUEOUS | | | |
| QC Lot: 12 APR 89-A QC Run: 12 APR 89-A | | | |
| Benzene | ND | ug/L | 0.50 |
| Toluene | ND | ug/L | 0.50 |
| Chlorobenzene | ND | ug/L | 0.50 |
| Ethyl benzene | ND | ug/L | 0.50 |
| Xylenes (total) | ND | ug/L | 1.0 |
| 1,3-Dichlorobenzene | ND | ug/L | 0.50 |
| 1,4-Dichlorobenzene | ND | ug/L | 0.50 |
| 1,2-Dichlorobenzene | ND | ug/L | 0.50 |

STANDARD
OPERATING
PROCEDURE

| | | |
|---|---|---|
| Subject or Title: **Corrective Action Report** | | Page 1 of 7 |
| SOP No.: LP-RMA-0022 | Revision No.: 1 | Effective Date: 8/9/88 |
| Supersedes: | | |

1.0 Purpose

1.1 To document and communicate quality control problems encountered during analysis.

1.2 To initiate an investigation of quality control problems.

1.3 To implement corrective actions designed to ensure that quality control problems will be minimized in future analyses.

1.4 To allow follow-up of quality control problems at a later date.

2.0 Policies

2.1 All errors, deficiencies, and out-of-control situations encountered with analytical standards, client samples and laboratory-generated quality control samples must be documented on the warning/out-of-control form contained in this S.O.P.

2.2 An investigation must be initiated immediately upon discovering a problem may exist.

2.3 Upon completing the investigation, corrective actions are undertaken by, and communicated to, the appropriate staff members in the department(s) in which the problem occurred.

| | |
|---|---|
| Prepared by: Gary Tolf | Date: 8/9/88 |
| Management Approval: Timothy Carlberg | Date: 8/15/88 |
| QA Officer Approval: Gary Tolf | Date: 8/15/88 |

2.4 The warning/out-of-control form must be signed and dated by the staff member(s) investigating the problem, a supervisor within the department that encountered the problem, and a member of the quality assurance department.

2.5 A copy of the signed form must be placed with the raw data and project folders of the affected samples, and provided to the Quality Assurance Department for archiving (it is highly recommended that the manager of the affected department also maintain a copy for communication and follow-up within the department), as described in 5.2 and 5.3.

3.0 Safety Issues

None

4.0 Procedure

4.1 Identification of Out-of-Control Situation

4.1.1 Situations which alert laboratory personnel that an out-of-control situation exists and corrective action may be necessary include, but are not limited to:

- QC data are outside the warning or acceptable limits for precision and accuracy,
- Blanks, LCS or SCS control samples contain contaminants above acceptable levels,
- There are unusual changes in detection limits,
- Deficiencies are detected by the Quality Assurance department during internal or external audits or from the results of performance evaluation samples,
- Inquiries concerning data quality are received from clients or other Enseco facilities.

4.2 Documentation of the Out-of-Control Situation

4.2.1 Briefly, but informatively describe the problem encountered on the warning/out-of-control form contained in this S.O.P. For example:

"The percent recovery for the Cyanide LCS1, QC lot 880915A, was 60%. The accuracy control limits are 75% - 125%".

**Rather than,**

"The LCS was out".

"The precision for Phenol in the BNA LCS, QC lot 880915A, was 66.7. The precision control limit is 42.0".

**Rather than,**

"The Phenol precision was no good".

"Aldrin was found in the OCP SCS, QC lot 880915A".

**Rather than,**

"The SCS was contaminated".

4.2.2 List all samples affected by the problem. Specify whether the problem affected only the laboratory quality control samples, the client samples, or both.

4.3 Document the investigation of the problem.

4.3.1 List each step of the analytical procedure which was investigated, and the result/conclusion. It is acceptable to document that no definitive conclusion can be drawn, however, specific follow-up investigations will then be required as a corrective action to ensure the problem does not persist.

4.3.2 List any additional samples and/or QC lots found during the investigation to be affected by the problem.

4.4 Document the corrective actions taken to resolve the problem.

4.4.1 List any immediate steps taken to resolve the problem with the affected samples, such as repreparation or reanalysis.

4.4.2 List the actions taken to ensure that the problem does not affect future analyses. Examples include (but are not limited to): Maintenance of laboratory equipment, specific changes in standard operating procedures, additional training of personnel, and the purchasing of reagents or standards from new sources/vendors.

4.5 Communicate the resolution of the problem and any necessary follow-up to appropriate staff members.

4.5.1 The warning/out-of-control form is signed and dated by the staff member investigating the problem.

4.5.2 The warning/out-of-control form is signed and dated by a supervisor within the department that encountered the problem.

4.5.3 The warning/out-of-control form is signed and dated by a member of the Quality Assurance Department.

4.5.4 The warning/out-of-control form is signed by a client manager (optional).

4.5.5 A copy of the warning/out-of-control form is:

- Placed with the raw data <u>and</u> project folders of any affected samples,
- Provided to the Quality Assurance Department for archiving and,
- Provided to the client manager when requested.

4.5.6 The problem and it's resolution is communicated to additional staff members in the affected department(s) at the next scheduled group meeting.

5.0 Responsibilities

5.1 The staff member (technician, analyst/chemist, supervisor, quality assurance personnel, client manager, technical director, et al) who encounters an error, deficiency or out-of-control situation is responsible for immediately documenting the problem as described in 4.2 above.

5.2 The supervisor/manager in the department where the problem occurred is responsible for:

- The timely investigation of the problem,
- The documentation of corrective actions,
- Notification to the Quality Assurance Department and affected client manager(s),
- Ensuring corrective actions are implemented and appropriate follow-up in undertaken,
- Ensuring that a copy of the warning/out-of-control form is placed with the raw data and provided to the appropriate personnel as described in 4.5.5 and 4.5.6 above and,
- Communicating the problem and its resolution to additional staff members in their department.

5.3 The Quality Assurance Department is responsible for:

- The timely acknowledgement of any warning/out-of-control forms after completion, archival of the signed forms in the project folder and QA files, and
- The timely approval of any S.O.P. changes necessitated by corrective actions.

5.4 The client manager is responsible for communicating to the client the affect a problem may have on the analysis of their samples (verbally and/or in the final report).

6.0 Comments

6.1 Laboratory supervisors and managers should encourage the use of the warning/out-of-control form by:

- Reiterating that Quality Control is the responsibility of all staff members and,

- The prompt addressing of major and minor problems brought to their attention.

WARNING/OUT-OF-CONTROL FORM

QC Lot _____

Associated Samples _____

PROBLEM: (Briefly decribe problem)

Analyst:
Date:

---

RESULTS/CONCLUSIONS of the Investigation:

Analyst:
Supervisor:
Date:

---

CORRECTIVE ACTIONS (including follow-up)

Supervisor:
QA Approval:
Date:

WARNING/OUT-OF-CONTROL FORM

QC Lot _____

Associated Samples _____

PROBLEM: (Briefly decribe problem)

Analyst:
Date:

- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -

RESULTS/CONCLUSIONS of the Investigation:

Analyst:
Supervisor:
Date:

- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -

CORRECTIVE ACTIONS (including follow-up)

Supervisor:
QA Approval:
Date:

STANDARD
OPERATING
PROCEDURE

| Subject or Title: CLP SAMPLE RECEIPT AND LOGGING | | Page 1 of 6 |
|---|---|---|
| SOP No.: LP-RMA-5001 | Revision No.: Original | Effective Date: 10/1/90 |
| Supersedes: None | | |

ENSECO PROPRIETARY INFORMATION STATEMENT

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1. Purpose:

   To assure traceability of the samples while in possession of the laboratory and to create analyses records in the laboratory computer for notification to lab analysts.

2. Policies:

   To receive and handle samples under the Chain-of-Custody procedures described in the Statement of Work for Organic Analysis, Exhibit F.

3. Safety: Not Applicable

| Prepared by: Jan Redenbarger | Date: 11/7/90 |
|---|---|
| Management Approval: Gary Toft | Date: 11-12-90 |
| QA Officer Approval: Jan Redenbarger | Date: 11/7/90 |

4. Procedures

4.1 Check cooler for radiation

4.2 Check condition of shipping container upon receipt

4.3 Check custody seals: intact/not intact

4.4 Check for presence or absence of EPA Chain-of-Custody Traffic Report or SAS packing list.

4.5 Verify case number with RAS or SAS booking. (Bookings are scheduled with RMAL by Sample Management Office (SMO)). If case number is not scheduled with RMAL call SMO (environmental program coordinator by region) for clarification.

4.6 Use CLP or EPA sample receipt checklist (See figures 1,2, and 3).

4.7 Unpack cooler

4.7.1 Check condition of samples.

4.7.2 Verify Chain of Custody, Traffic Report or SAS packing list, tags with sample labels.

4.8 Air bill or air bill sticker numbers should be on Chain-of-Custody Traffic Report/SAS packing list, tags should also be on Chain-of-Custody.

4.9 Sign, date, and time Chain-of-Custody, Traffic Report/SAS packing list, and air bill if no discrepancies are found.

4.9.1 If discrepancies are found contact SMO. Contact the Environmental Program Coordinator by region. Document agreement among the forms and any discrepancies found on sample receipt checklist with date, time, and initials.

4.10 Check paper work (Chain-of-Custody, Traffic Report or packing list for designated QC (matrix spike and duplicate). If QC is not indicated sample custodian will select QC.

4.11 Select SDG number

The SDG number is the lowest sample number in the first group of samples received under the SDG. In determining lowest sample number, consider both alphabetic and numeric characters in the sample number.

A Sample with a lower number could arrive in a later sample shipment so be aware that the SDG number may not necessarily be the lowest sample number. The SDG number appears on all data deliverables.

4.12 Assign QC number (EPA Organic), pull RMA's project number through LIMS by client (SMO*) return (see figure 4).

- Region
- Program
- Project
- Add Project

- or duplicate from project with existing region (by using the Log-in Book for EPA Organic).

Assign QC number (CLP Organic), pull RMA's project number through LIMS (by existing client in the right program). PA will set up all new clients in LIMS.

4.13 Stamp information on project folder (see figure 5)

- QC number
- Contact number
- Controller
- Turnaround
- Region
- Price
- Parameters
- Others
- No type
- Date received
- Date due

(Note all rush projects on folder)

4.14 Label samples with project number, plus EPA or CLP initials so that samples are located in the correct area of the cooler.
Example: 011829 EPA or 011829 CLP

4.15 Log-in to LIMS by matrix (aqueous or soil)

Each matrix is a separate project for EPA or CLP.

4.16 Print out group code (EPA)
- sample list

Print out group code (CLP)
- sample list
- acceptance letter

4.17 Take picture of samples for project folder. (Take separate picture of broken samples).

4.18 Complete project folder (see figure 6).
- SDG package which contains:
  - SDG cover sheet
  - original Traffic Report/SAS packing list
  - mail to SMO

4.18.1 If project has LIMS print out
- SDG cover sheet
- copy Traffic Report/SAS packing list
- Chain-of-Custody
- air bill
- check list
- picture
- (CLP) cross-reference of samples
- (CLP) acceptance letter
- write project number, case number, number of sample matrix and parameters on (EPA) Chain-of-Custody. Special instruction in LIMS, if any.
- Special instructions in LIMS, if any.

4.19 Record projects in EPA Organic or CLP Organic Log Book

4.20 Give project folder to PA

PA will review project folders and put away in designated area.

4.21 Put sample away in walk-in cooler or refrigerator; volatile samples are stored in separate refrigerators by matrix.

4.22 Write sample project number and shelf number on board.

5. Responsibilities:

Sample Custodian-Responsible for receiving the EPA samples (logging, handling, and storage).

6. Definitions:

6.1 CLP - Contract Laboratory Program

6.2 EPA - Environmental Protection Agency

6.3 LIMS - Laboratory Information Management System, commercial and custom software programs designed to perform calculations, check results, generate reports, and ensure data integrity and security.

6.4 PA - Program Administrator

6.5 SAS - Special Analytical Services

6.6 Sample - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

6.7 Sample Delivery Group (SDG) - a unit within a single Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, which ever occurs first:

- Case; or
- Each 20 field samples within a Case; or
- Each 14-day calendar period during which field samples in a Case or SDG.

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.

6.8 Sample Number (EPA Sample Number) - a unique identification number designated by EPA for each sample. The EPA sample number appears on the sample Traffic Report which documents information on that sample.

6.9 Special instructions - typed instructions in LIMS to the operations groups and analysts that are necessary to complete the work and can not by indicated by using one of the computer tests.

6.10 Traffic Report (TR) - an EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which documents sample condition and receipt by the laboratory.

Figure 1

EPA SAMPLE RECEIPT CHECKLIST

| YES | NO | |
|---|---|---|
| ____ | ____ | 1a. Chain of Custody present? |
| ____ | ____ | 1b. Chain of Custody filled out correctly? |
| ____ | ____ | 2a. Traffic Reports or Packing List present? |
| ____ | ____ | 2b. Traffic Reports or Packing List filled out correctly? |
| ____ | ____ | 3a. Tags present on sample containers? |
| ____ | ____ | 3b. Tags filled out correctly? |
| ____ | ____ | 4. Custody seals present on sample cooler? |
| ____ | ____ | 5. Custody seals present on sample containers? |
| ____ | ____ | 6. Sample labels present on sample containers? |
| ____ | ____ | 7. Samples intact? |
| ____ | ____ | 8. Samples cold upon receipt? |
| ____ | ____ | 9. Chain of Custody, Traffic Reports (or Packing List), Tag, and Label agree? |
| ____ | ____ | 10. Number and type of samples and parameters agree with booking? |

If NO is checked, note action taken:

If SMO is contacted, note date and person contacted:

Checklist completed by:

Name: _____

Date: _____

Figure 2



# CLP SAMPLE RECEIPT CHECKLIST

| YES | NO | | |
|---|---|---|---|
| _____ | _____ | 1a. | Chain of Custody present? |
| _____ | _____ | 1b. | Chain of Custody filled out correctly? |
| _____ | _____ | 2. | Custody seals present on sample cooler? |
| _____ | _____ | 3. | Custody seals present on sample containers? |
| _____ | _____ | 4. | Sample labels present on sample containers? |
| _____ | _____ | 5. | Samples intact? |
| _____ | _____ | 6. | Samples cold upon receipt? |
| _____ | _____ | 7. | Chain of Custody and Label agree? |

If NO is checked, note action taken:

Checklist completed by:

Name: _____

Date: _____

Figure 3

**RMAL Industrial Sample Checklist** Revision 2.0 March, 1989

Project #: _____ Date/Time Received: _____

mpany Name & Sampling Site : _____

*Cooler # (s): _____ _____ _____ _____

Temperatures: _____ _____ _____ _____

* Place copy of airbill inside all non-RMAL coolers. Describe here.

| **UNPACKING & LABELING CHECK POINTS:** | Y | N | INITIALS |
|---|---|---|---|
| 1. Radiation Checked; (record reading if > 15 mr): | ___ | ___ | _____ |
| 2. Cooler seals Intact: | ___ | ___ | _____ |
| 3. Chain of Custody Present: | ___ | ___ | _____ |
| 4. Bottles broken or leaking (comment if Y):<br>**- photograph broken bottles-** | ___ | ___ | _____ |
| 5. Containers labeled (comment if N): | ___ | ___ | _____ |
| 6. Aqueous: ___ Soil: ___ Waste: ___ | | | _____ |
| 7. Chain of Custody signed with date, time & lab: | ___ | ___ | _____ |
| . CoC agrees with bottle count (comment if N): | ___ | ___ | _____ |
| 9. CoC agrees with labels (comment if N): | ___ | ___ | _____ |
| 10. VOA samples filled completely (comment if N): | ___ | ___ | _____ |
| 11. VOA samples preserved: | ___ | ___ | _____ |
| 12. Sediment present in "D" bottles: | ___ | ___ | _____ |
| 13. Short holding times: | ___ | ___ | _____ |
| 14. Matrix QC verified: | ___ | ___ | _____ |
| 15. Multi phase samples present (comment if Y):<br>**- photograph multiphase samples -** | ___ | ___ | _____ |
| 16. Clear picture taken & labeled: | ___ | ___ | _____ |
| 17. Clear picture of sub samples taken: | ___ | ___ | _____ |

18. Comments; include action taken to resolve discrepancies / problems. Include a hardcopy of VAX mail or extra paper if more space is needed.

_____

_____

_____

_____ Sign & Date: _____

Figure 4

```
Log-in

Limsmenu
Project/Data Maintenance
Screen Client
    EPA Organic (SMO*)
    CLP Organic
    Client (SMO*)    Return
    Soft Key (F-19)    Program Return

    Shift ~ Return
    (Program Search)
    Soft Key    Project    (F19)    Return
    Soft Key    Add Project    (F11)    Return

    Name Case #    SDG #    Final SDG #
    Return
    Type None
    Return
    Case #    SDG #    SAS #   Level
    Return

Description:  (Example) two waters, full HSL.
    Return
Soft key (Page 2) F14
Calculate holding times from collection dates?   N
Return
Actual start date
    Return
Receipt    date    time
    Return
         Do
More functions (F17)
Soft key    accept delivery    (F11)
    Do
    F-10
    Group Code    return
    Add Group Code (F-11)
    Description Example: samples 1-2 (MS,SD)
         Do

    Add test    (F12)
         Example (625-RAS-A)    Do
         (F12)  (624-RAS-A)    Do
         (F12)  (608-RAS-A)    Do
    (F10)
    Sample Return
    Add Sample (F11)
    Aliquot (SA) Sample     Return    Group Code A    Return
    Client Description Example (MHT58)    Return
    Collection Date:  "Return Time"    Return
    Receipt Date:  "Return Time"    Return
    Lab Description:  Example (3-11, 2-12, and 3-12)
```

Figure 4 (con't)

```
    Return
    Do
    For QC
    Duplicate Sample    (F14)
Ctrl U (0001) Aliquot (MS) Return    Group Code (A)    Return
    Do
    Duplicate Sample    (F14)
Ctrl U (to sample number indicated for QC)
    Return
Aliquot (MS) Matrix Spike
Group Code (if necessary)
Client Description    Return
    Do
    Duplicate Sample    (F14)
Ctrl U
    Return
Aliquot (SD) matrix spike duplicate
         Do
    Duplicate Sample    (F14)
Change
Aliquot (SA)    Return
Client Description
Collection Date    Return    Time    Return
Lab Description

         Do
    F10
    Group Code    Return
    Ctrl R    No-Yes    Return
    (copy of Group Code)
    F10 Samples
    Ctrl R
    (copy of sample list)
    for project folder
```

Figure 4 (con't)

| | Test ID |
|---|---|
| GC/MS Analyses (Aqueous) | |
| CLP/HSL Semivolatiles | 625-RAS-A |
| CLP/HSL Volatiles Library Search | 624-RAS-A |
| GC/MS Analyses (Solids) | |
| CLP/HSL Semivolatiles | 8270-RAS-S-S |
| CLP/HSL Volatiles | 8240-RAS-S |
| GC Analyses (Aqueous) | |
| CLP/HSL Organochlorine Pesticides/PCB | 608-RAS-A |
| GC Analyses (Solids) | |
| CLP/HSL Organochlorine Pesticides/PCB | 8080-RAS-S-S |
| Organic Prep Analyses | |
| Percent Moisture* | Percent Water-OMS-S |
| pH* | pH-MET-RAS-S |

* These tests do not apply to MS and MSD samples.

NOTE: Holding times for CLP projects are normally calculated from the received date.

Figure 5

Case/SAS No: _____ NO. & TYPE _____

Contract No: _____ Confirmed: _____

Controller: _____ Region: _____ DATE REC'D _____

Turnaround: _____ Price: _____

Parameters: Metals _____ CN _____ DATE DUE _____

Others: _____

_____

RAS Invoice No. _____ SAS Invoice No: _____

Figure 6

SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER SHEET

Lab Name: Rocky Mountain Analytical Laboratory  Contract No.: _____

Lab Code: ENSECO  Case No.: _____  SAS No.: _____

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____  Sample Receipt Date: ___/___/___
(Lowest EPA Sample Number in first shipment of samples received under SDG)  (MM/DD/YY)

Last Sample in SDG: _____  Sample Receipt Date: ___/___/___
(Highest EPA Sample Number in last shipment of samples received under SDG)  (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

| | | | |
|---|---|---|---|
| 1 | _____ | 11 | _____ |
| 2 | _____ | 12 | _____ |
| 3 | _____ | 13 | _____ |
| 4 | _____ | 14 | _____ |
| 5 | _____ | 15 | _____ |
| 6 | _____ | 16 | _____ |
| 7 | _____ | 17 | _____ |
| 8 | _____ | 18 | _____ |
| 9 | _____ | 19 | _____ |
| 10 | _____ | 20 | _____ |

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____  _____
Sample Custodian  Date

STANDARD
OPERATING
PROCEDURE

| | |
|---|---|
| Subject or Title: SAMPLE STORAGE | Page 1 of 3 |

| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| LP-RMA-5002 | 1.0 | 06/25/91 |

Supersedes:
Original

ENSECO PROPRIETARY INFORMATION STATEMENT

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1. Purpose:

To effectively organize and maintain completed samples until they are either disposed of or returned to the client.

2. Policies:

Sufficient refrigerator space to maintain unused EPA sample volume for 60 days after data submission and sample extracts for 365 days after data submission. Samples must be stored in an atmosphere demonstrated to be

| | |
|---|---|
| Prepared by: QA Department | Date: 06/25/91 |
| Management Approval: Tina Kigan | Date: 7-1-91 |
| QA Officer Approval: Jan Redenbarger | Date: 7-1-91 |

free from all potential contaminants. Volatile samples must be stored in a refrigerator used only for storage of volatile samples from this contract. Refer to the attached diagram for the storage locations for both organic and inorganic samples.

3. Safety:

Not applicable.

4. Procedure:

4.1 Check and document physical condition of sample.

4.2 Verify documentation and parameter assignment.

4.3 Log into LIMS (See SOP: LP-RMA-5001).

4.4 Send acknowledgement letter to client or SDG package.

4.5 Store sample according to preservation guidelines.

4.6 Transfer sample to lab with proper documentation.

4.7 Document analytical work.

4.8 Return unused sample to sample control.

4.9 Samples are kept in Archive area.

4.10 Return sample to client or arrange for sample disposal.

5. Responsibilities:

Sample custodian is responsible for maintaining and disposing of completed samples.

6. Definitions

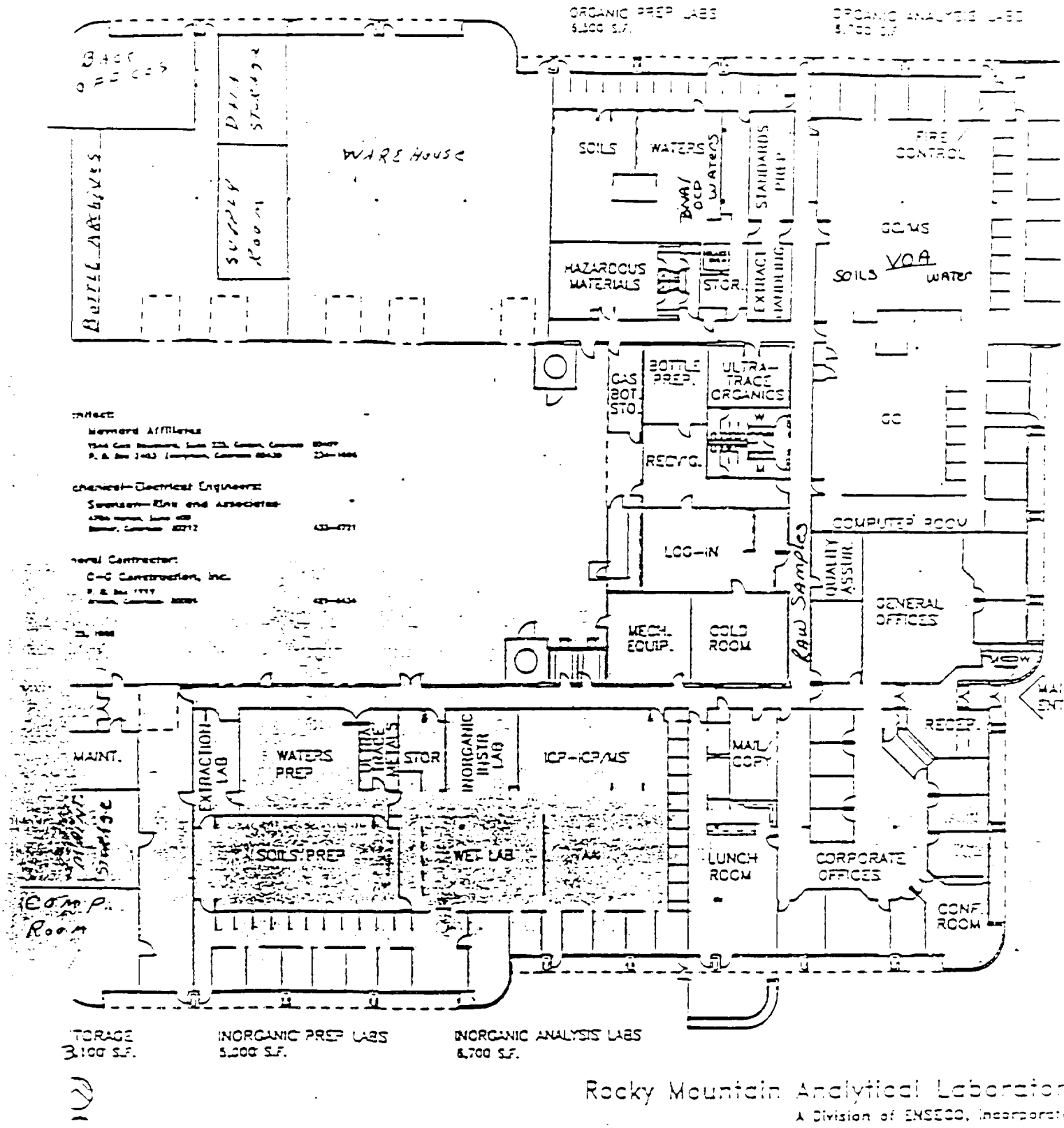
6.1 LIMS - Laboratory Information Management System, commercial and custom software programs designed to perform calculations, check results, generate reports, and ensure data integrity and security.

6.2 Sample - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

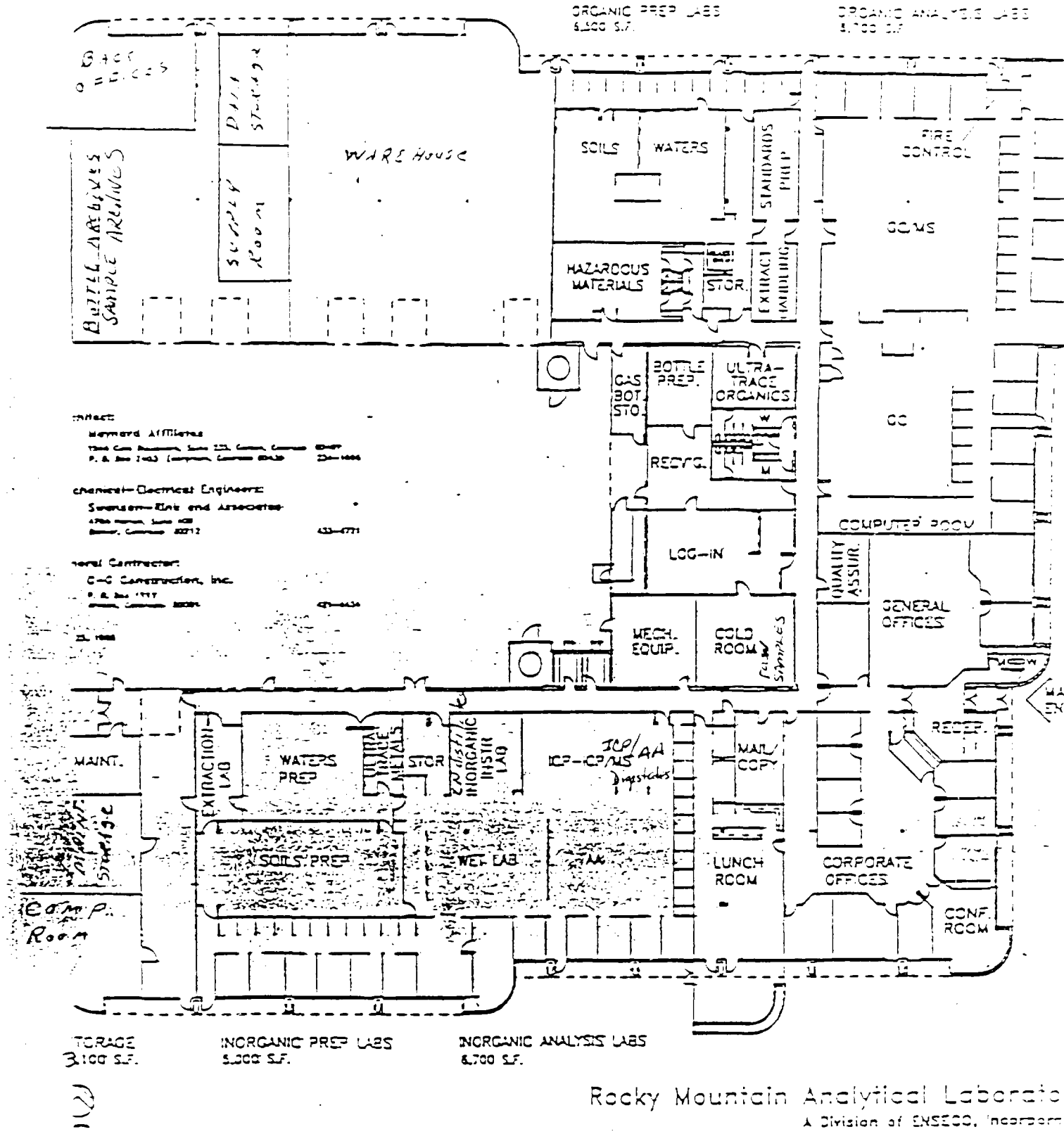
6.3 Sample Delivery Group (SDG) - a unit within a single Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, which ever occurs first:

- Case; or
- Each 20 field samples within a Case; or
- Each 14-day calendar period during which field samples in a Case or SDG.

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.




EPA Sample Storage Areas
For Organic Analyses




EPA Sample Storage Area
for Inorganic Analysis

STANDARD
OPERATING
PROCEDURE

| Subject or Title: BUILDING SECURITY | | Page 1 of 3 |
|---|---|---|
| SOP No.: LP-RMA-5003 | Revision No.: Original | Effective Date: 09/28/90 |
| Supersedes: | | |

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1. Purpose:

   The purpose of building security is to guarantee data security and confidentiality for the client as well as providing analytical data which is legally defensible.

2. Policies:

   RMAL's security policy includes controlled access to the building, testing areas and data files, confidentially agreements with all personnel. identification badges for all personnel, electronic security and fire alarm systems. All visitors are also assigned visitor badges and are accompanied by RMAL staff during their stay in the facility.

| Prepared by: Jan Rodenburgen | Date: 11/7/90 |
|---|---|
| Management Approval: Gary Tok | Date: 11-12-90 |
| QA Officer Approval: Jan Rodenburgen | Date: 11/7/90 |

3. Safety Issues: Not Applicable

4. Procedure:

4.1 Building Security

4.1.1 All exterior doors to the facility will remain locked at all times with the exception of the front entrance.

4.1.2 During the hours of 7:00 a.m. to 6:00 p.m., the front entrance or main reception area is controlled by the receptionist and secured by locked entries. The alarm system is not activated during this time period.

4.1.3 The last employee to leave the facility must page the building to ensure he or she is the last person in the facility, they then call the alarm company to identify themself by name and code. The alarm system is activated, during this time period to prevent all other exterior doors from being useable, including sample receiving and the patio doors.

4.1.4 Sample receiving during the hours of 6:00 p.m.to 7:00 a.m. is permitted only with the assistance by a member of sample control.

4.2 Personnel Identification

4.2.1 All employees and visitors are required to wear security badges at all times while on the premises of all ENSECO divisions.

4.2.2 The personnel administrator is responsible for issuing a picture I.D. badge to an employee on the employee's first day of employment. Each employee is responsible for his/her badge. Additionally, each employee will be required to sign a "Confidentiality Agreement" which is included in the employee's personnel file.

4.2.3 The receptionist is responsible for issuing a badge to each visitor to the facility. Visitors must request a badge from the front office of the division they visit, sign the visitor log and must be accompanied by an ENSECO employee before access to any building will be allowed.

4.3 Building Alarm System

4.3.1 Each employee will receive a copy of the security manual, personal security code and security training at the time of their orientation provided by a member of the Human Resources department. The procedure is confidential information and can only be obtained from the Personnel Department.

5. Responsibilities:

5.1 It is the responsibility of each employee to maintain confidentiality of all clients data.

5.2 The Personnel Department is responsible for issuing employee identification badges and having signed "Confidentiality Agreements" in each employee's personnel file.

5.3 The receptionist is responsible for issuing visitor badges and for visitor sign-in during normal business hours.

5.4 Employees escorting visitors are responsible for ensuring that visitation procedures are followed and that data confidentiality has not been compromised.

6. Comments:

Enseco

STANDARD OPERATING PROCEDURE

Subject or Title: **Bottle Preparation and Cooler Shipment**



| SOP No.: | Revision No.: | Effective Date: |
|---|---|---|
| LP-RMA-0056 | 1.0 | July 23, 1990 |

Supersedes: Original

1. Purpose

   To assure that the proper procedures are followed when preparing sample bottles and coolers for shipment.

2. Policies

   The sample bottles are prepared according to ENSECO QAPP Revision 3.3. Coolers are shipped by either UPS or overnight courier requirements.

3. Safety Issues

   Due to the use of both base and acid as preservatives, safety glasses should be worn along with lab coats and gloves.

4. Procedure

   4.1 The client defines the scope of work to a Program Administrator and the PA completes a bottle order sheet which is given to the sample control technician (see example sheet #7.)

   4.2 The sample technician fills the bottle order using the appropriate bottles and preservatives as stated on the attached guideline sheet (see example sheet #1.) The bottles the laboratory provides for sampling and analysis are purchased from I-Chem. These bottles are provided with certificates of analysis and assigned QC lots (see example sheet #2.) The appropriate QC lots are noted on each bottle order sheet and on each glass bottle.

   4.3 The VOA trip blanks are labeled with the date, the sample technician's name and the words "trip blank."

| Prepared by: | Date: |
|---|---|
| Bob Musil | July 23, 1990 |
| Management Approval: | Date: |
| [signature] | July 25, 1990 |
| QA Officer Approval: | Date: |
| [signature] | 7/25/90 |

4.4 The following paperwork is enclosed with the cooler(s):

4.4.1. Sample labels for each bottle sent

4.4.2 Chain-of-custody forms

4.4.3. Cooler labels to be used for shipping and returning cooler(s)

4.4.4 Cooler seals

4.4.5 Guideline sheet for sample bottles and preservatives

4.5 The sample bottles are securely packed in the correct number of shipping containers with styrofoam. The styrofoam sleeves are placed around the bottles and styrofoam sheets are layered on top of the bottles before taping the cooler(s) shut.

4.6 The cooler(s) are taped shut with strapping tape and labeled with recipient's address.

4.7 Cooler seals are placed over the lid of each cooler(s).

4.8 Prepare shipping bill according to the client's request, whether it be UPS or Federal Express, as follows:

4.8.1. Federal Express

4.8.1.1 An airbill must be completed with the sample control technician's name, date and telephone number along with the recipient's name, address and telephone number (see example sheet #3.)

4.8.1.2 Confirm whether the charges are going to bill sender or bill recipient's Federal Express number and check the appropriate box.

4.8.1.3 Check the appropriate box for the service requested e.g. priority 1, standard air, deliver weekday or Saturday delivery.

4.8.1.4 Annotate the number of coolers being shipped, the total weight and declared value, if any.

4.8.1.5 The multiple shipment labels (see example sheet #4) must be used if shipping more than one cooler. Document the number of coolers being shipped, the date, and the number of the master airbill which is located at the top of the original airbill.

4.8.1.6 Place one bar code label on each cooler, excluding the cooler which will be shipped with the master airbill. A copy of the the bar code list is placed on the space provided on the back of the master airbill.

4.8.2 UPS (see example sheet # 5)

4.8.2.1 Address UPS pickup record with the client's name and address.

4.8.2.2 A blank space is provided for the zone number on the pick up record. The zone number is determined by using the first three digits of the zip code e.g. 80000. Use the zone chart (see example sheet #6) to determine the UPS zone.

5. Responsibility

Bottle preparation and cooler shipment is the responsibility of the sample control technicians. Initiating the bottle order form(s) is the responsibility of the Program Administrator.

6. Comments

It is important to pay close attention to which preservative (acid or base) is being added to each type of bottle.

7. Definitions

None.

Example Sheet #1

Enseco

## GUIDELINES FOR SAMPLE BOTTLES AND PRESERVATIVES

.E: In addition to the preservatives stated below, all samples should be cooled to 4°C

| Number | Parameters | Container | Preservatives |
|---|---|---|---|
| LIQUID: | Aqueous | | |
| 1 | Alkalinity, BOD, Chloride, Color, Res. Chlorine, pH, Chromium (VI), Conductance, Fluoride, Nitrite, MBAS, Ortho-Phos., Solids, Sulfate, Sulfite, Turbidity | 32oz poly (WM) | None |
| 2 | Ammonia, COD, Nitrate, TKN, TON Nitrate + Nitrite, Total Phos., TOC, Phenolics | 16oz glass (BR) | 2 mL 50% Sulfuric Acid |
| 3 | TPH, Oil & Grease | 32oz glass (BR) | 4 mL 50% Sulfuric Acid |
| 4 | Metals, Hardness | 16oz poly (WM) | 10 mL 20% Nitric Acid |
| 5 | Gross Alpha, Gross Beta, Uranium, Radium 226, Radium 228 | Two 32oz poly (BR) | 10 mL 20% Nitric Acid |
| 6 | Total and/or Free Cyanide | 8oz poly (WM) | 2 mL 50% Sodium Hydroxide |
| 7 | Sulfide | 8oz poly (WM) | 1 mL 1N Zinc Acetate + 1 mL 50% Sodium Hydroxide |
| 8 | Fecal or Total Coliform (use 2 bottles if both required) | 4 1/2 oz. poly, sterilized | None |
| 10 | THM | Three 40 mL glass vials | 100 uL Sodium Thiosulfate |
| 11 | VOA, Purgeable Organics | Three 40 mL glass vials | 200 uL 50% Hydrochloric Acid |
| 12 | Base Neutral/Acid Compounds | Two 32oz glass(BR) | None |
| 13 | Pesticides, PCBs | Two 32oz glass(BR) | None |
| 14 | Herbicides | 1 32oz glass(BR) | None |
| 15 | TOX--Single: | 8oz glass (BR) (amber) | 1 mL 50% Sulfuric Acid |
| | --Quad: | Four 8oz glass OR 1 32oz glass (amber) | 1 mL 50% Sulfuric Acid<br>4 mL 50% Sulfuric Acid |
| 20, 21 | Bulk water analysis | 1/2 Gal OR 1 Gal glass (WM) | None |
| .OLID: | Soils, Sludges, Wastes | | |
| 30 | Organics,TPH,Metals,RAD, Oil & Grease | 16 oz glass (WM) | None |
| 31 | Wet Chem not listed for #30 | 8 oz glass (WM) | None |
| [illegible] | [illegible] | 4 oz glass (WM) | None |

CHEM

CHEM RESEARCH

Example Sheet #2

## CERTIFICATE OF ANALYSIS

Analysis of Lot Number BR[illegible]

The following compounds were analyzed for but not detected, or detected at a concentration of less than 5 micrograms/liter:

| | | |
|---|---|---|
| Chloromethane | Bromomethane | Vinyl Chloride |
| Chloroethane | Methylene Chloride | Acetone |
| Carbon Disulfide | 1,1-Dichloroethane | 1,1-Dichloroethene |
| Chloroform | 1,2-Dichloroethane | trans-1,2-Dichloroethene |
| 2-Butanone | 1,1,1-Trichloroethane | Carbon tetrachloride |
| Vinyl acetate | Bromodichloromethane | 1,2-Dichloropropane |
| Trichloroethene | Dibromochloromethane | 1,1,2-Trichloroethane |
| Benzene | Bromoform | cis-1,1-Dichloroethene |
| 2-Hexanone | 4-Methyl-2-pentanone | Tetrachloroethene |
| Toluene | Chlorobenzene | Ethylbenzene |
| Styrene | Xylenes | 1,2-Dichlorobenzene |
| 1,3-Dichlorobenzene | 1,4-Dichlorobenzene | trans-1,3-Dichloropropene |
| 1,1,2,2-Tetrachloroethane | 2-Chloroethyl vinyl ether | |

Please retain for your records and [illegible] correspondence with CHEM regarding lot BR[illegible].

Certified
Anita Rodd
President

Hayward, California 94545 (415) 782-3905 New Castle, Delaware 19720 (302) 322-3808

IMPROVE COPY LEGIBILITY
PRESS HARD – PLEASE PRINT
USING A BALL POINT PEN

FEDERAL EXPRESS

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

PACKAGE TRACKING NUMBER 1708140092

MASTER AIRBILL NUMBER

1708140092

1 7 0 8 1 4 0 0 9

Sender's Federal Express Account Number: 1010-2922-4
Date: 10-23-89

1 From (Your Name) Please Print: YOUR NAME
Your Phone Number (Very Important): 303421-6611
Company: ROCKY MOUNTAIN ANALYTICAL LAB
Department/Floor No.:
Street Address: 4955 YARROW STREET
City: ARVADA State: CO ZIP Required: 80002

2 To (Recipient's Name) Please Print: JOHN SMITH
Recipient's Phone Number (Very Important): IF GIVEN
Company: SAMPLES UNLIMITED
Department/Floor No.:
Exact Street Address (We Cannot Deliver to P.O. Boxes or P.O. # Zip Codes): 555 S. 5th ST.
City: NO WHERE State: CO ZIP Required: 80000

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

PAYMENT: [X] Bill Sender [ ] Bill Recipient's FedEx Acct. No. Fill in Account Number below [ ] Bill 3rd Party FedEx Acct. No. Fill in Account Number below [ ] Bill Credit Card Fill in Credit Card Number below [ ] Cash

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

4 SERVICES
1 [X] PRIORITY 1 Overnight Delivery
2 [ ] COURIER-PAK OVERNIGHT ENVELOPE*
3 [ ] OVERNIGHT BOX
4 [ ] OVERNIGHT TUBE
5 [ ] STANDARD AIR Delivery not later than second business day
6 [ ] OVERNIGHT LETTER*
7 [ ]
8 [ ]
9 [ ]
10 [ ]
* Declared Value Limit $100

DELIVERY AND SPECIAL HANDLING
1 [ ] HOLD FOR PICK-UP (Fill in Box 4)
2 [X] DELIVER WEEKDAY
3 DELIVER SATURDAY (Extra charge) [ ]
4 [ ] DANGEROUS GOODS (Extra charge)
5 [ ] CONSTANT SURVEILLANCE SERVICE (CSS) (Extra charge) (Release Signature Not Applicable)
6 [ ] DRY ICE ___ lbs.
7 [ ] OTHER SPECIAL SERVICE
8 [ ]
9 [ ] SATURDAY PICK UP (Extra charge)
10 [ ]
11 [ ]
12 [ ] HOLIDAY DELIVERY (If offered) (Extra charge)

| PACKAGES | WEIGHT | YOUR DECLARED VALUE (Ken Right) | OVER SIZE |
|---|---|---|---|
| 1 | 40 | | |
| Total 1 | Total 40 | Total | |

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

FEDEX Corp. Employee No.

Date/Time for FEDEX Use

SERVICE CONDITIONS, DECLARED VALUE AND LIMIT OF LIABILITY

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

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

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

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

Release Signature

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

PART #111800
REVISION DATE 7/88
PRINTED IN U.S.A. EXEM
009
© 1988 F.E.C.

ORIGIN COPY

1708140092

EXAMPLE SHEET #3

**MULTIPLE PACKAGE SHIPMENT LABELS**

| | |
|---|---|
| SHIPMENT DATE | 10-23-89 |
| MASTER AIRBILL NUMBER | 1708140092 |

2 OF 4 — 4036174852

DESCRIPTION

3 OF 4 — 4036174861

DESCRIPTION

___ OF ___ — 4036174877

DESCRIPTION

___ OF ___ — 4036174886

DESCRIPTION

___ OF ___ — 4036174895

DESCRIPTION

PART #105972 WCSC REV. 10/88 © 1988 F.E.C.
SENDER'S COPY 8-89

DOMESTIC AND INTERNATIONAL MULTIPLE PACKAGE SHIPMENT LABELS

**FEDEX COURIER: These labels are designed to be used for multiple package shipments. This system should be used for all FedEx Express packages both Domestic and International.**

Enter shipment date and master airbill number. Each package within the shipment should be sequentially numb[illegible] starting with Package Number 2 of ___ (Package Number 1 will have the master airbill affi[illegible]). The shipper may enter a description of the commodity if desired (r[illegible] for international sh[illegible]nts). If all five (5) tracking numbers are not used, those numbers should be voided.

An address label reflecting exact recipient address must be affixed to each package within a [illegible]ltiple package shipment. Place the entire 7" label that includes the barcode and the customer [illegible]kage tracking number directly above the recipient address label on each package within the [illegible]pment except the package which has the original airbill attached.

Destroy any unused labels.

- **After completion, the MPS Sender copy (pink) is given to the sender**
- **The self adhesive MPS copy is attached to the back of the Recipient copy of the master airbill for Domestic shipments. Attach it to the back of the Hub Manifest copy for International shipments originating in the U.S. and to the back of the Origin Accounting copy for shipments originating outside the U.S., Hawaii and Alaska**

4036174852

MPS — 4036174852

FEDERAL EXPRESS — 4036174852

CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

4036174861

MPS — 4036174861

FEDERAL EXPRESS — 4036174861

CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

4036174877

MPS — 4036174877

FEDERAL EXPRESS — 4036174877

CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

4036174886

MPS — 4036174886

FEDERAL EXPRESS — 4036174886

CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

4036174895

MPS — 4036174895

FEDERAL EXPRESS — 4036174895

CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

EXAMPLE SHEET #4

**United Parcel Service**

PICKUP RECORD

DATE / /

REC'D FROM

PICKUP RECORD NO 081778289

CHECK CODE 5

LBS

CL 810-

ENTER EACH PACKAGE ON A SEPARATE LINE. IF RECORD IS VOIDED, PLEASE GIVE TO DRIVER.
INCREASE FRACTIONS OF A POUND TO NEXT FULL POUND.

| REFERENCE NO | NAME — STREET — CITY | STATE | ZIP CODE | TYPE SERVICE: GROUND ZONE In State | GROUND ZONE Out of State | GROUND LBS | AIR ZONE | AIR LBS | (✓) OS* | DECLARED VALUE** IF IN EXCESS OF $100.00 | COD*** AMOUNT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | SPARTON TECH 9621 COORS Rd. N.W. ALBUQUERQUE | NM | 87114 | | 4 | 4 | | | | | |
| 2 | EAST 2200 COTTONTAIL LANE SOMERSET | NJ | 08873 | | 7 | 2 | | | | | |
| 3 | | | | | | | | | | | |
| 4 | | | | | | | | | | | |
| 5 | | | | | | | | | | | |
| 6 | | | | | | | | | | | |
| 7 | | | | | | | | | | | |
| 8 | | | | | | | | | | | |
| 9 | | | | | | | | | | | |
| 10 | | | | | | | | | | | |
| 11 | | | | | | | | | | | |
| 12 | | | | | | | | | | | |
| 13 | | | | | | | | | | | |
| 14 | | | | | | | | | | | |
| 15 | | | | | | | | | | | |

EXAMPLE SHEET #5

| ED BY | PICKUP TIME | NO PKGS | NO CALLS |
|---|---|---|---|
| | | | |

* OS (Oversize) applies if less than 25 lbs and more than 84 inches in length and girth combined.

** Unless a greater value is declared in writing on this receipt, the shipper hereby declares and agrees that the released value of each package or article not enclosed in a package covered by this $100, which is a reasonable value under the circumstances surrounding the transportation. The rules relating to liability established by the Warsaw Convention and any amendments thereto shall the international carriage of any shipment hereunder insofar as the same is governed thereby. The entry of a C.O.D. amount is not a declaration of value. In addition, the maximum value for an package is $25,000 and the maximum carrier liability is $25,000. Claims not made to carrier within 9 months of the scheduled delivery date are waived.

*** All checks in payment of C.O.D.'s accepted at shipper's risk.

6-88 (800M BKS. 12-88)U

EXAMPLE SHEET #6

# GROUND SERVICE ZONE CHART

For Shippers with ZIP Codes 800-01 to 802-99

## Service to 48 Continental United States

To determine zone, take first three digits of ZIP Code to which parcel is addressed and refer to chart below

| ZIP CODE PREFIXES | UPS ZONE |
|---|---|
| 004-005 | 7 |
| 010-043 | 7 |
| 044 | 8 |
| 045 | 7 |
| 046-048 | 8 |
| 049-089 | 7 |
| 100-139 | 7 |
| 140-147 | 6 |
| 148-149 | 7 |
| 150-167 | 6 |
| 168-199 | 7 |
| 200-214 | 7 |
| 215 | 6 |
| 216-225 | 7 |
| 226 | 6 |
| 227 | 7 |
| 228-229 | 6 |
| 230-239 | 7 |
| 240-253 | 6 |
| 254 | 7 |
| 255-274 | 6 |
| 275-279 | 7 |
| 280-282 | 6 |
| 283-285 | 7 |
| 286-293 | 6 |
| 294-295 | 7 |
| 296-299 | 6 |
| 300-319 | 6 |
| 320-322 | 7 |
| 323-325 | 6 |
| 326-339 | 7 |
| 342-349 | 7 |
| 350-369 | 6 |
| 370-372 | 5 |

| ZIP CODE PREFIXES | UPS ZONE |
|---|---|
| 373-374 | 6 |
| 375 | 5 |
| 376-379 | 6 |
| 380-384 | 5 |
| 385 | 6 |
| 386-392 | 5 |
| 393-395 | 6 |
| 396-397 | 5 |
| 399 | 6 |
| 400-418 | 6 |
| 420 | 5 |
| 421-422 | 6 |
| 423-424 | 5 |
| 425-459 | 6 |
| 460-466 | 5 |
| 467-468 | 6 |
| 469 | 5 |
| 470-471 | 6 |
| 472 | 5 |
| 473 | 6 |
| 474-479 | 5 |
| 480-489 | 6 |
| 490-491 | 5 |
| 492 | 6 |
| 493-495 | 5 |
| 496-497 | 6 |
| 498-499 | 5 |
| 500-503 | 4 |
| 504 | 5 |
| 505 | 4 |
| 506-507 | 5 |
| 508-516 | 4 |
| 520-560 | 5 |
| 561-562 | 4 |
| 563-567 | 5 |

| ZIP CODE PREFIXES | UPS ZONE |
|---|---|
| 570-576 | 4 |
| 577 | 3 |
| 580-583 | 5 |
| 584-593 | 4 |
| 594-595 | 5 |
| 596-597 | 4 |
| 598-599 | 5 |
| 600-639 | 5 |
| 640-649 | 4 |
| 650-659 | 5 |
| 660-675 | 4 |
| 676-679 | 3 |
| 680-689 | 4 |
| 690-693 | 3 |
| 700-704 | 6 |
| 705-729 | 5 |
| 730-732 | 4 |
| 733 | 5 |
| 734-738 | 4 |
| 739 | 3 |
| 740-746 | 4 |
| 747 | 5 |
| 748 | 4 |
| 749-761 | 5 |
| 762-763 | 4 |
| 764-789 | 5 |
| 790-799 | 4 |
| 800-810 | 2 |
| 811 | 3 |
| 812 | 2 |
| 813-815 | 3 |
| 816-820 | 2 |
| 821 | 4 |
| 822-823 | 3 |

| ZIP CODE PREFIXES | UPS ZONE |
|---|---|
| 824 | 4 |
| 825-827 | 3 |
| 828 | 4 |
| 829-830 | 3 |
| 831-834 | 4 |
| 835-838 | 5 |
| 840-853 | 4 |
| 854 | 5 |
| 855-873 | 4 |
| 874-877 | 3 |
| 878-883 | 4 |
| 884 | 3 |
| 885 | 4 |
| 889-892 | 5 |
| 893 | 4 |
| 894-897 | 5 |
| 898 | 4 |
| 900-954 | 5 |
| 955 | 6 |
| 956-961 | 5 |
| 970-979 | 5 |
| 980-982 | 6 |
| 983-984 | 5 |
| 985 | 6 |
| 986 | 5 |
| 988-994 | 5 |

See separate charts for UPS Next Day Air, 2nd Day Air and International services.
Air service is provided to all points in Alaska, Hawaii and Puerto Rico.

017232 REV 2

Example Sheet #7

BOTTLE ORDER

RMAL Client ID:_____ Request filled by:_____

Request By:_____ Date:_____ Time:_____

Coolers Billed To (specify Client ID):_____

Blue Ice Required: YES NO
Special Requirements:_____

==========

DI_____Gallons Carbon Free_____Gallons Milli-Q_____Gallons

| No. of bottles | Standard Water | No. of Bottles | Bulk Water |
|---|---|---|---|
| _____ | 1. 32oz poly (WM) | _____ | 20. 1/2 gallon glass |
| _____ | 2. 16oz glass (BR), 50% H2SO4 | _____ | 21. 1 gallon glass |
| _____ | 3. 32oz glass (BR), 50% H2SO4 | | |
| _____ | 4. 16oz poly (WM), 20% HNO3 | | |
| _____ | 5. 2-32oz poly (BR), 20% HNO3 | | Solids |
| _____ | 6. 8oz poly (WM), 50% NaOH | | |
| _____ | 7. 8oz poly (WM), Zn Ac + NaOH | _____ | 30. 16oz glass (WM) |
| _____ | 8. 4.5oz poly, sterilize | _____ | 31. 8oz glass (WM) |
| _____ | 10. 3-40mL glass w/septa,Na2S2O3 | _____ | 32. 4oz glass (WM) |
| _____ | 10A. Trip Blank | | |
| _____ | 11. 3-40mL glass w/septa,HCL | | |
| _____ | 11A. Trip blank | | |
| _____ | 12. 2-32oz glass (BR) | | |
| _____ | 13. 2-32oz glass (BR) | | |
| _____ | 14. 32oz glass (BR) | | |
| _____ | 15. Single: 8oz amber glass (BR) | | |
| | Quad: 32oz amber glass (BR) | | |
| | 50% H2SO4 | | |

==========

Ship To:

_____

_____

_____

_____

_____

Client P.O.#_____

Pick-up on:___/___/___Time:_____

Delivered By (Date):___/___/___

Ship By:_____

_____

UPS Charges:_____

Fed X Charges:_____

Client Fed X Account #:_____

==========

Invoice #:_____

Sample Safe / Cooler numbers:_____

Bottle lot #'s by type:_____

_____

STANDARD
OPERATING
PROCEDURE

| Subject or Title: SAMPLE RECEIPT AND CHAIN OF CUSTODY | | Page 1 of 2 |
|---|---|---|
| SOP No.: LP-RMA-0005 | Revision No.: Original | Effective Date: 12/9/87 |
| Supersedes: | | |

1. Purpose:

To document receipt of all samples to the laboratory. To notify lab personnel of all incoming samples. To notify lab personnel of arriving samples that contain short holding parameters. To record the transfer of samples from the client to the lab.

2. Policies:

Always assign a project number to every group of samples that arrive at the lab regardless of whether work is proceeded on them or not.

Project numbers are assigned in numerical order. USGS and MKE samples receive separate series of numbers. MKE samples require special chain of custody tracking.

3. Safety:

Always wear gloves and glasses while unpacking coolers. Coolers containing strong smelling samples must be unpacked under the hood area.

4. Procedure:

a. As samples arrive they are given a unique project number for each group of samples from one client and recorded in the log book (Figure 1).
b. Fill out the Sample checklist (Figure 2) while unpacking the samples.
c. For samples arriving by a courier check that the custody seals are intact.
d. Open the coolers, unpack the samples and check the information written on the chain of custody against what was received. Note any discrepancies such as missing samples, or broken bottles on the chain of custody form.
e. Label all the samples (usually by sampling sites) with a project number and unique sample number (1,2,3,etc.). Record these numbers on the chain of custody next to the client identifications.

| Prepared by: | Date: |
|---|---|
| Management Approval: | Date: 12/10/87 |
| QA Officer Approval: | Date: 12/9/87 |

f. Sign and date the Chain of Custody (Figure 3). For samples hand delivered have the client sign and relinquish the custody. Always retain the top copy with the samples and only give a bottom copy to the client.
g. Look for any inorganic short holding parameters and sign in these samples on the inorganic short holding clipboard (Figure 4). Look for any volatile parameters and sign these samples in on the Volatile clipboard (Figure 5).
h. Take a picture of the samples. Label a manila file folder with the project number. Place the picture, checklist, chain of custody and any paperwork received in the folder.
i. Deliver the file folder to the appropriate project manager.
j. Place the samples in boxes and store in the walk in cooler on special shelves pending log in. Bottles needed to analyze the short holding parameters are placed in a special location in the walk in cooler.

5. Responsibilities:

Sample receiving personnel are responsible for signing the chain of custody upon receipt of samples, for knowing the location of the samples except when used by an analyst, and for signing out maximum security samples. Sample receiving personnel are responsible for noting the short holding parameters only when indicated on the paperwork from the client. Client managers must notify sample receiving if others are to be included.

6. Comments:

For maximum security of samples (beyond the storage in the secured facility) an internal chain of custody is provided. Analysts must sign for the samples in a special book and sign them in on return. The samples are stored in one of 3 locked refrigerators.

| PROJECT # | CLIENT NAME | PROJECT NAME/ # | # of COOLER | PARAMETERS/COMMENTS | DATE REC'D. | TIME REC'D | MATRX |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
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FIGURE 2

## SAMPLE CHECKLIST

PROJECT # | | | | | | DIV: | | | |

COMPANY NAME: _____

SAMPLE CHECKLIST DONE BY: _____

| _____COOLER(S)#_____(RMAL/CLIENT) | Y | N |
|---|---|---|
| PICTURE TAKEN: | | |
| SEALS INTACT: | | |
| COOLER TEMP OK: | | |
| BOTTLES BROKEN OR LEAKING: | | |
| CONTAINERS LABELED: | | |
| RADIATION DETECTION: | | |
| CHAIN OF CUSTODY: | | |
| CC AGREES WITH SAMPLES: | | |
| VOA SAMPLES FILLED COMPLETLY: | | |
| SEDIMENT PRESENT IN WATERS: | | |
| SAMPLE CORRECTLY PRESERVED: | | |
| SHORT HOLDING TIMES: | | |

( )MS ( )VOA ( )60Z ( )IN

SAMPLE MATRIX: ( )WATER ( )SOIL ( )WASTE

OTHER: _____

TYPE OF BOTTLES: ( )RMA ( )CLIENT

DISCREPANCES: _____

Date_____

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| PROJECT NUMBER : | | | | | | | | |
| SAMPLE NUMBERS : | | | | | | | | |
| COLL. DATE/ TIME : | | | | | | | | |
| REC. DATE/ TIME : | | | | | | | | |
| PUT ON LIST BY : | | | | | | | | |
| MASS/SPEC-VOA : | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| G/C-VOA : | | | | | | | | |
| CO2 | | | | | | | | |
| HEX | | | | | | | | |
| TCLP | | | | | | | | |
| UNBROWN : | | | | | | | | |
| ANALYST : | | | | | | | | |
| # OF VIAL S/SAMPLE: | | | | | | | | |
| COMMENTS : | | | | | | | | |

INORGANIC SHORT HOLDING TIME (≤ 48 hrs) PARAMETERS RECEIVED _____

| | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Project Number | | | | | | | | | | | | | | | | | | |
| Sample Numbers | | | | | | | | | | | | | | | | | | |
| Client | | | | | | | | | | | | | | | | | | |
| Date Collected | | | | | | | | | | | | | | | | | | |
| Special Instructions | | | | | | | | | | | | | | | | | | |
| Logged By / Time | | | | | | | | | | | | | | | | | | |
| Approved By (CSR) | | | | | | | | | | | | | | | | | | |
| ASAP | Dissolved Oxygen($O_2$) | | | | | | | | | | | | | | | | | |
| | Sulfite ($SO_3^{2-}$) | | | | | | | | | | | | | | | | | |
| 24 HOURS | pH (S) | | | | | | | | | | | | | | | | | |
| | pH (Quad) | | | | | | | | | | | | | | | | | |
| | Conductance (S) | | | | | | | | | | | | | | | | | |
| | Conductance (Quad) | | | | | | | | | | | | | | | | | |
| | Alkalinity | | | | | | | | | | | | | | | | | |
| | Chlorine, Residual | | | | | | | | | | | | | | | | | |
| | Chromium, Hexavalent ($Cr^{+6}$) | | | | | | | | | | | | | | | | | |
| | Coliform, Fecal | | | | | | | | | | | | | | | | | |
| | Coliform, Total | | | | | | | | | | | | | | | | | |
| | Odor | | | | | | | | | | | | | | | | | |
| 48 HOURS | BOD | | | | | | | | | | | | | | | | | |
| | Color | | | | | | | | | | | | | | | | | |
| | MBAS (Surfactants) | | | | | | | | | | | | | | | | | |
| | Nitrate ($NO^{3-}$) | | | | | | | | | | | | | | | | | |
| | Nitrite ($NO_2^-$) | | | | | | | | | | | | | | | | | |
| | ortho-Phosphate | | | | | | | | | | | | | | | | | |
| | Turbidity | | | | | | | | | | | | | | | | | |
| | Settleable Solids | | | | | | | | | | | | | | | | | |
| OTHER | TDS (Need Conductance (S)) | | | | | | | | | | | | | | | | | |
| | Ion Balance (Need pH (S), Conductance (S), Alkalinity) | | | | | | | | | | | | | | | | | |

Enseco - Rocky Mountain Analytical
7555 Yarrow Street
Arvada, Colorado 80002
303/421-6611 Facsimile 303/431-7171

# CHAIN OF CUSTODY

No. 4999

Attn:

Enseco Client

Project

Sampling Co.

Sampling Site

Team Leader

## SAMPLE SAFE™ CONDITIONS

1. Packed by Seal #
2. Seal Intact Upon Receipt by Sampling Co. Yes No
3. Condition of Contents
4. Sealed for Shipping by
5. Initial Contents Temp °C Seal #
6. Sampling Status Done Continuing Until
7. Seal Intact Upon Receipt by Laboratory: Yes No
8. Contents Temperature Upon Receipt by Lab °C
9. Condition of Contents

| Date | Time | Sample ID/Description | Sample Type | No. Containers | Analysis Parameters | Remarks |
|---|---|---|---|---|---|---|
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
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| | | | | | | |

## CUSTODY TRANSFERS PRIOR TO SHIPPING

| Relinquished by (signed) | Received by (signed) | Date | Time |
|---|---|---|---|
| 1 | | | |
| 2 | | | |
| 3 | | | |

## SHIPPING DETAILS

Delivered to Shipper by

Method of Shipment Airbill #

Received for Lab Signed Date/Time

Enseco Project No

White and Pink Copies t Yellow to Sampler

SS 001

# 10. DATA REDUCTION, VALIDATION, AND REPORTING

## Data Reduction and Validation

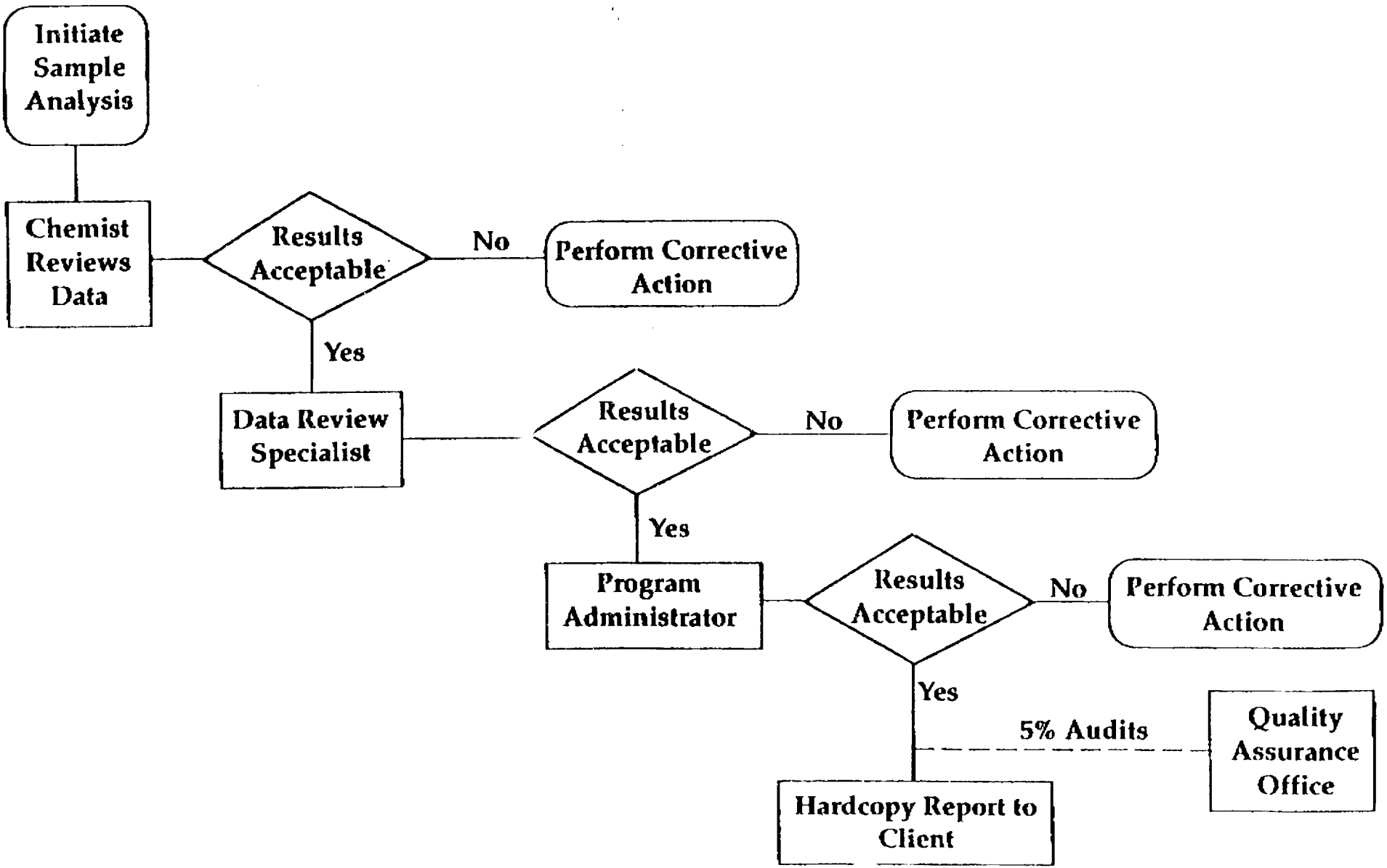
All analytical data generated within Enseco laboratories are extensively reviewed prior to report generation to assure the validity of the reported data. The data validation process consists of data generation, reduction, and three levels of documented review, as described below (also see Figure 10-1). In each stage, the review process is documented by the signature of the reviewer and the date reviewed.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, anomaly forms are complete; holding times are documented, etc.).

Figure 10-1

# Data Validation Scheme



The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated Level 1 review. The analyst then passes the data package to an independent reviewer, who performs a Level 2 review.

Level 2 review is performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; anomaly forms are complete; holding times are documented, etc.);
- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

An important element of Level 2 review is the documentation of any errors that have been identified and corrected during the review

process. Enseco believes that the data package submitted by the analyst for Level 2 review should be free of errors. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Level 2 data review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Before the report is released to the client, the data are reviewed for completeness and to ensure that the data meet the overall objectives of the project. This review is labeled Level 3 review and is typically done by the Program Administrator.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

In addition to the three levels of review discussed above, the Divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

**Data Reporting**

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. In general, Enseco reports contain:

General Discussion: Description of sample types, tests performed, any problems encountered and general comments are given.

Analytical Data: Data are reported by sample or by test. Pertinent information including dates sampled, received, prepared, and extracted are included on each results page. The Enseco reporting limit for each analyte is also given.

Laboratory Performance QC Information: The results (Percent Recovery and Relative Percent Difference) of the Laboratory Control Samples analyzed with the project are listed, together with the control limits. Also, the analytical results for method blanks generated during analysis of organic and metals parameters are given.

Matrix-Specific QC Information: Results of any sample duplicates, matrix spikes, matrix spike duplicates or other project-specific QC requested by the client are also reported.

Methodology: Reference for analytical methodology used is cited.

Custom Services: Special services including data interpretation, special consultation, and raw data packages (when requested) are included.

## II. INTERNAL QC CHECKS

The Enseco QA/QC program monitors data quality with internal QC checks. Internal QC checks are used to answer two questions:

1) Are laboratory operations "in control," (i.e., operating within acceptable QC guidelines), during data generation?

2) What effect does the sample matrix have on the data being generated?

The first question is answered by Laboratory Performance QC. Laboratory performance QC is based on the use of a standard, control matrix to generate precision and accuracy data that are compared, on a daily basis, to control limits. This information, in conjunction with method blank data, is used to assess daily laboratory performance.

The second question is addressed with Matrix-Specific QC. Matrix-Specific QC is based on the use of an actual environmental sample for precision and accuracy determinations and commonly relies on the analysis of matrix spikes, matrix duplicates, and matrix spike duplicates. This information, supplemented with field blank results, is used to assess the effect of the matrix and field conditions on analytical data.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. Matrix-Specific QC is available as an option to the client and should be specified based on the types of matrices to be analyzed and the Data Quality Objectives (DQOs) and regulatory requirements of the project. A complete discussion of the Enseco Internal QC Check program follows.

### Laboratory Performance QC Program

Laboratory Performance QC is performed for every routine Enseco analysis to demonstrate that laboratory operations are "in control". The main elements of Laboratory Performance QC are:

# APPENDIX D

## GENERAL STANDARD OPERATING PROCEDURE FOR COMPUCHEM LABORATORIES, INC.

## 3.0 Quality Assurance Policy Statement

The Management of CompuChem® Laboratories is fully and firmly committed to the quality assurance program described in this QA Plan. Each director, manager and supervisor as well as their staff members, as assigned in accordance with this plan, is obligated to comply with its stated requirements, responsibilities and objectives.

The primary QA objective is to develop and implement procedures for sample receiving, chain-of-custody, sample preparation, laboratory analysis, data validation, and reporting that will provide data that are legally defensible in a court of law. Key aspects of these procedures are described in this QA Plan, while specific details are included in the laboratory's SOPs and QA SOPs -- all three references serving to document the elements of CompuChem's QA Program.

The QA Program will be maintained and expanded or modified as necessary, to ensure all reported data are of uncompromising quality. In order to determine whether QA objectives are met, sufficient quality control will be generated to evaluate precision, accuracy and completeness, and when possible, a statement regarding representativeness and comparability will be provided.

# 4.1 Quality Assurance Management

## 4.1 Introduction

With over 400 employees, CompuChem® offers the scientific and technical expertise needed to service the analytical and informational needs of our customers. In addition to our experienced analytical laboratory personnel (with specialized skills in organic and inorganic analyses) CompuChem® utilizes a computer systems staff that plans, develops, and implements software systems for data management and sample scheduling and control. To ensure that the analytical needs of our clients are met, customer service representatives are assigned to each account, providing a liaison between the customer and the laboratory.

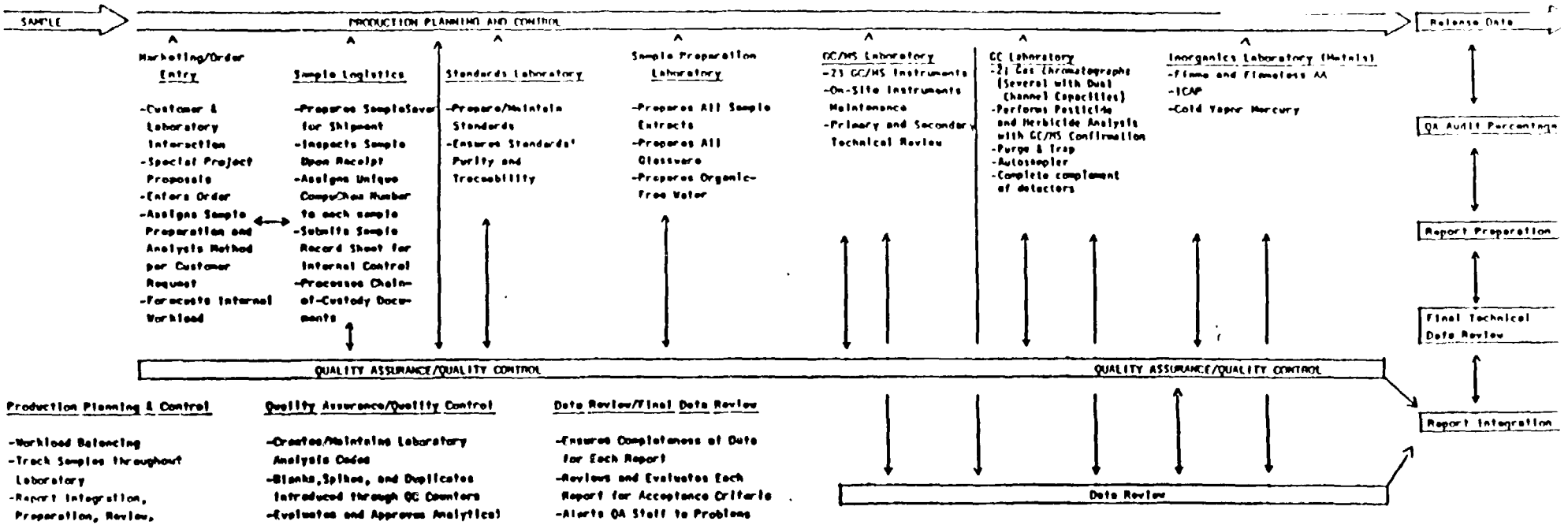
For our involvement with the EPA, dialogue has been established and is maintained with the Program Office in Washington, DC through our Project Officer and with our Deputy Project Officer located in our region (Region IV).

The following section describes the operational and functional responsibilities of key lab personnel, including the duties and services performed as they relate to product quality. Additionally, the roles and responsibilities of the Quality Assurance Department and its organizational relationship to lab management are identified. Please refer to the organizational charts at the end of this section for an overview of these relationships.

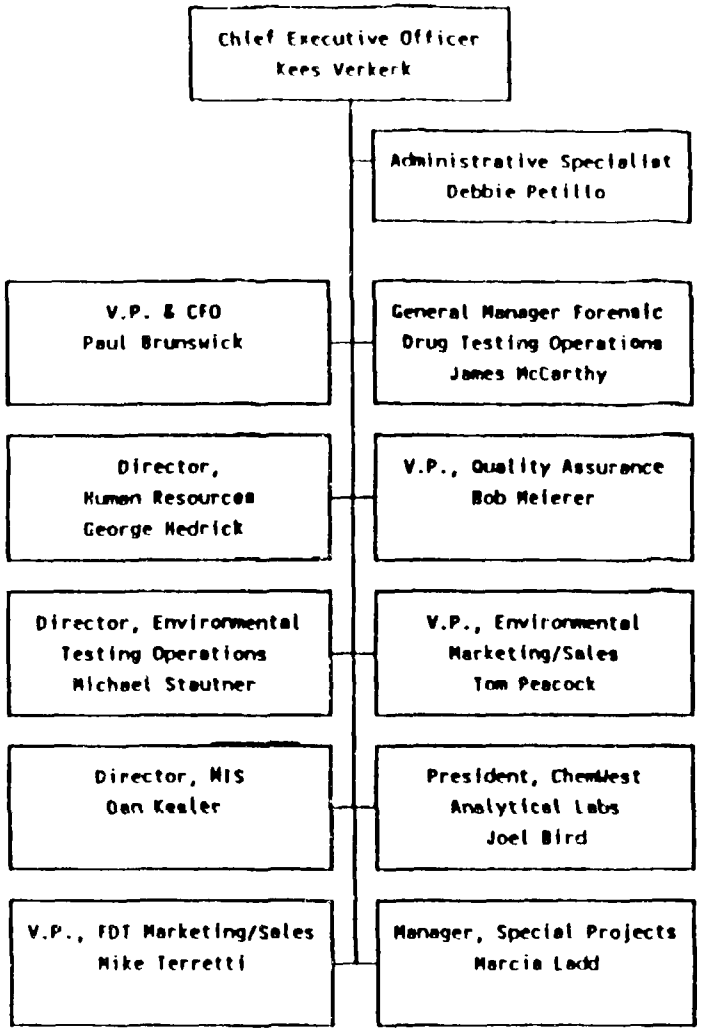
The Quality Assurance staff monitors and reviews all laboratory units and operates independently of production areas. All quality control criteria are documented and compliance is verified at each level of laboratory data review.

In-lab data validation and independent QA auditing SOPs describe the details of these quality control functions. Section 8.2 presents a general overview of the data review/data validation processes. The QA Department is responsible for, among other things, verifying the integrity of these functions and documenting performance for lab management review.

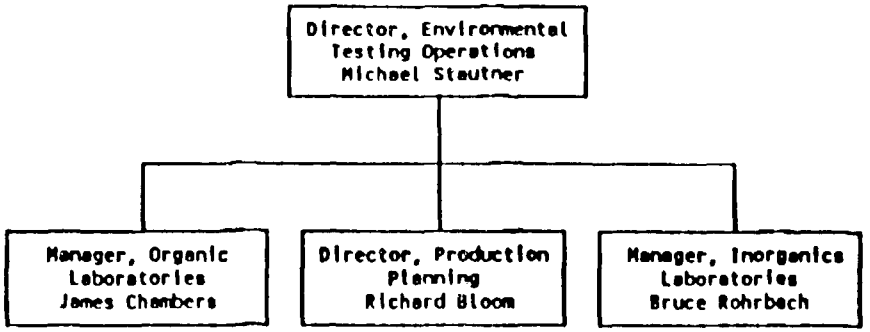
# Laboratory Operations/Quality Assurance Relationships



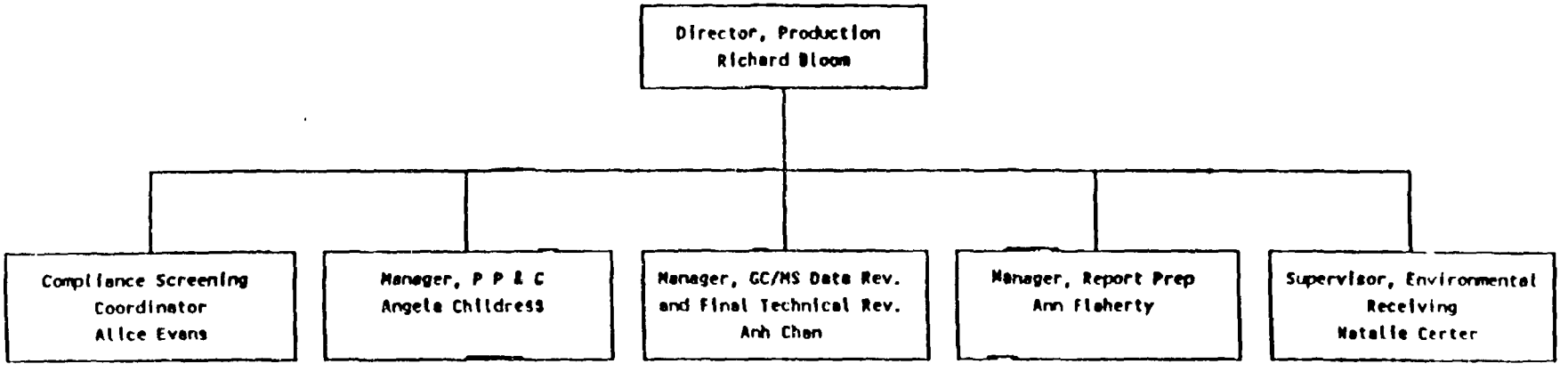
CompuChem Corporation
May 1, 1989



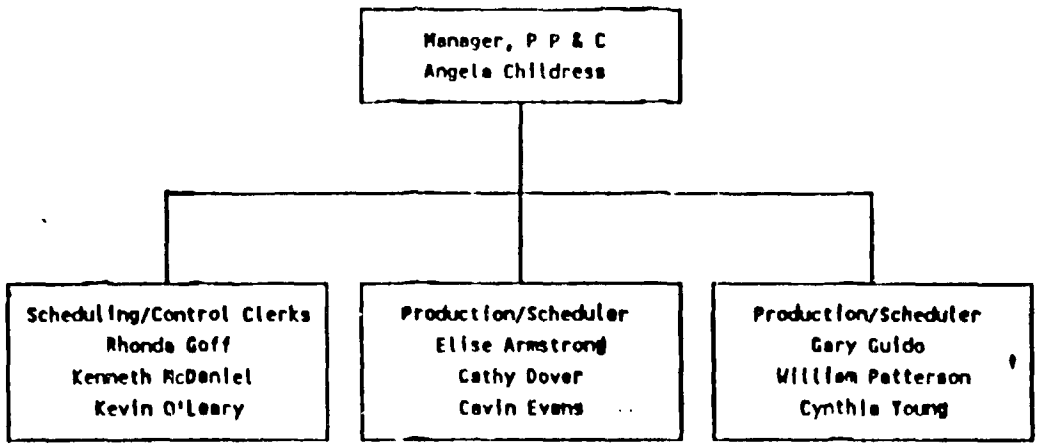
CompuChem Laboratories, Inc.
Environmental Testing Operations
May 1, 1989



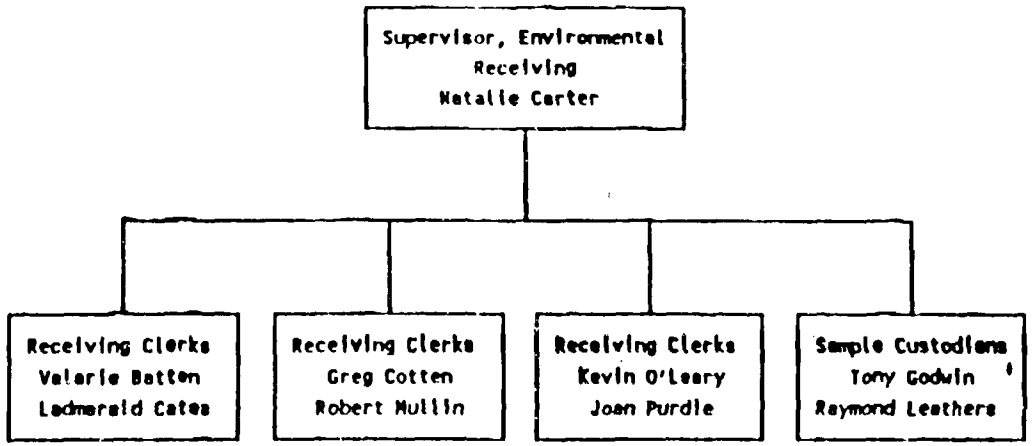
CompuChem Laboratories, Inc.
Production
May 1, 1989



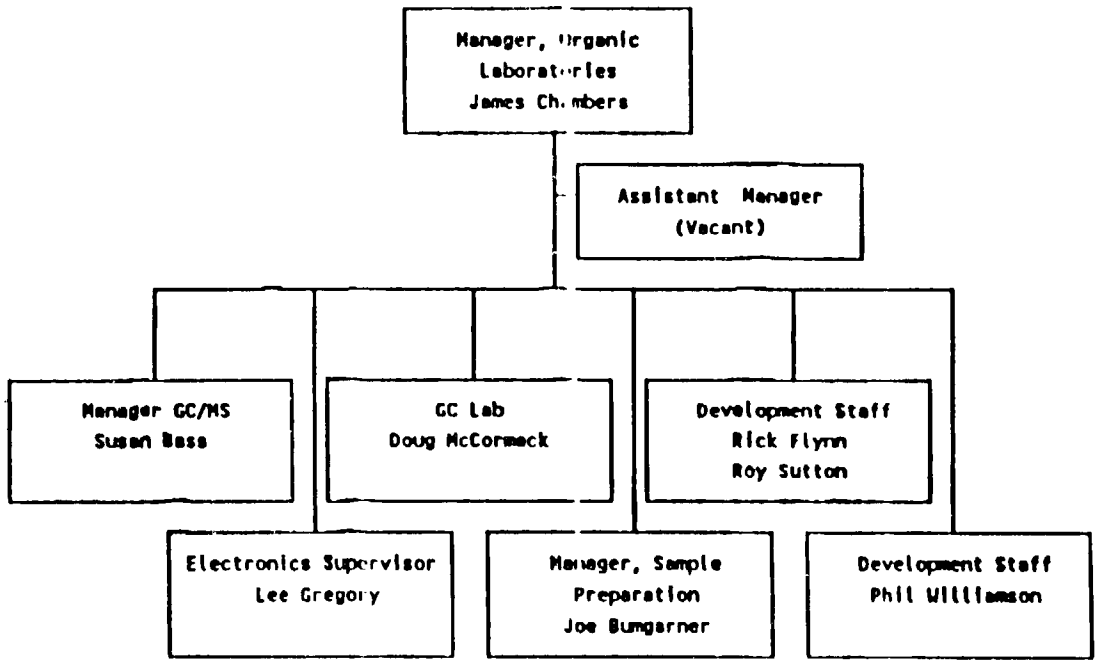
CompuChem Laboratories, Inc.
P P & C
May 1, 1989

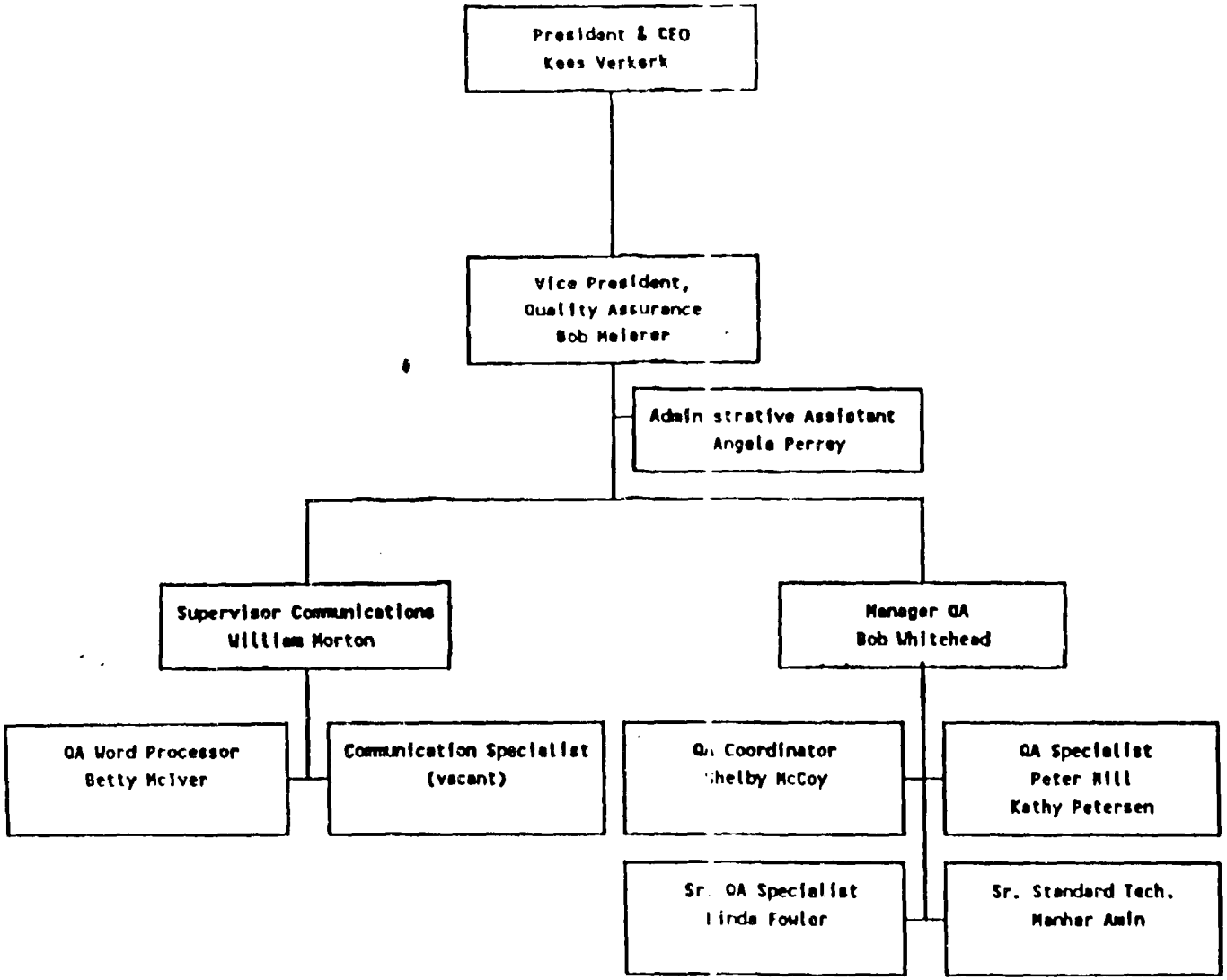


CompuChem Laboratories, Inc.
Environmental Receiving
May 1, 1989

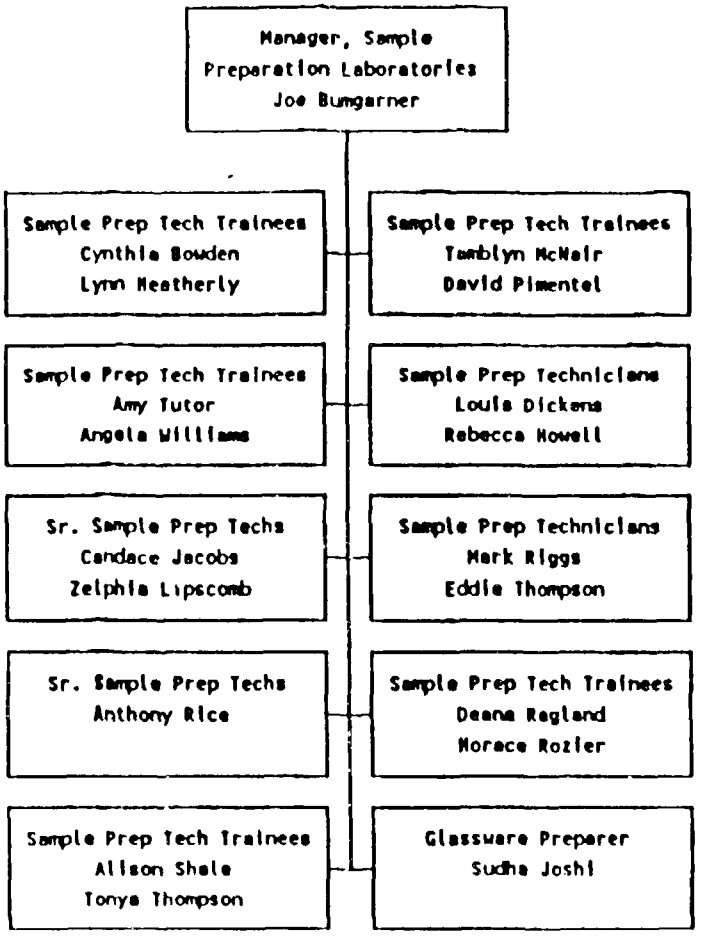


CompuChem Laboratories
Organics Laboratory
May 1, 1989

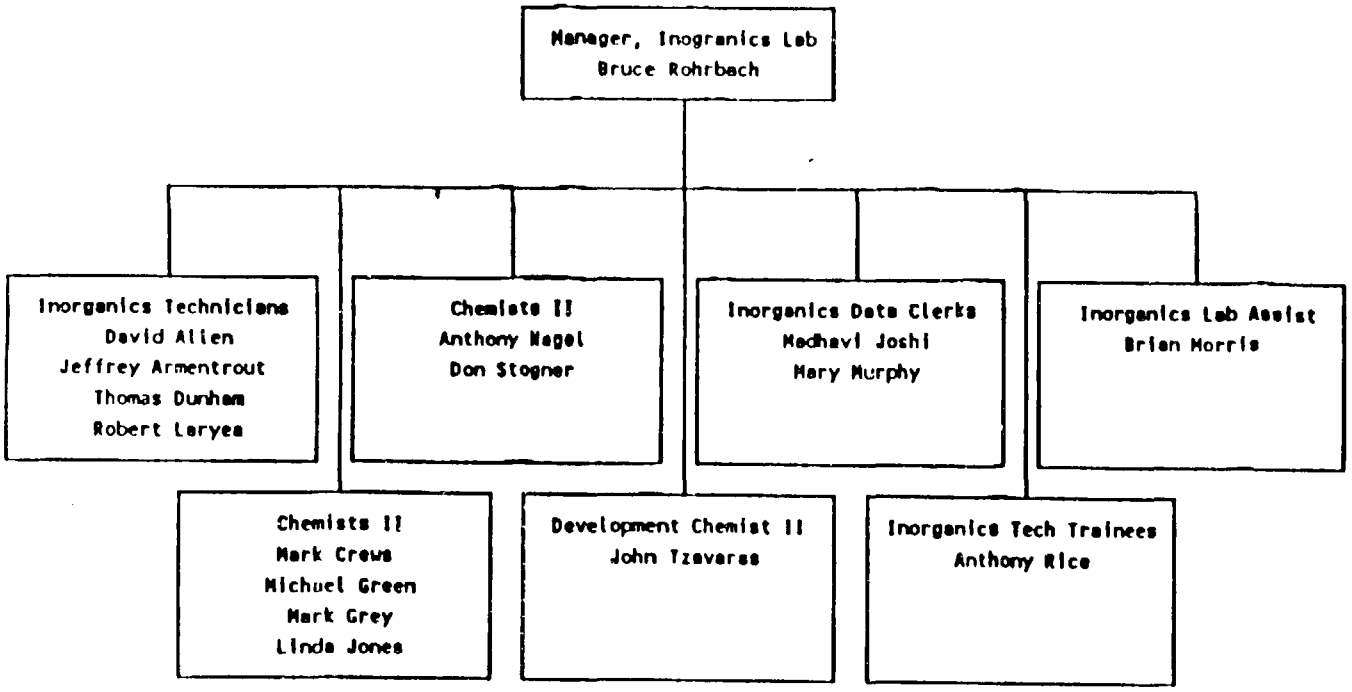



President & CEO
Kees Verkerk
Vice President,
Quality Assurance
Bob Meierer
Administrative Assistant
Angela Perrey
Supervisor Communications
William Morton
Manager QA
Bob Whitehead
QA Word Processor
Betty McIver
Communication Specialist
(vacant)
QA Coordinator
Shelby McCoy
QA Specialist
Peter Hill
Kathy Petersen
Sr. QA Specialist
Linda Fowler
Sr. Standard Tech.
Manher Amin

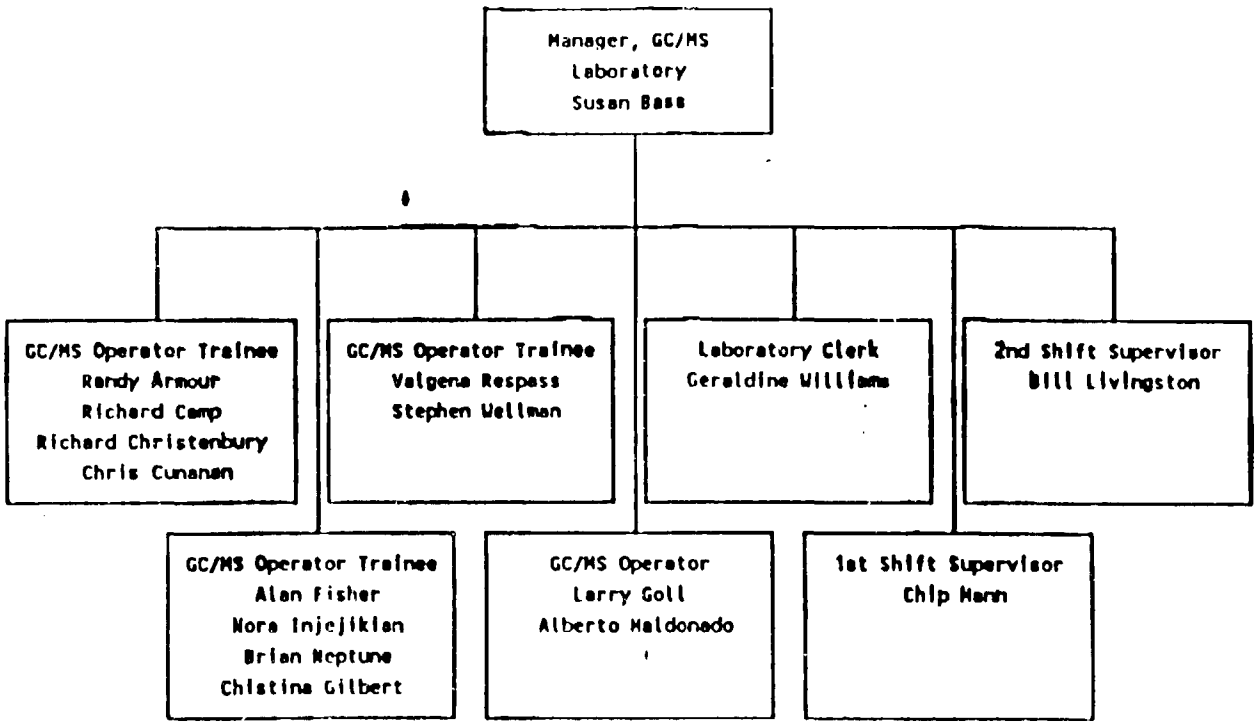
CompuChem Laboratories, Inc.
Sample Preparation Laboratories
May 1, 1989



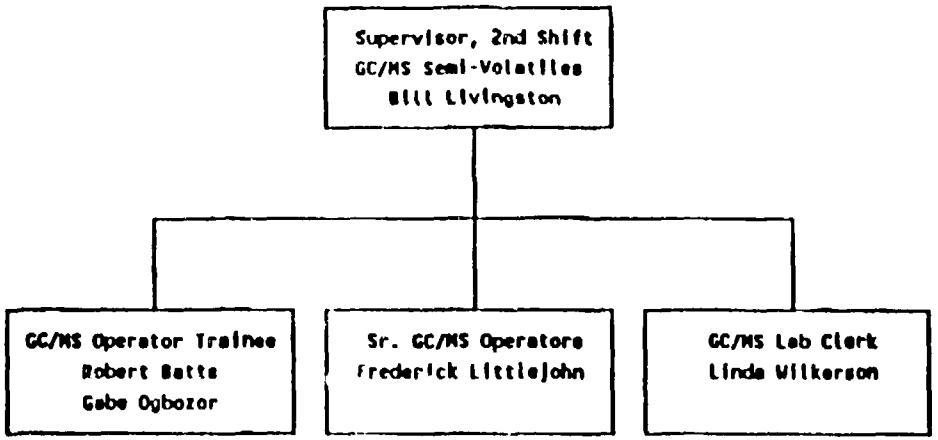
CompuChem Laboratories, Inc.
Inorganics Laboratory
May 1, 1989



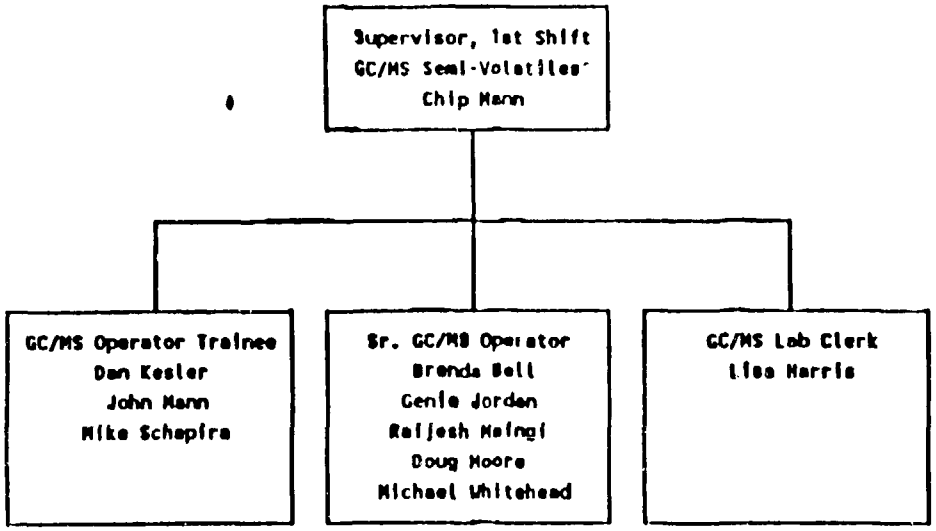
CompuChem Laboratories, Inc.
GC/MS Volatiles Laboratory
May 1, 1989



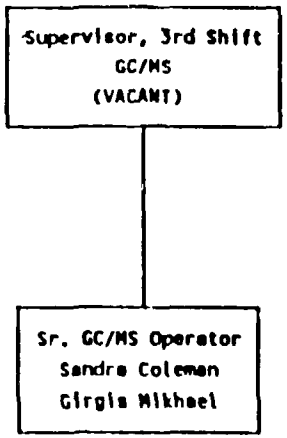
CompuChem Laboratories, Inc.
GC/MS Lab 2nd Shift
May 1, 1989



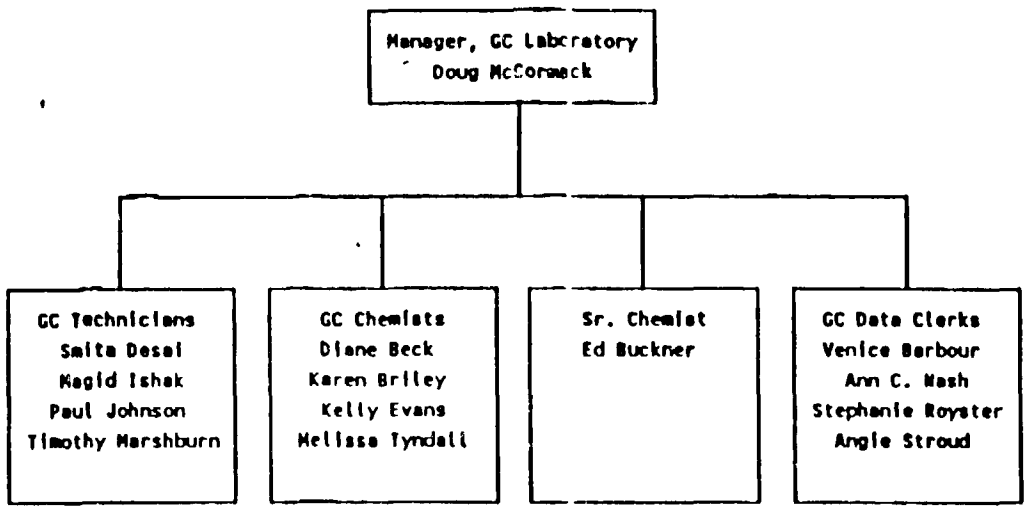
CompuChem Laboratories, Inc.
1st Shift GC/MS
May 1, 1989



CompuChem Laboratories, Inc.
GC/MS Lab 3rd Shift
May 1, 1989



ComplxChem Laboratories, Inc.
GC Laboratory
May 1, 1989



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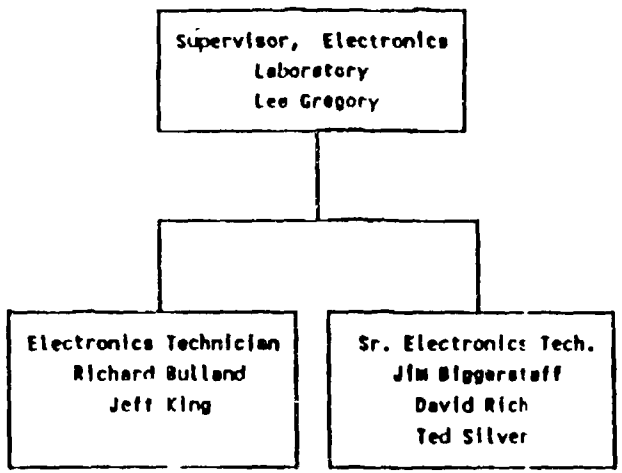
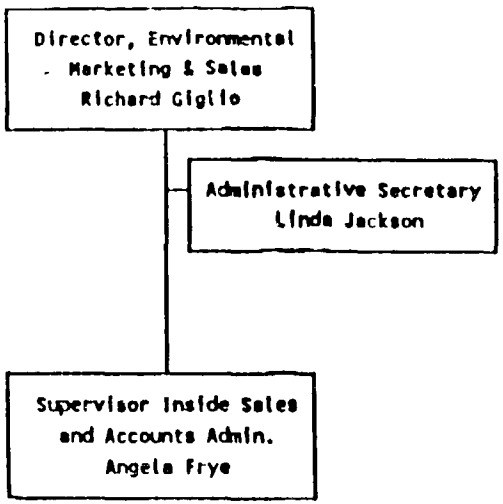
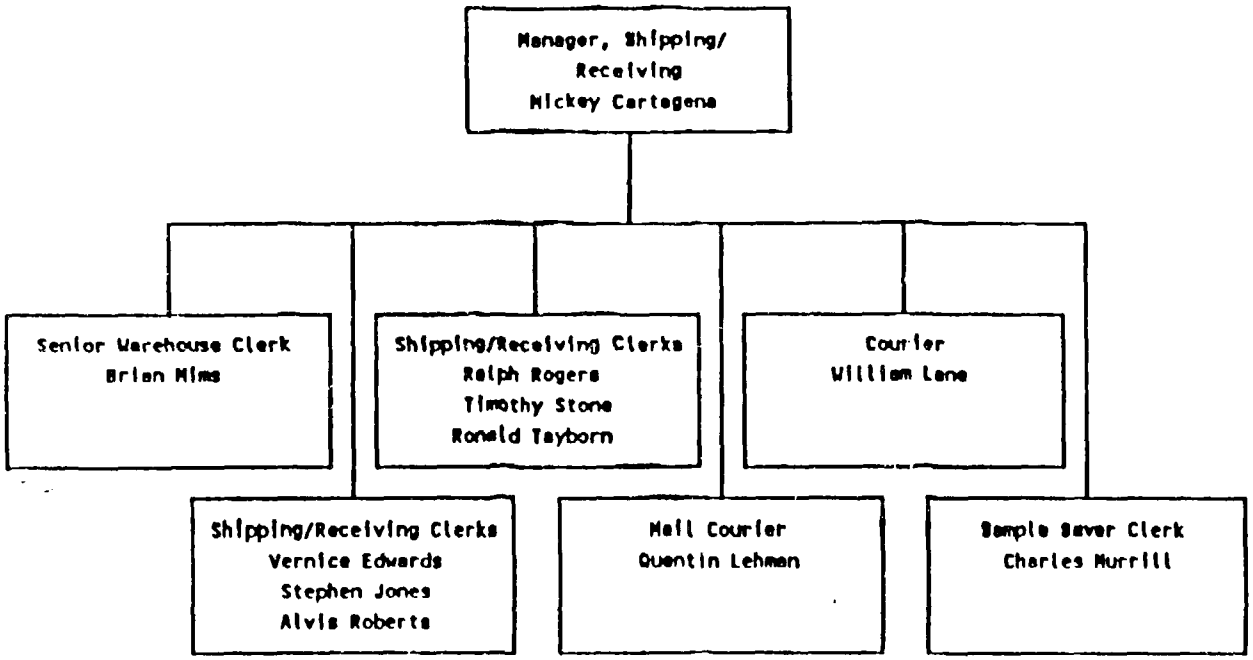
Lab Instrumentation
May 1, 1989



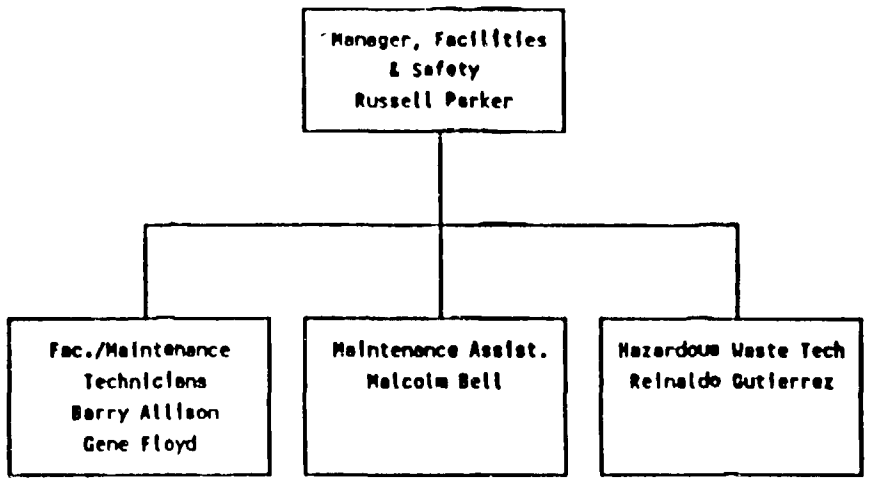
CompuChem Laboratories, Inc.
Environmental Marketing & Sales
May 1, 1989



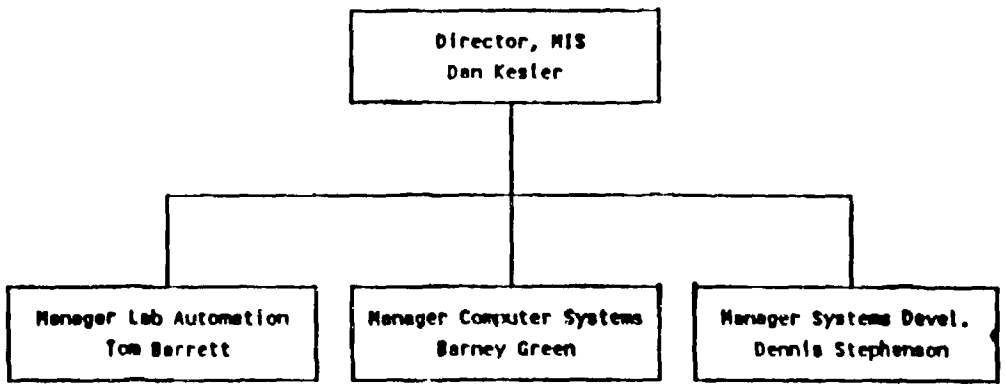
CompuChem Laboratories, Inc.
Shipping/Receiving
May 1, 1989



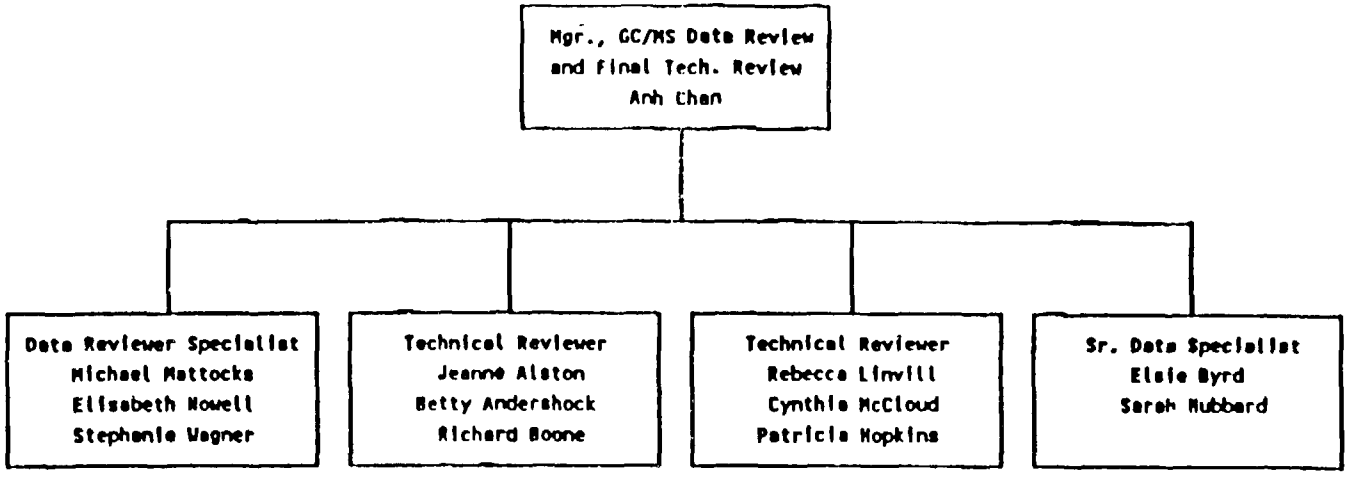
CompuChem Laboratories, Inc.
Facilities and Safety
May 1, 1989



CompuChem Laboratories, Inc.
MIS
May 1, 1989



CompuChem Laboratories, Inc.
Data Review and Final Technical Review
May 1, 1989



## 4.2 Assignment of Responsibilities

The main objectives of CompuChem's Quality Assurance Program are to assure that our laboratories generate data of known quality, that data quality meets or exceeds all QC/QA criteria, and that the records necessary to document laboratory performance are maintained. Additionally, the QA Department has the responsibility of providing feedback to management and to identify and implement policies to improve quality. The success or failure of the program is, to a great extent, dependent on the capabilities of those assigned the responsibility of carrying it out.

The following is a brief summary of the responsibilities and authorities assigned to each of the QA Department staff members:

### Director of Quality Assurance

To be certain that the laboratory achieves all QA Program objectives, the Director of Quality Assurance monitors and directs the quality activities of QA Department and Lab personnel. The Director acts in strict adherence to the procedures and requirements stated in the Quality Assurance Plan.

The Director of QA reports directly to the corporation's CEO, is a member of the Executive Staff, and is organizationally and functionally independent of all personnel directly involved in the operation of the technical program. The Director has the authority to terminate non-conforming work at any time.

Additional responsibilities and duties include:

Preparing and overseeing the preparation of the QA Plan;

Monitoring the QA Program as documented in the QA Plan and ensuring that all elements are carried out as written;

Developing and implementing new QA programs, including statistical procedures, additional quality control measures, validation of new methods, etc.;

Conducting scheduled or unannounced audits and inspections, reporting findings to management, and when needed, ensuring that corrective action is taken;

Maintaining current documentation of all measurement procedures routinely used in the laboratories, including those used by subcontractors;

Periodically informing management of the status of the QA Program;

Seeking out and evaluating new ideas and current developments in the field of QA and recommending means for their application where advisable;

Implementing or modifying analytical methods to conform with recognized standards and/or fundamentals of Good Laboratory Practices, including alteration of analysis/procedure codes used by the Computerized Laboratory Management System (CLMS);

Final authority to terminate or alter any incorrect or improper analytical or measurement procedure in order to conform to requirements of the QA Plan;

Training, directing and qualifying personnel in specified laboratory QC and analytical procedures, or designating qualified individuals to do so;

Review and advise lab management on requirements and applicability of specific Quality Assurance Project Plans (QAPPs), new statements-of-work (SOWs), RFQs, RFPS, IFBs, and other contract-related issues.

Reviewing corrective action reports for out-of-control events and verifying that remedial action has been taken to restore control;

Assuring that subcontractor laboratories are complying with the QA Program;

Serving as point-of-contact for exchange of QA/QC information and approving, along with the Laboratory Director, release of QA/QC information;

Direct the activities of the QA Department technical staff and Communications Specialist.

Manager of Quality Assurance

The Manager of Quality Assurance reports to the Director of Quality Assurance and functions independently from the laboratory technical program. The Manager is primarily responsible for overseeing and directing the activities of the Quality Assurance staff, consisting of Sr. QA Specialists, QA Specialists, Sr. Standards Chemists, and clerical staff. The Manager of QA, the QA Specialists and the Sr. QA Specialists have authority to approve data. Other than these individiuals, only the Final Tehcnical Reivewer has the authority for final data approval.

Additional responsibilities and duties include:

Provide QA reports to management, as described in Section 10.0;

Oversee the laboratory's participation in external QA/QC programs;

Coordinate external (on-site) or internal QA/QC audits or inspections;

Review and approve all laboratory-generated data qualifying notices;

Write Quality Assurance Notices, used to document exceptions to QC acceptance criteria or other matters affecting data useability or interpretation, for inclusion in data packages;

Provide training to QA and laboratory staff;

Provide assistance on special projects as required by the QA Director;

Oversee all subcontractor QA Programs, including administration of subcontractor proficiency studies and on-site audits;

Assume the responsibilities of the QA Director, if necessary.

Quality Assurance Staff

The Quality Assurance Staff is responsible for carrying out quality activities as directed by the QA Manager and QA Director. All members of the Quality Assurance staff function independently from the laboratory technical programs.

Additional responsibilities and duties include:

Create and revise SOPs, including integration into proper document control format, maintaining a chronology and distributing SOPs to laboratory stations;

Ensure that the laboratories meet all requirements as documented in the QA Plan, as well as the specific QA and lab SOP manuals;

Audit and spot-check work in process for quality and completeness;

Provide deviation/exception reports to laboratory managers and the QA Director regarding out-of-control analyses and provide recommendations for corrective action;

Oversee corrective action as required;

Generate, analyze and document QC/QA data (much of the QC data is generated by the laboratory staff in the normal course of producing analytical data, or by using the CLMS during data acquisition or data entry);

Based upon laboratory performance statistics and/or statement-of-work requirements, establish and update control limits using QC data from routine sample analyses;

Provide information and documentation for internal/external audits or inspections;

Function as a liaison between the QA Manager/Director and personnel within the laboratories;

Communicate QA Program objectives and requirements to clients and external auditors;

Reinforce GLP within the laboratory;

Communicate any quality concerns to the QA Manager/Director;

Communicate any safety concerns to the Manager of Facilities and Safety;

Review and approve Performance Evaluation (PE) and Proficiency Testing (PT) sample data;

Review PE and PT scores, coordinating lab personnel review of unacceptable scores and associated data, and assembling findings into unified document for response to certifying agency;

Initiate and document corrective action (if necessary) related to unacceptable scores and associated data, and assembling findings into unified document for response to certifying agency;

Initiate and document corrective action (if necessary) related to unacceptable PE/PT scores or audit deficiency reports;

Introduce internal "blind" PE samples into the CLMS (or mailing to subcontractor labs) and report performance to management (this includes both initial and continuing certification of performance, and is required of all subcontractor laboratories);

Document approval and traceability of all standards to NBS, USEPA or other certified source;

Document calibration traceability of all thermometers, balances and Class-S weights used in daily calibrations;

Audit the Environmental Site Profile (remote reporting) database, summarizing findings in reports to management;

Conduct routine Performance Audits and System Audits, summarizing findings in report to management;

Audit computer output tabulating all keycard accesses to restricted areas, summarizing findings in report to management.

Laboratory Personnel and Management

A variety of Quality Control functions and duties directly or indirectly affecting data quality are performed by laboratory personnel and management. Key responsibilities of this nature include:

Perform scheduled, routine preventative maintenance or instruments or oversee work done under service contracts;

Assure compliance with methods and SOPs and as written as directed by the Quality Assurance Director;

Verify that all instruments meet calibration and tuning requirements;

Respond to corrective action requirements;

Follow GLPs and recommendations of the QA Department for improving quality and safety;

Perform and document action steps based on established QC acceptance criteria;

Provide adequate and documented training of personnel.

## 4.3 Communications

The Quality Assurance Department communicates to other areas of the laboratory and to Management via several different types of reports, the key QA reports as described in Section 10.0 . The Director of QA and the QA staff also distribute inter-office memoranda to appropriate laboratory management detailing the results of internal and external audits, blind interlaboratory proficiency studies, blind internal proficiency studies, and deficiency reports/corrective action needs. Good Laboratory Practices and successful performance on various studies and audits are also reinforced through these memoranda.

Section 10.0 provides additional details regarding the monthly and quarterly reports distributed to all laboratory managers and appropriate senior and executive staff.

## 4.4 Document Control

Document control procedures, as described in section 1.4.1 of the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I (EPA-600/9-76-005), are used in the production of the QA Plan and other documents vital to the operation of the laboratory. This document control system, described in the QA SOP Manual, includes official distribution lists, a historical file of all updated standard operating procedures, and appropriate signature levels for the ensurance of correct methods, procedures and techniques.

## 4.5 Personnel Qualifications

CompuChem, located in Research Triangle Park, NC, and within minutes of three major university campuses, is ideally positioned for recruiting both degreed scientists and experienced professionals. Many applied science graduates join the organization as entry-level technicians, and progress through extensive training into senior chemist, data review/validation, and QA positions. In most technician positions and instrument operator positions, the training period lasts from six months to one year, depending on the level of experience required and complexities of the position/instrumentation.

Job descriptions, including required educational, training and experience qualifications, are incorporated into individual personnel records and are available for review in the Human Resources Department. These records also serve to document additional cross-training and participation in continuing education programs.

QA Plan users are referred to Section 17.0 for resumes of key personnel. Also included in this are the USEPA's requirements for qualifications of technical personnel involved in the analysis of EPA samples. CompuChem's technical personnel meet or exceed these requirements in all cases. Suitable staff are nominated in the QA SOP to deputize for senior technical staff and QA management in their absence.

Figure 4-2

# Organization Chart

## Role of QA in Env. Lab Operations

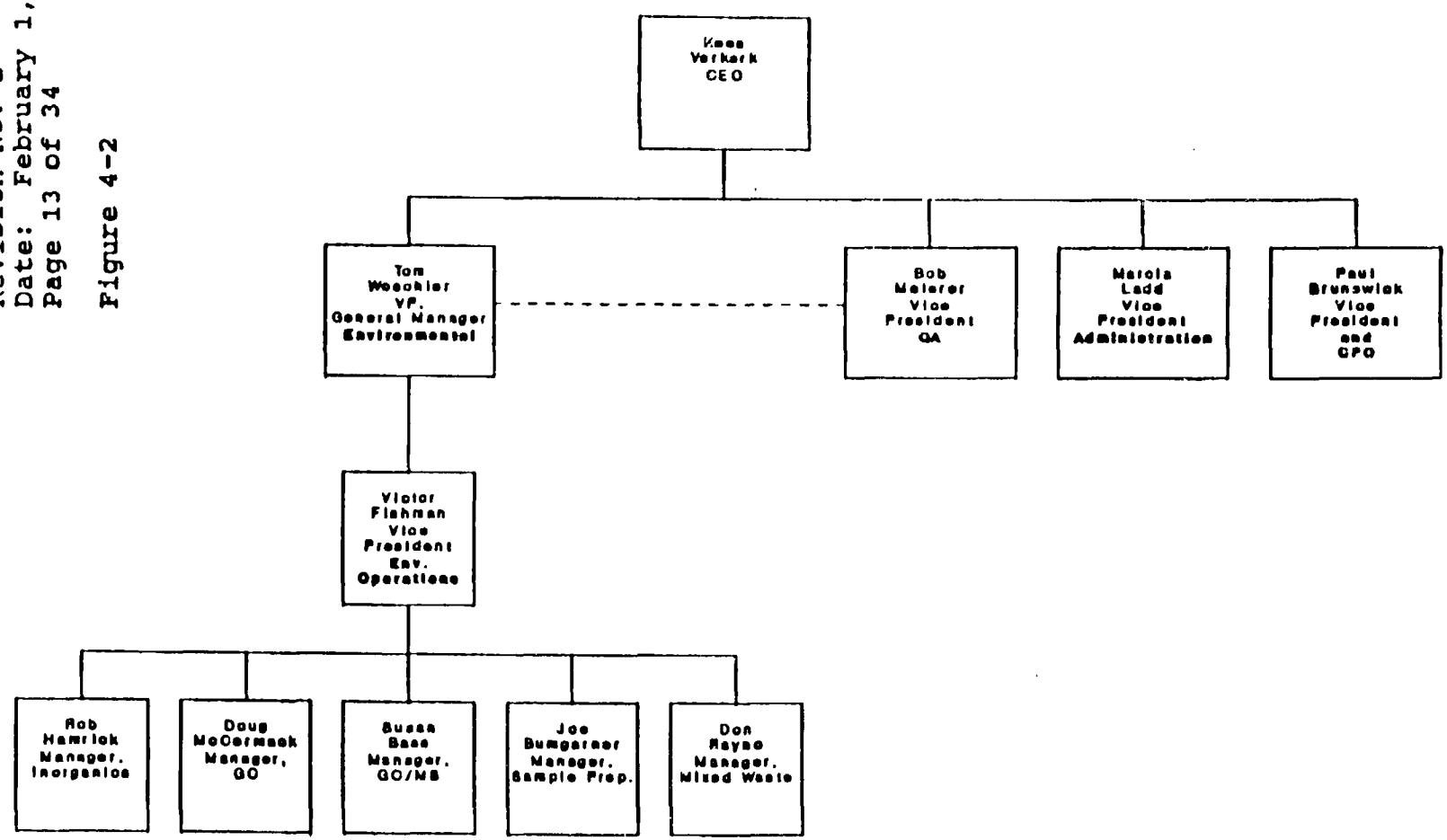


Figure 4-3

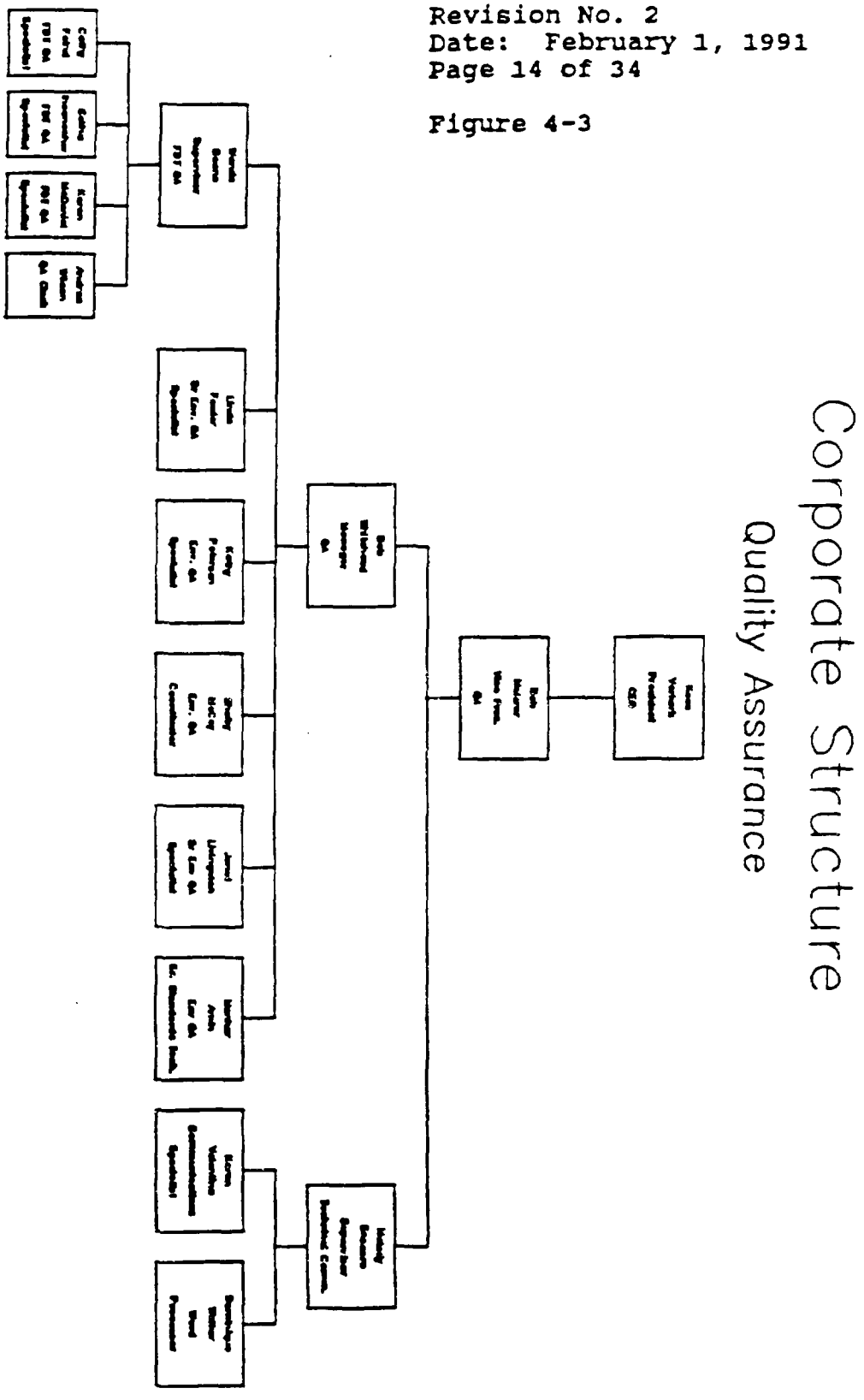


Figure 4-4

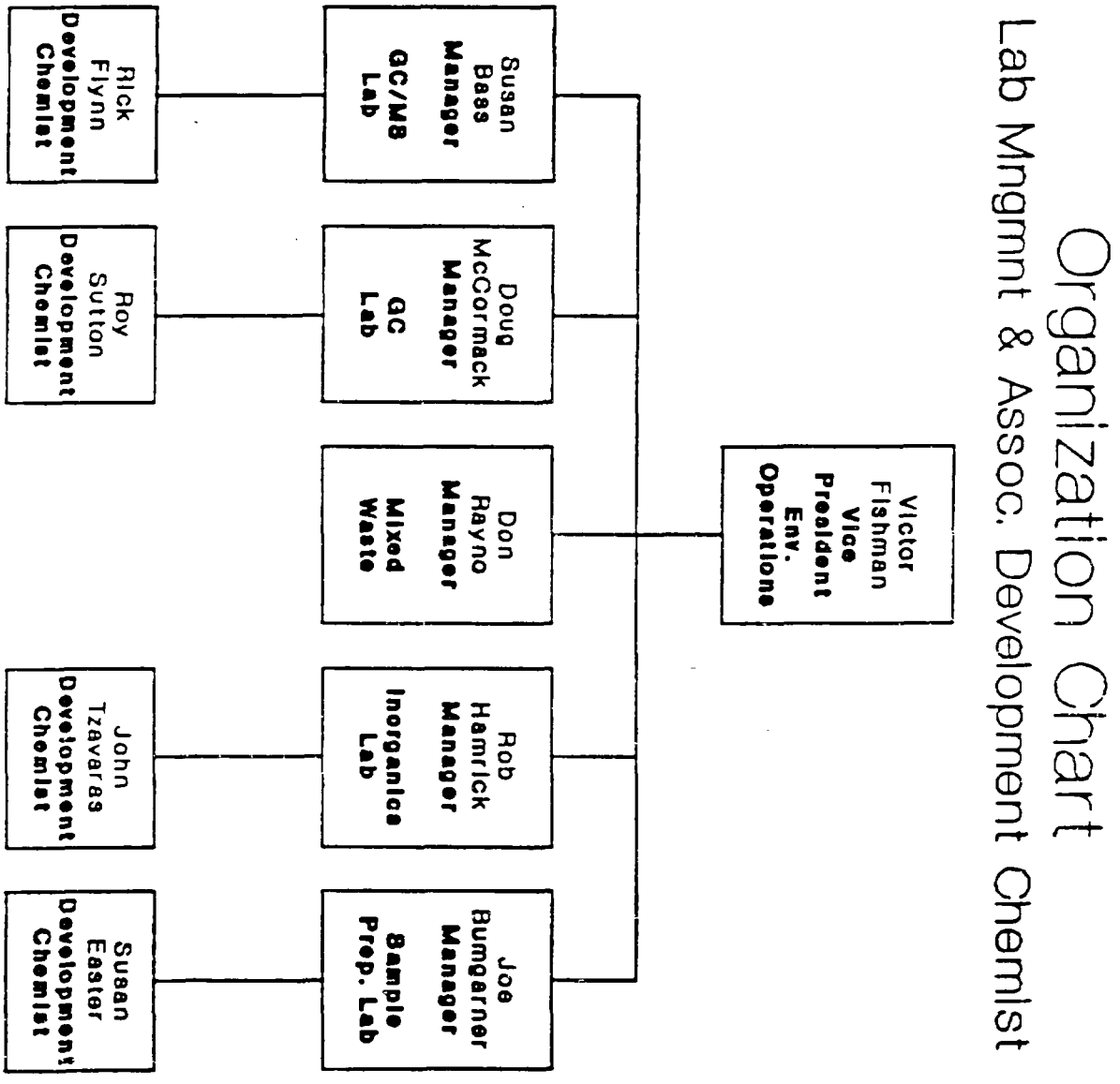

Organization Chart
Lab Mngmnt & Assoc. Development Chemist
Victor Fishman Vice President Env. Operations
Susan Bass Manager GC/MS Lab
Doug McCormack Manager GC Lab
Don Rayno Manager Mixed Waste
Rob Hamrick Manager Inorganics Lab
Joe Bumgarner Manager Sample Prep. Lab
Rick Flynn Development Chemist
Roy Sutton Development Chemist
John Tzavaras Development Chemist
Susan Easter Development Chemist

Figure 4-5

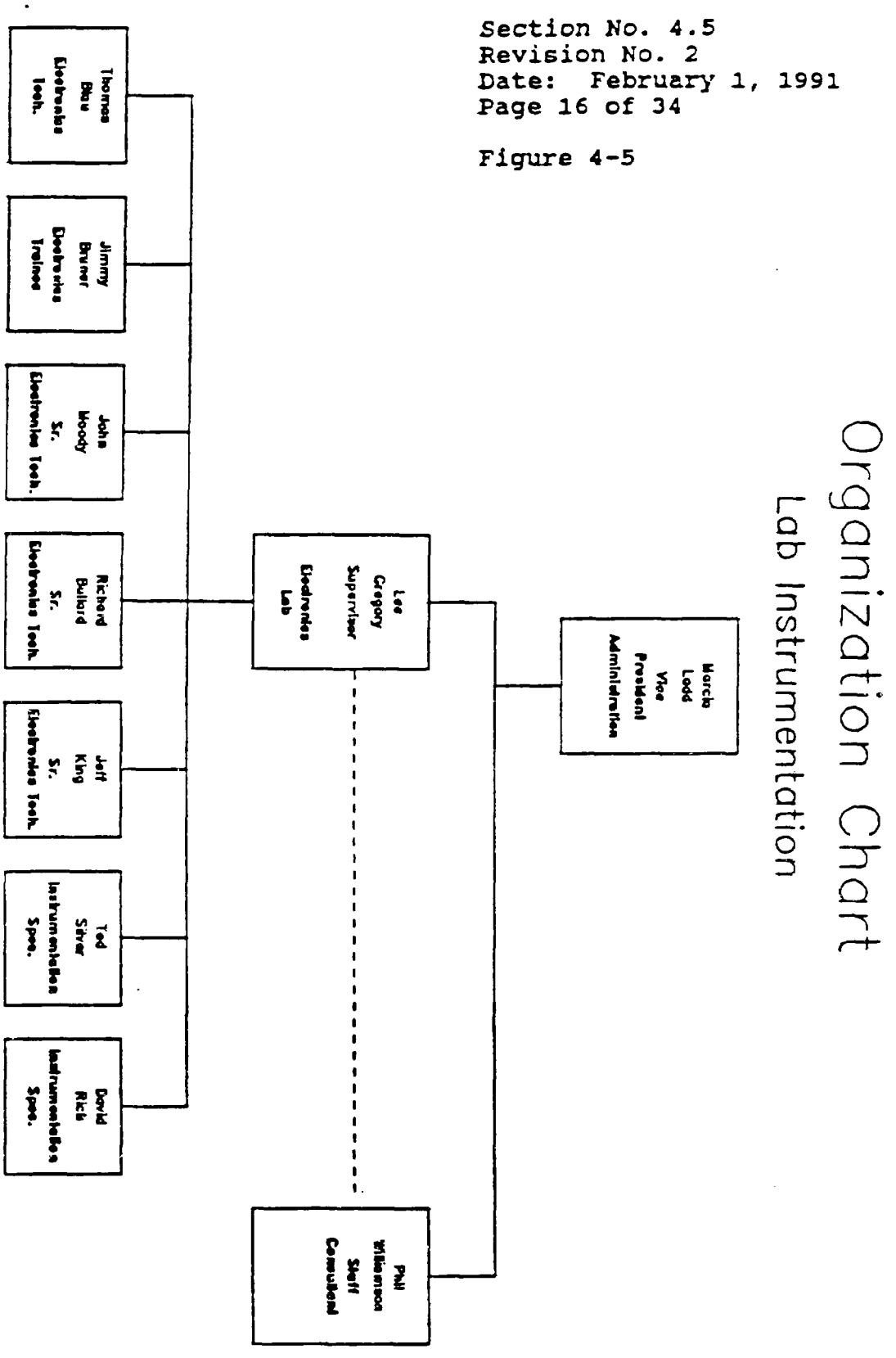

Organization Chart
Lab Instrumentation
Marcia Ladd Vice President Administration
Lee Gregory Supervisor Electronics Lab
Phil Williamson Staff Consultant
Thomas Blue Electronics Tech.
Jimmy Bruner Electronics Trainee
John Moody Sr. Electronics Tech.
Richard Bullard Sr. Electronics Tech.
Jeff King Sr. Electronics Tech.
Ted Silver Instrumentation Spec.
David Rich Instrumentation Spec.

Figure 4-6

# Organization Chart

## Environmental Operations

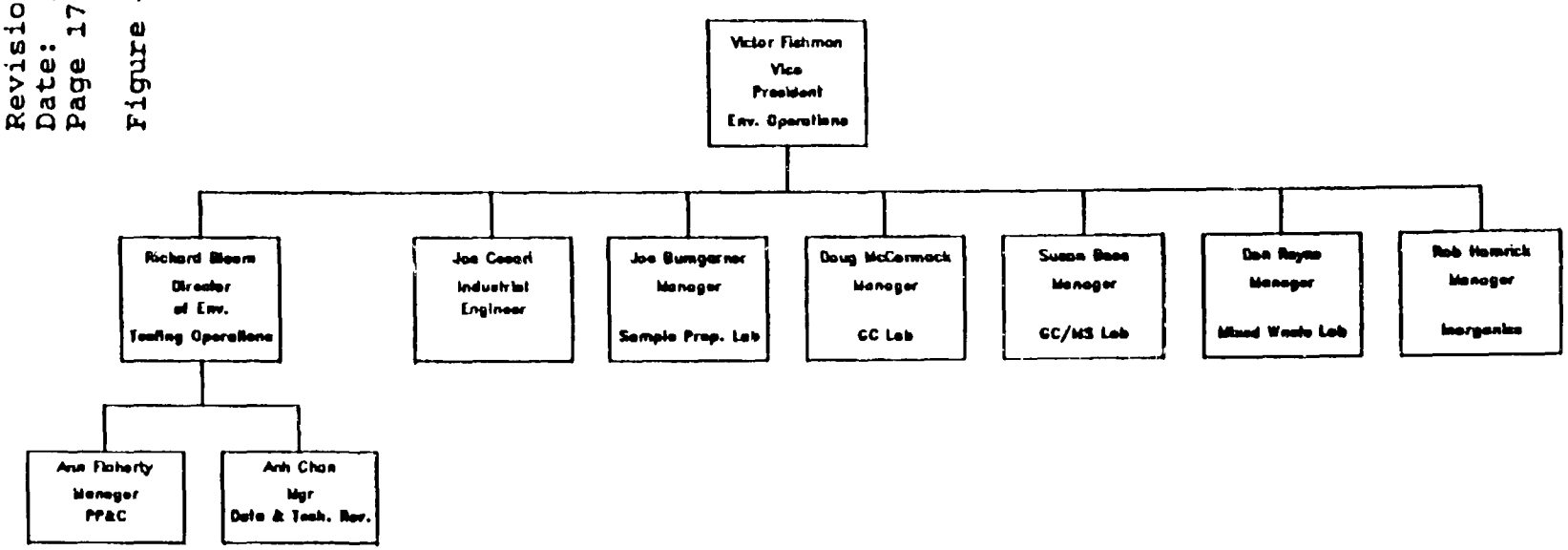
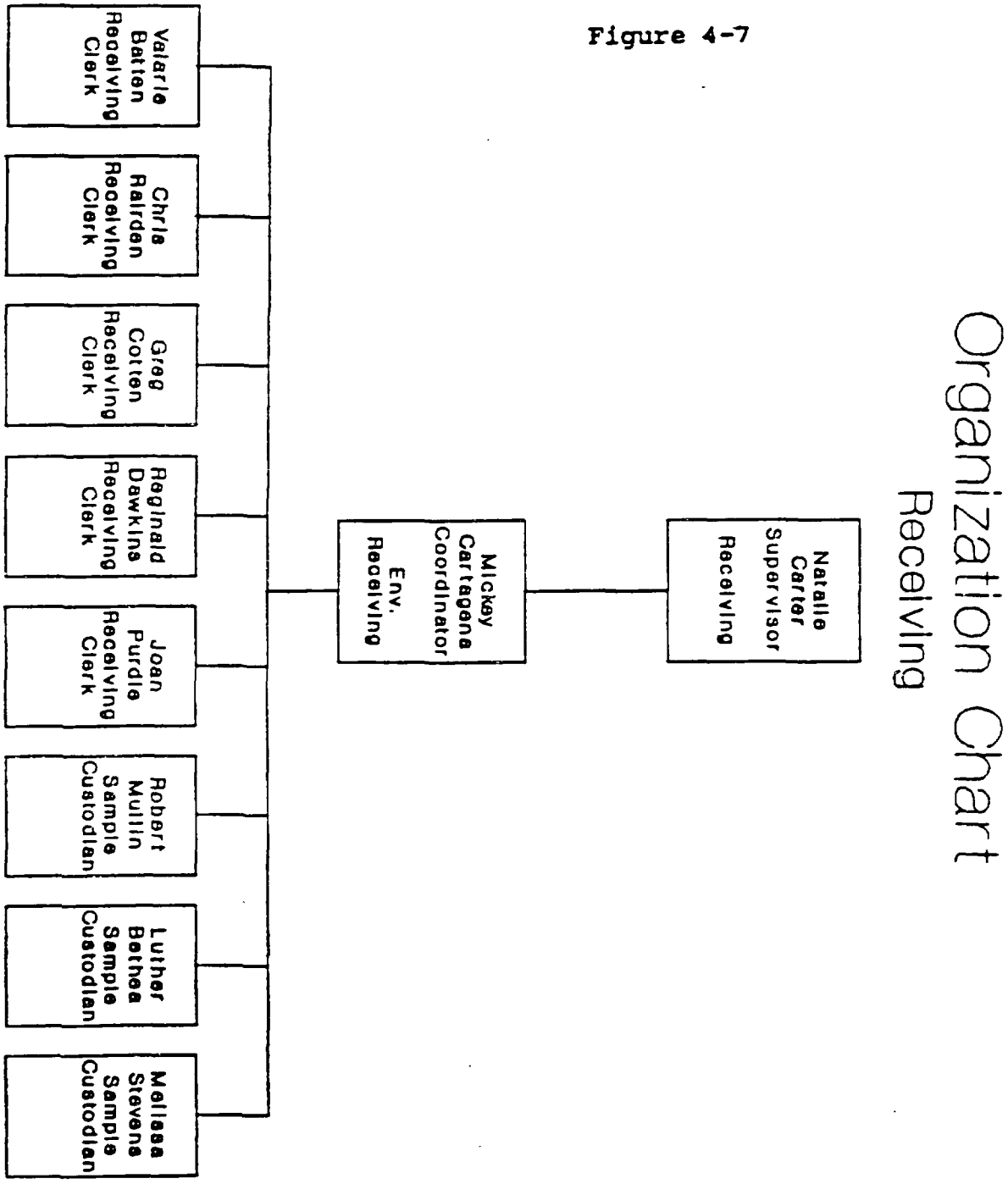


Figure 4-7

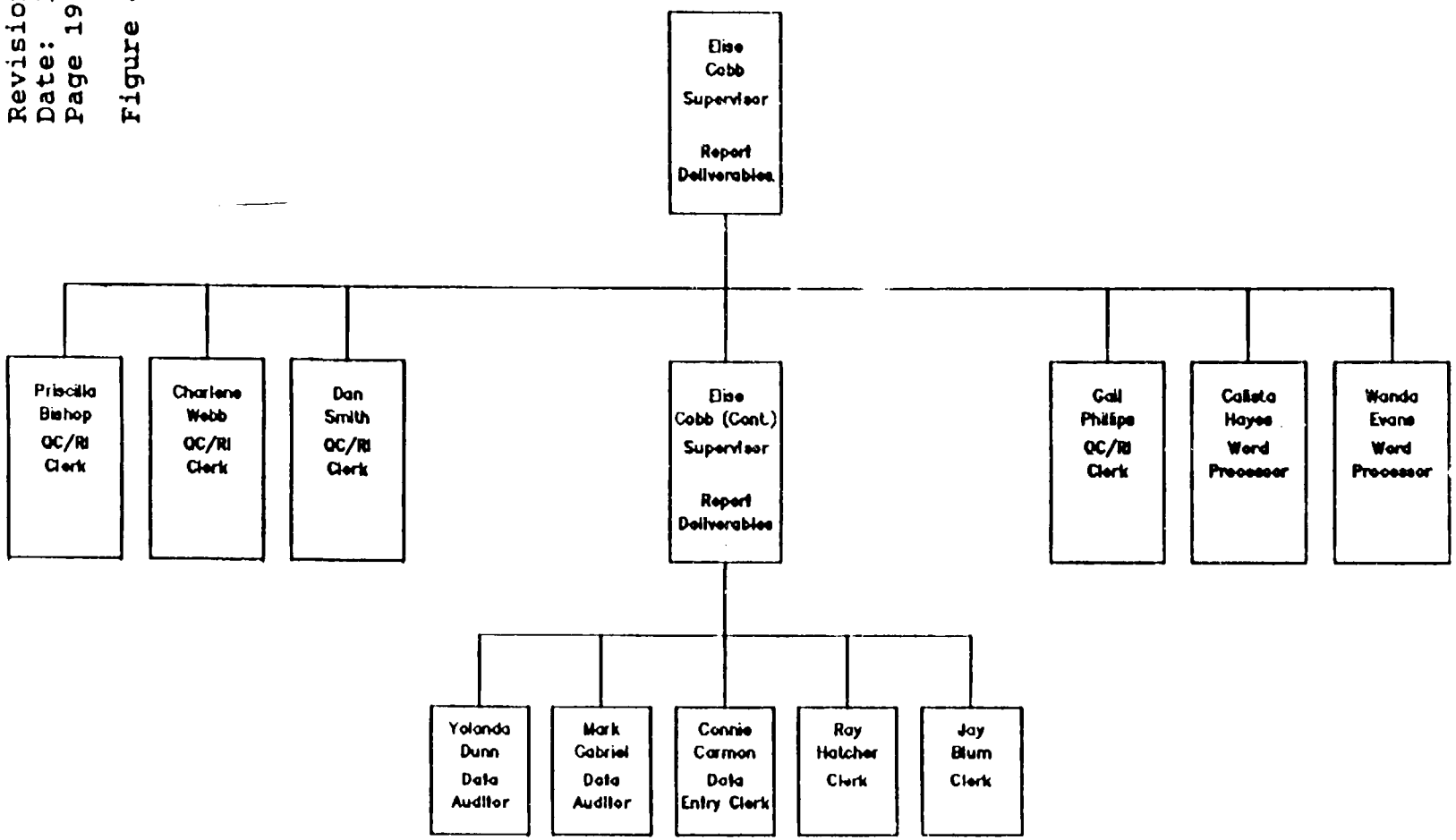



January 1991

Figure 4-8

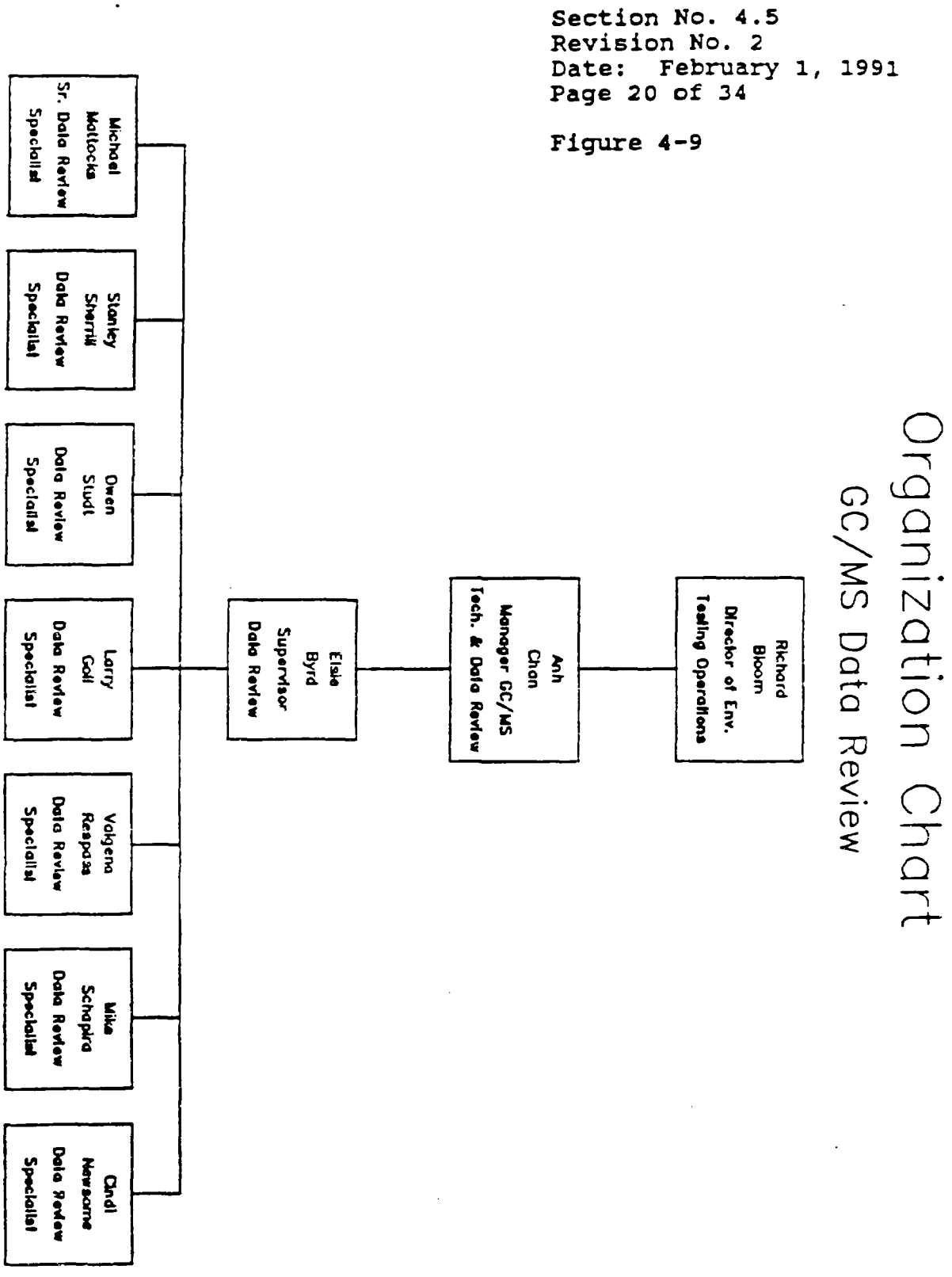
# Organization Chart

## Report Deliverables




January 1991

Figure 4-9




January 1991

Figure 4-10

# Organization Chart

## Final Technical Review

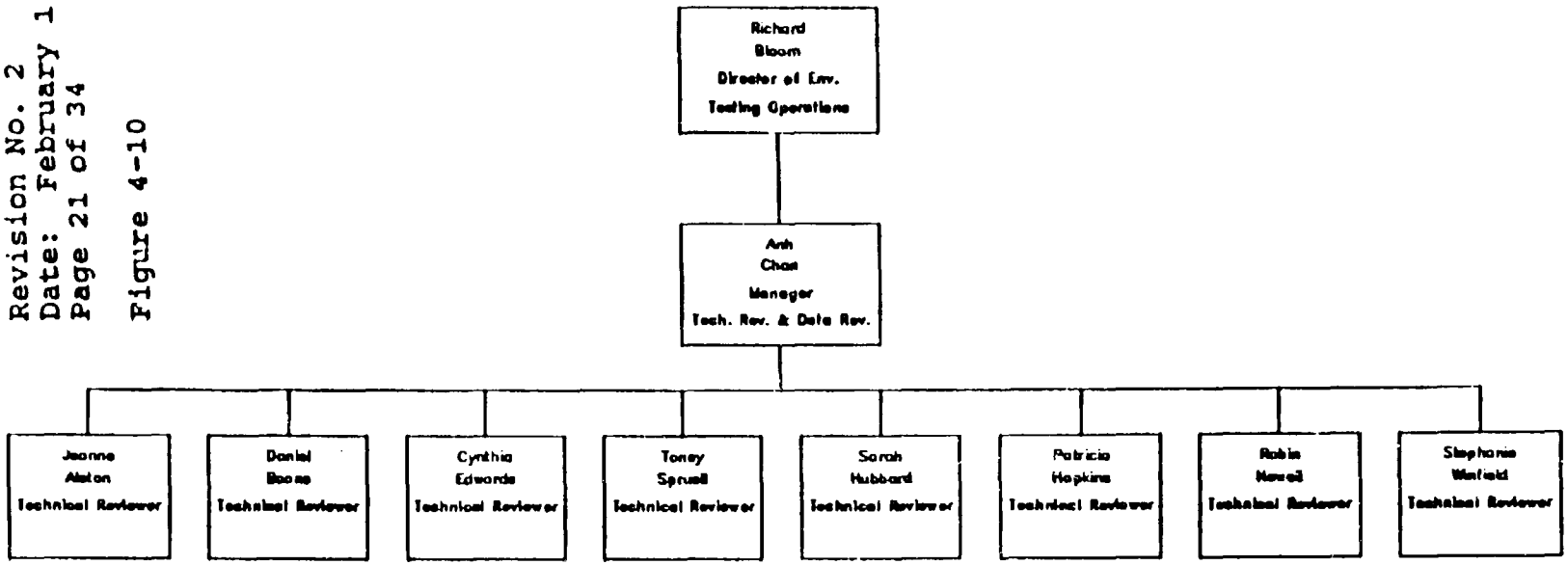
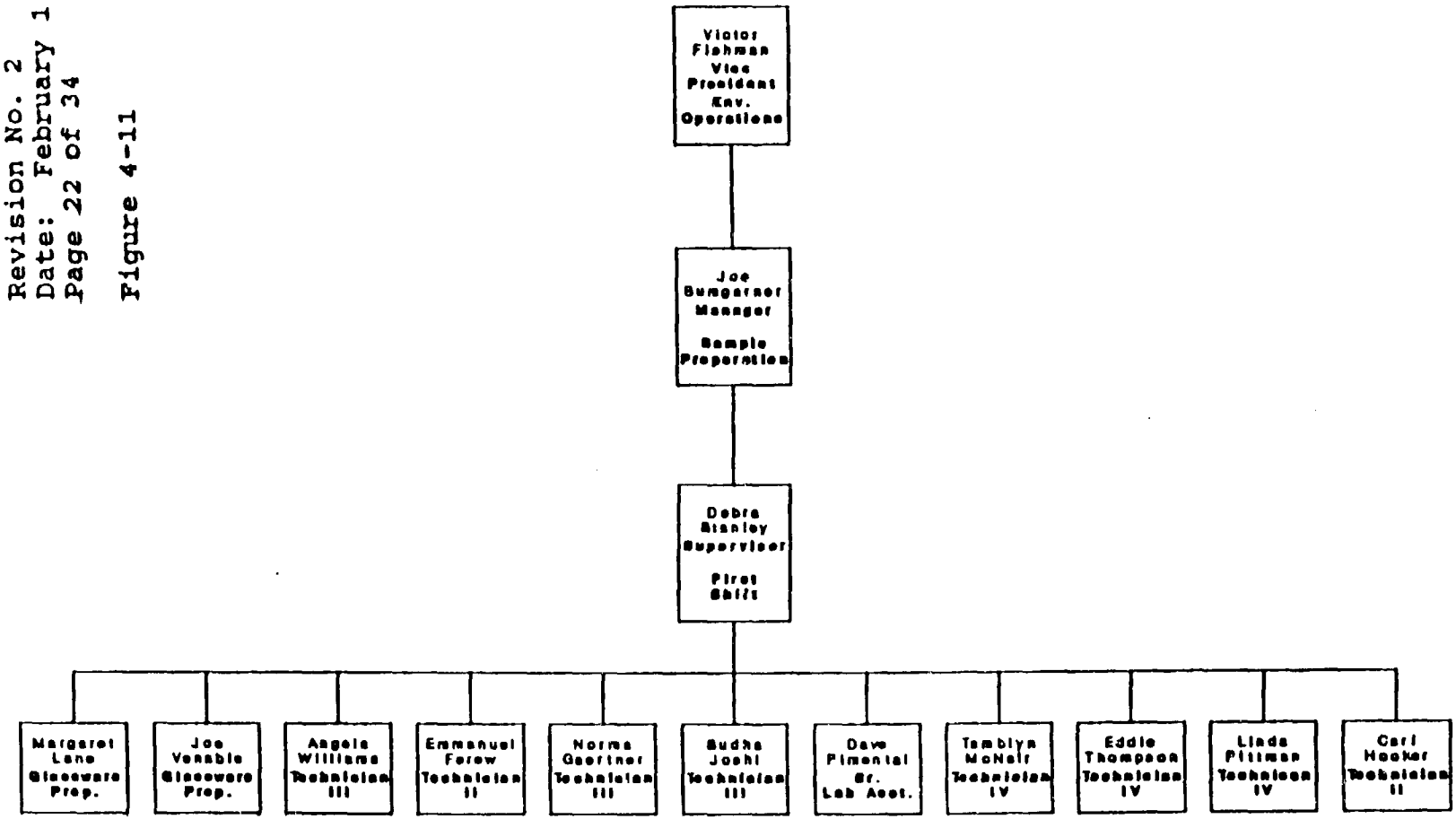


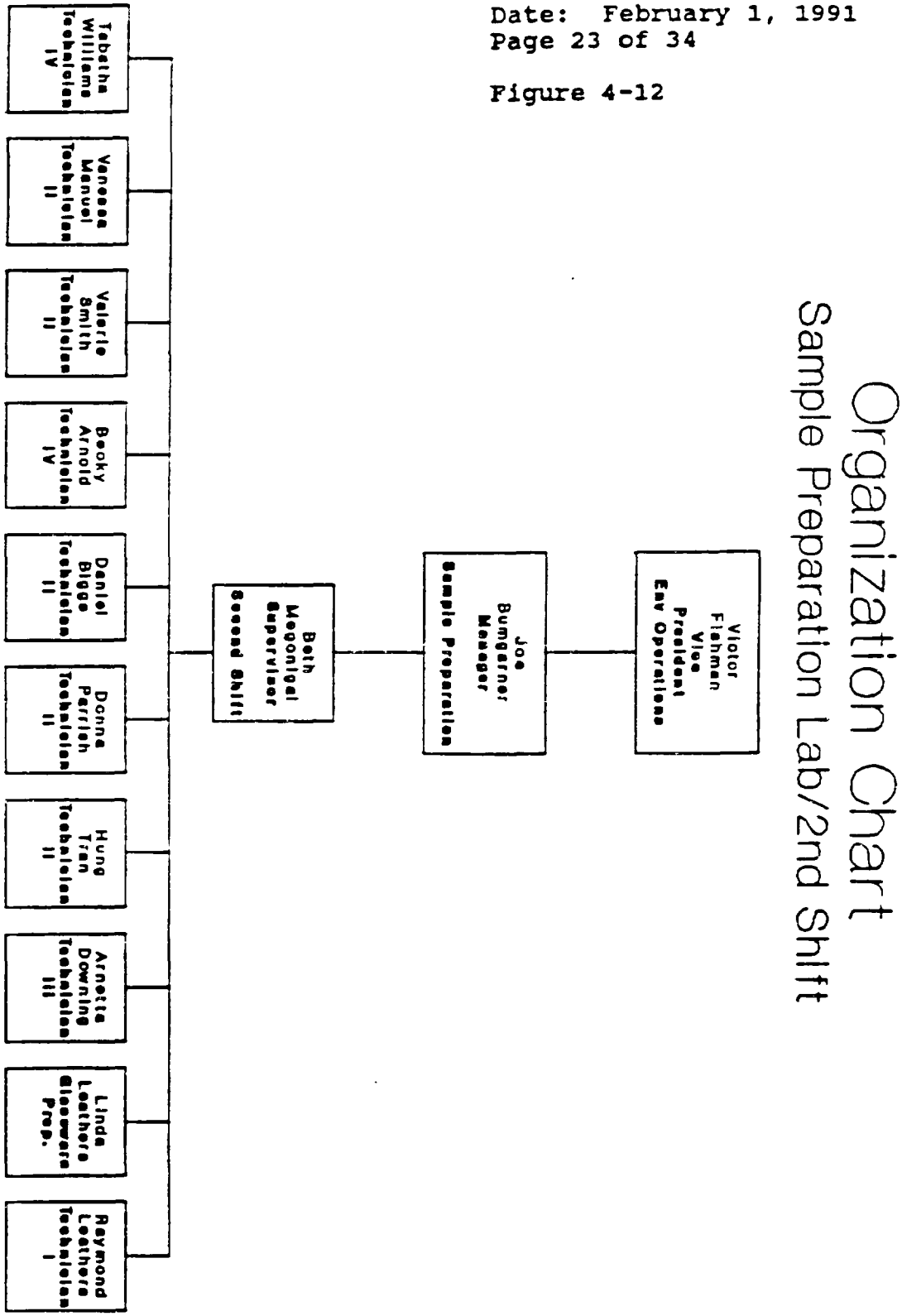
Figure 4-11

# Organization Chart
## Sample Preparation Lab/1st Shift




January 1991

Figure 4-12


Organization Chart
Sample Preparation Lab/2nd Shift
Victor Fishman
Vice President
Env Operations
Joe Bumgarner
Manager
Sample Preparation
Beth Mcgonigal
Supervisor
Second Shift
Tabatha Williams
Technician IV
Vanessa Manuel
Technician II
Valerie Smith
Technician II
Becky Arnold
Technician IV
Daniel Biggs
Technician II
Donna Parrish
Technician II
Hung Tran
Technician II
Arnetta Downing
Technician III
Linda Leathers
Glassware Prep.
Raymond Leathers
Technician I

Figure

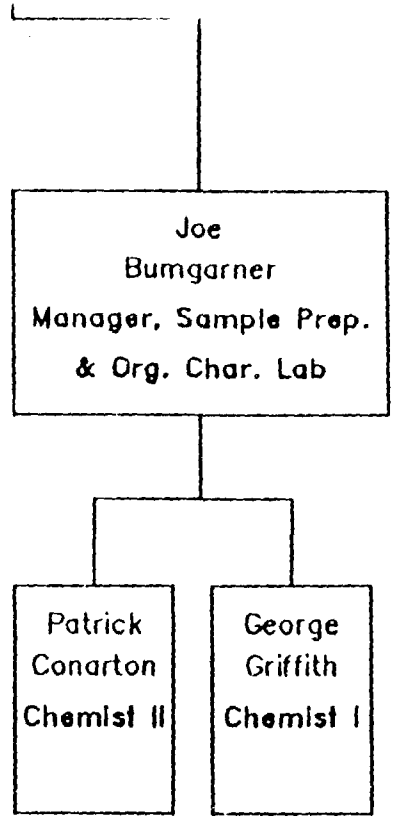
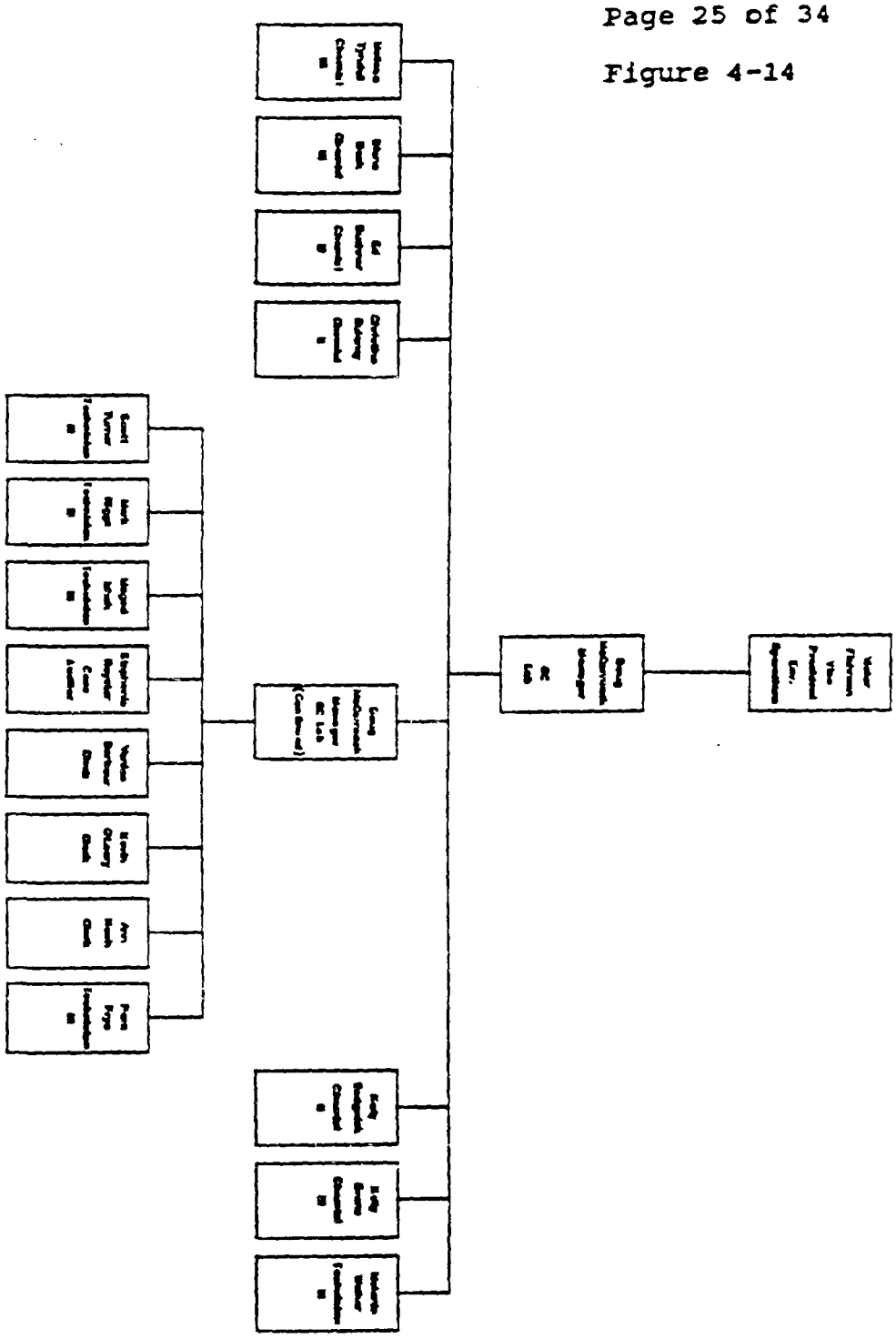

Joe
Bumgarner
Manager, Sample Prep.
& Org. Char. Lab
Patrick
Conarton
Chemist II
George
Griffith
Chemist I

Figure 4-14



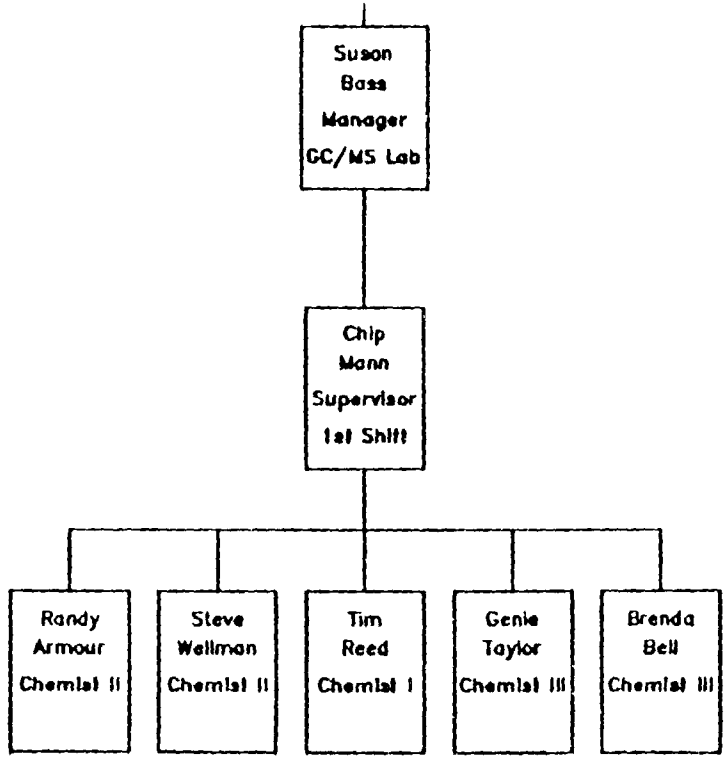

Susan
Bass
Manager
GC/MS Lab
Chip
Mann
Supervisor
1st Shift
Randy
Armour
Chemist II
Steve
Wellman
Chemist II
Tim
Reed
Chemist I
Genie
Taylor
Chemist III
Brenda
Bell
Chemist III

Figure 4-16

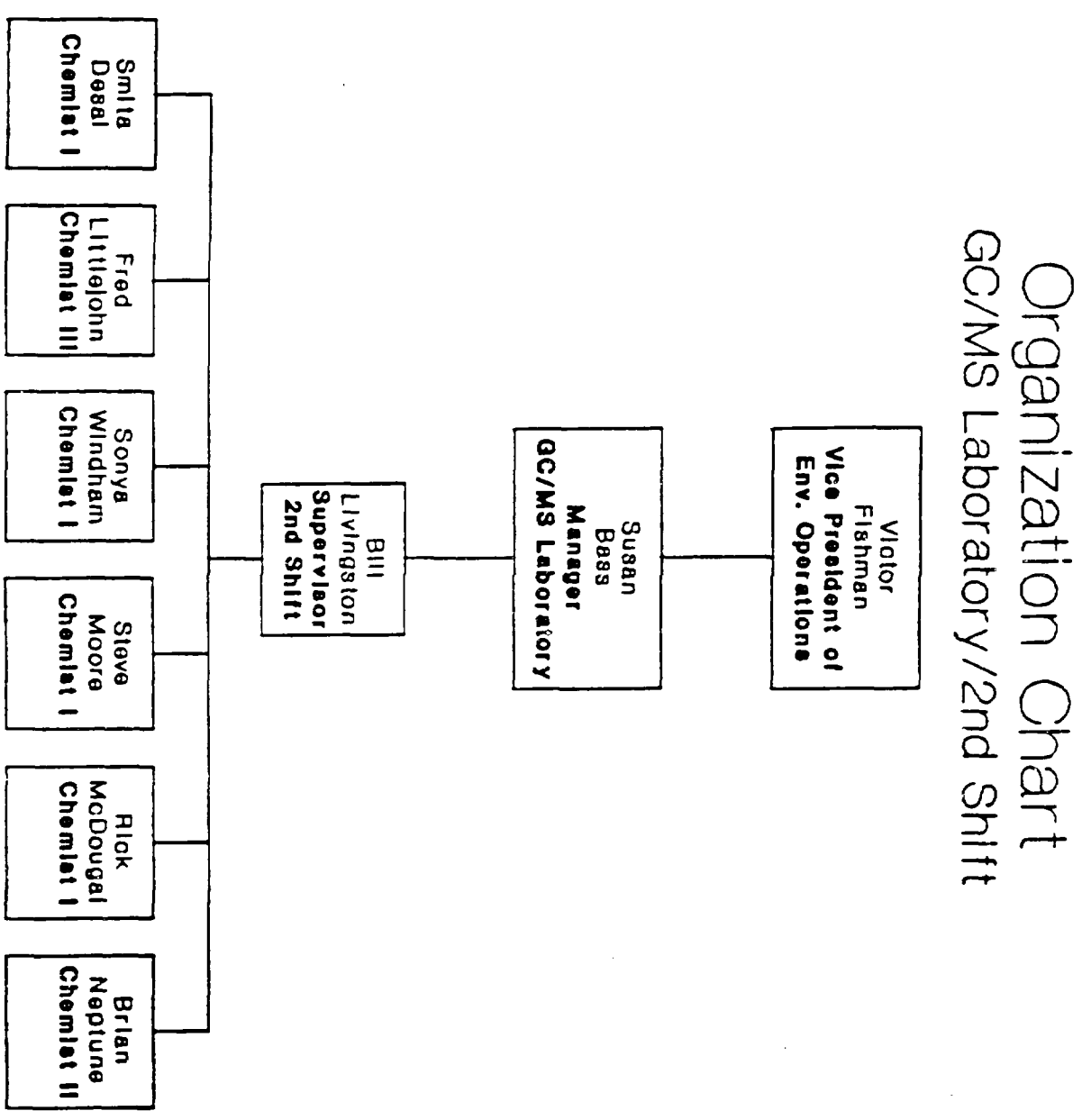

Organization Chart
GC/MS Laboratory/2nd Shift
Victor Fishman
Vice President of Env. Operations
Susan Bass
Manager GC/MS Laboratory
Bill Livingston
Supervisor 2nd Shift
Smita Desai
Chemist I
Fred Littlejohn
Chemist III
Sonya Windham
Chemist I
Steve Moore
Chemist I
Rick McDougal
Chemist I
Brian Neptune
Chemist II

Figure 4-17

# Organization Chart
## GC/MS Laboratory/3rd Shift, Weekends

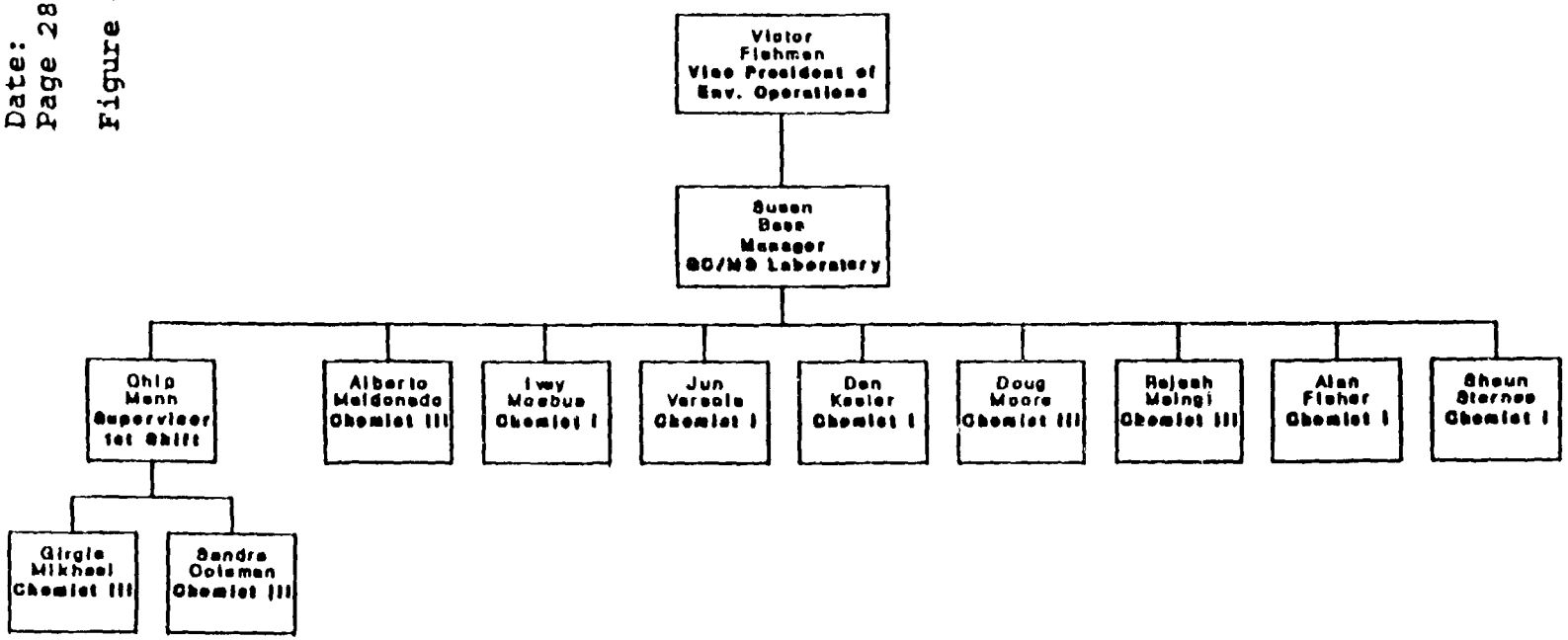


Figure 4-18

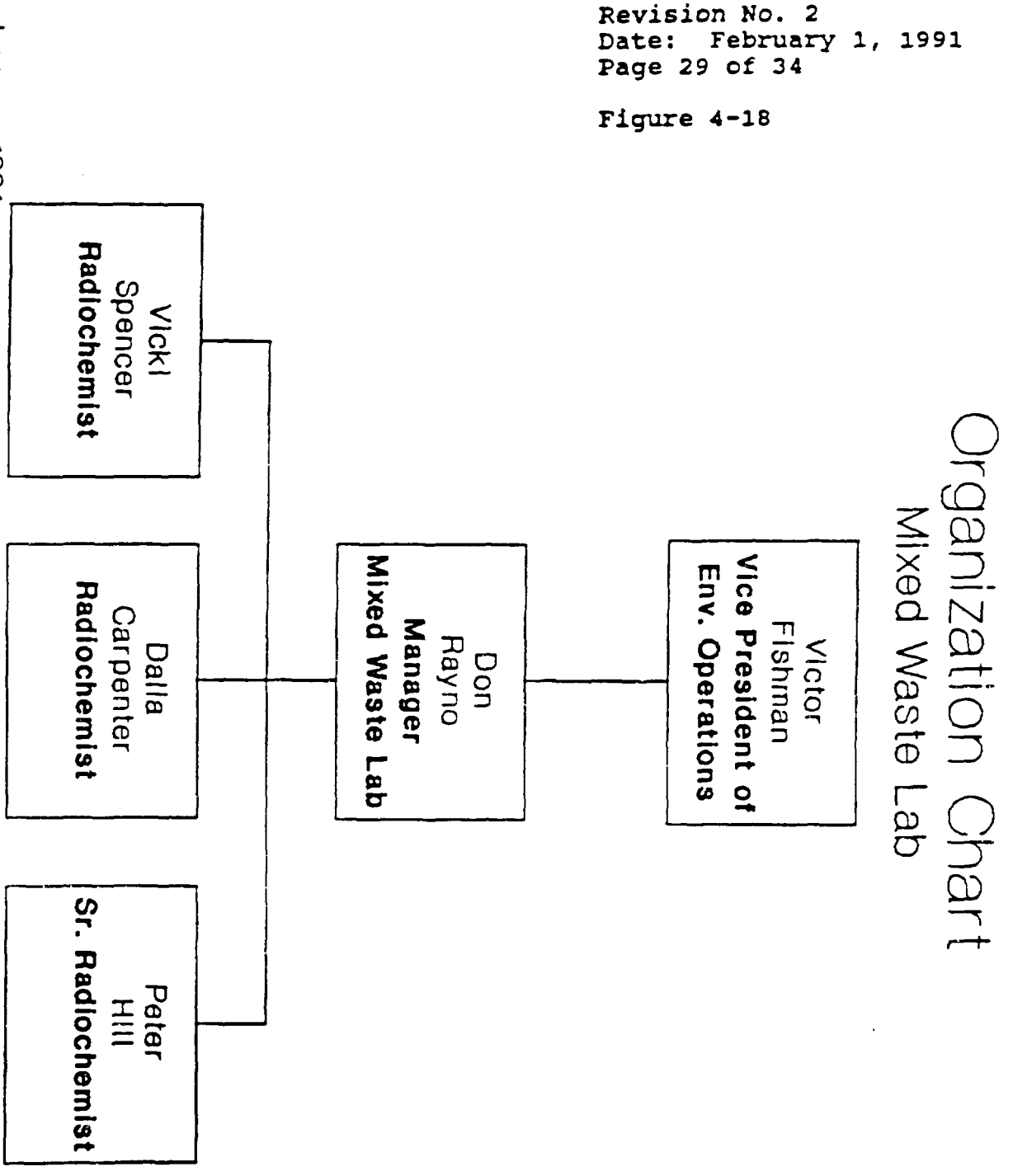
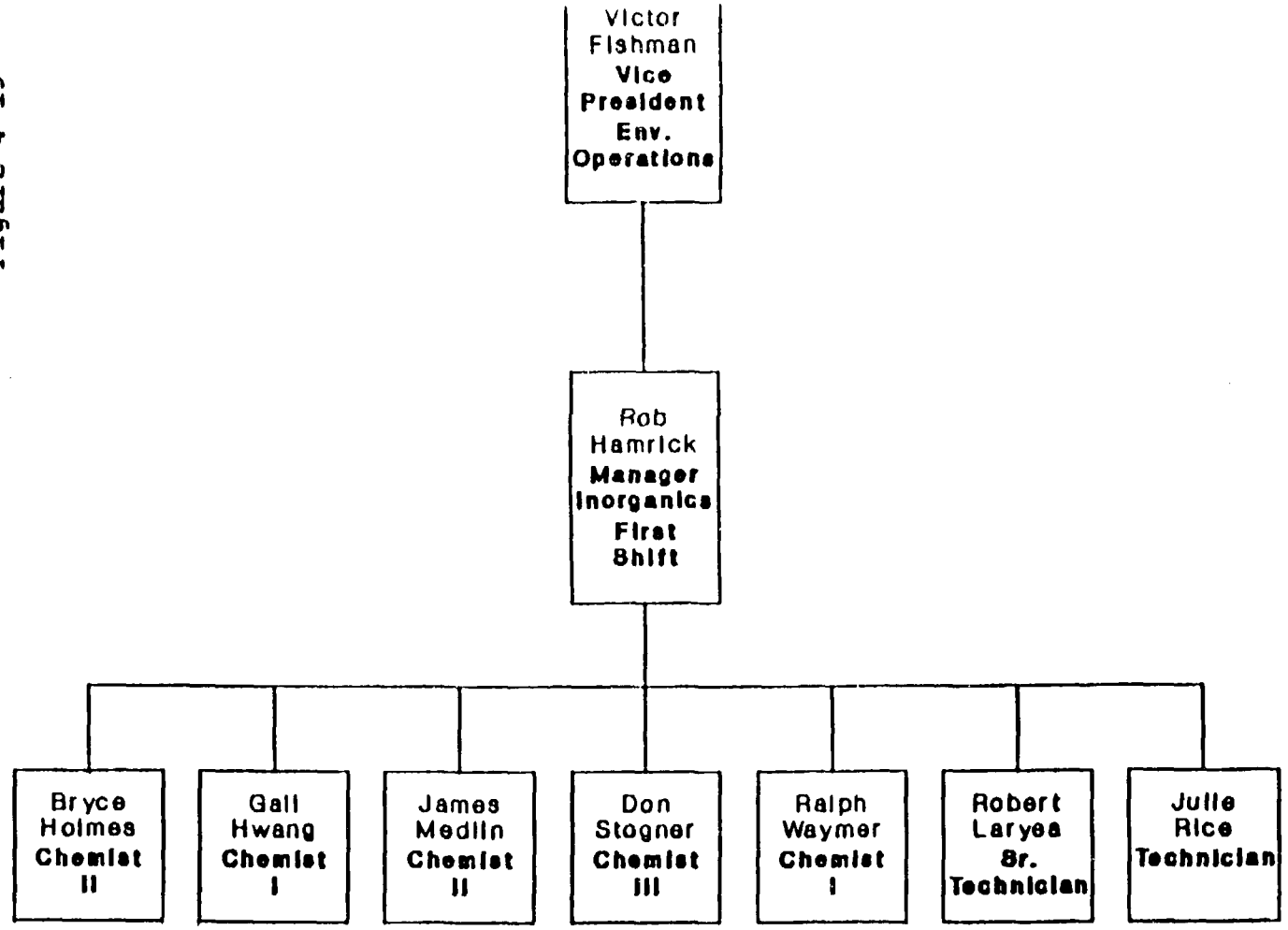
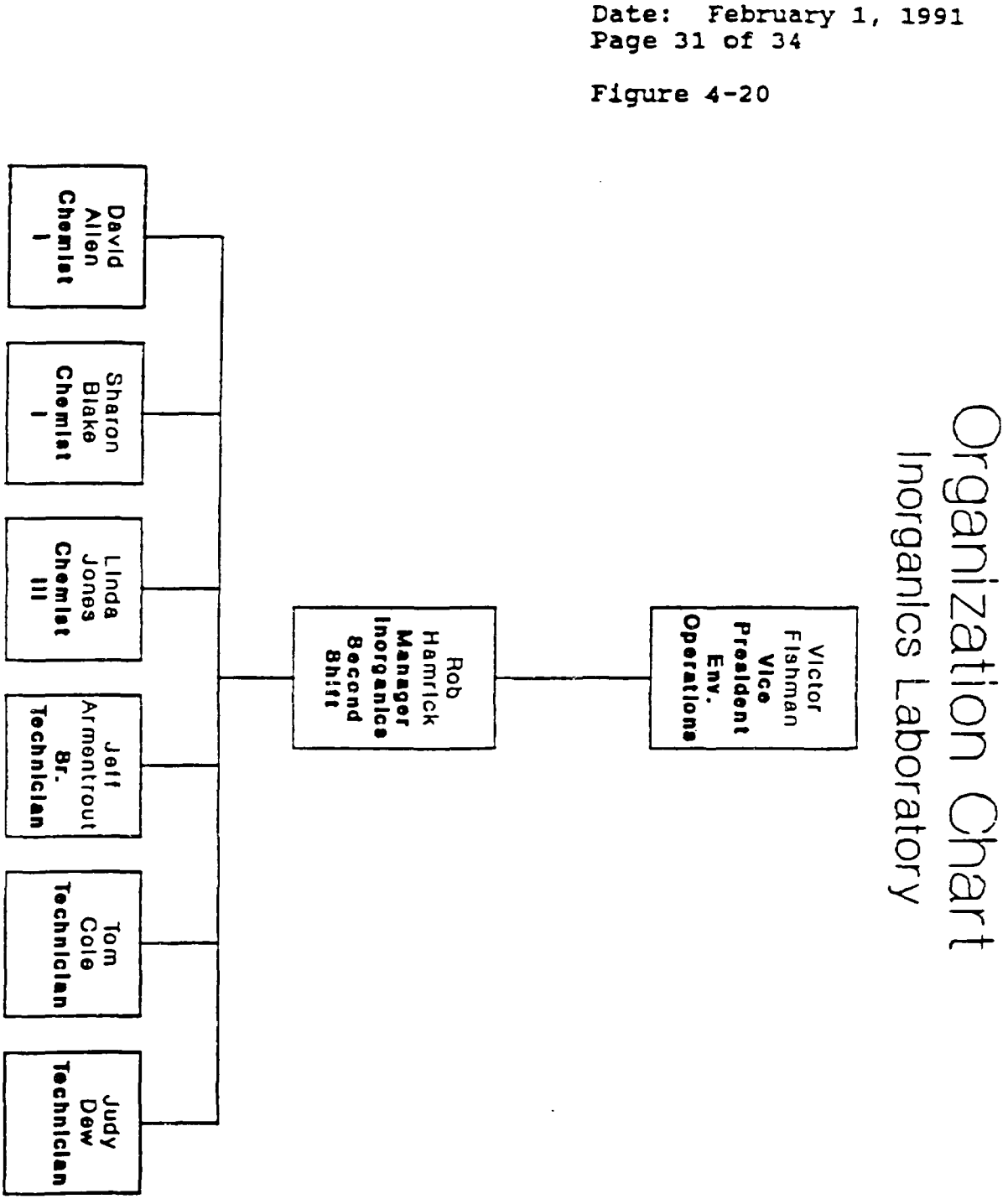

Organization Chart
Mixed Waste Lab
Victor
Fishman
Vice President of
Env. Operations
Don
Rayno
Manager
Mixed Waste Lab
Vicki
Spencer
Radiochemist
Dalla
Carpenter
Radiochemist
Peter
Hill
Sr. Radiochemist

Figure 4-19




January 1991

Figure 4-20




January 1991

## 4.5 QA Program Assessment

The Director of QA and the department staff conduct periodic assessments of the total QA program. Based on these assessments, a written status report of QA activities and progress is forwarded to management. The following items are addressed in these reports, most being addressed in the Monthly QA Activities Report to management as described in Section 10.0. All items are addressed at least semi-annually in the QA report to the CEO.

1. Status of or changes to QA Program Plan
2. Status of QA Project Plans, if any
3. Measures of data quality
4. Significant QA problems, accomplishments, and recommendations
5. Results of performance audits
6. Results of system audits
7. Status of QA requirements for contracts
8. Summary of QA training (internal and external QA/QC seminars and courses)

Chain-of-Custody

The basic components for maintaining sample chain-of-custody (C-O-C) are:

1) samples must be relinquished into the possession of an authorized laboratory staff member, or

2) samples must be within the authorized staff member's line-of-sight, or

3) samples must be locked in a secured storage area with restricted access.

Furthermore, any change of possession or custody must be documented on appropriate chain-of-custody forms. This documentation must include both the initials of the individual relinquishing the sample and those of the individual receiving the sample, as well as the date of the custody transfer.

CompuChem® accomplishes these objectives through an elaborate document control system. This system includes procedures for documentation of the receipt of the sample into the laboratory using preprinted, numbered chain-of-custody records (although many clients provide their own C-O-C records which suffice). These records include information about the individuals collecting the samples, the collection date, time and location, and the type of analyses required. CompuChem's clients are responsible for field chain-of-custody, sample collection, handling and shipping.

When the samples are received in the laboratory, the C-O-C documents are signed and dated by the Receiving Clerk. The samples are logged into the Computerized Laboratory Management System (CLMS), and assigned unique sample identification numbers. The samples are then relinquished to the possession of a Sample Custodian, who has sole access to the locked sample storage refrigerators. The CLMS schedules the appropriate analyses and tracks the progress of sample

processing throughout the laboratory. Samples do not remain outside refrigeration more than 2 hours from receipt.

The custody of each sample can be determined at any point in time by reviewing Scheduling Details within the CLMS. A "paper trail" also accompanies the movement of the sample (or extracts, aliquots or digestates created from the as-received sample) throughout the lab, serving to document internally all changes in custody.

The integrity of the samples within the laboratory is assured by the security of the facility itself. Building security is controlled by an electronic card entry system. The exterior doors and the doors of various controlled-access areas are equipped with card readers. Each memeber of the staff has an access card, which must be prominently displayed on their person, that is coded only for those areas where their job functions require access. The system also maintains a record of the movements, or attempted movements, of the staff throughout the building. A computer printout of this record is audited by a member of the Quality Assurance Department for verification of card coding and card entry transactions.

When the analysis is complete, the final extracts (for extractable portions of the sample) are kept in a locked freezer (if required) under sole custody of the Sample Custodian.

A complete description of CompuChem's sample tracking procedures and additional chain-of-custody details can be found in the Production, Planning and Control SOPs.

# APPENDIX F

## Drinking Water Requirements

# APPENDIX F

## Drinking Water Requirements

Samples identified by the client as "Drinking Water Samples" (i.e., for drinking water compliance monitoring) require certain special handling and reporting procedures, but are otherwise handled by the Computerized Laboratory Management System (CLMS) in much the same way as non-compliance samples.

The Sales Representatives in the Marketing Department are responsible for placing the order in the CLMS, ensuring that the appropriate analysis codes are chosen. Only analysis codes describing EPA-approved drinking water methods may be used. The tables on the following pages identify the particular methodologies utilized in processing drinking water samples.

For compliance monitoring in North Carolina, following the "Rules Governing Public Water Supplies" (amended February 1, 1987), all certified commercial laboratories are required to report results of analyses to both the Public Water Supply Branch and the supplier of water (client). The rules specify the particular reporting forms to be used and the time period in which reports are to be submitted.

In evaluating drinking water sample data, the QC criteria applied are as specified in the referenced method. Where unspecified, CompuChem® employs those criteria outlined in the Federal Register (October 26, 1984 600-series methods) for "Water and Wastewater", presented in Section 9.5 of the QA Plan. Once a database of sufficient size is generated, control limits for precision and accuracy will be generated based on historical data for aqueous sample analyses.

In order to continue providing analytical services for compliance monitoring, CompuChem® must maintain certification through the various drinking/potable water certifying agencies. The North Carolina Department of Human Resources (NCDHR), Division of Health Services, regulates certifications, performance evaluations and annual on-site laboratory inspections for these services in North Carolina. CompuChem® also maintains drinking water certifications in a number of other states, many of which accept reciprocal certification through the NCDHR.

METHODS USED BY COMPUCHEM®
FOR POTABLE WATER ANALYSIS

| Volatile Organic Contaminants/THMS | Method Used * |
|---|---|
| Bromobenzene | 524.1 |
| Bromochloromethane | 524.1 |
| Bromdichloromethane | 524.1 |
| Bromoform | 524.1 |
| Bromomethane | 524.1 |
| sec-Butylbenzene | 524.1 |
| tert-Butylbenzene | 524.1 |
| Carbon tetrachloride | 524.1 |
| Chlorobenzene | 524.1 |
| Chlorodibromethane | 524.1 |
| Chloroethane | 524.1 |
| Chloroform | 524.1 |
| Chloromethane | 524.1 |
| o-Chlorotoluene | 524.1 |
| p-Chlorotoluene | 524.1 |
| 1,2-Dibromo3-Chloropropane | 504,524.1 |
| Dibromethane | 524.1 |
| o-Dichlorobenzene | 524.1 |
| m-Dichlorobenzene | 524.1 |
| p-Dichlorobenzene | 524.1 |
| Dichlorodifluoromethane | 524.1 |
| 1,1-Dichloroethane | 524.1 |
| 1,2-Dichloroethane | 524.1 |
| 1,1-Dichloroethylene | 524.1 |
| cis-1,2-Dichloroethylene | 524.1 |
| trans-1,2-Dichloroethylene | 524.1 |
| Dichloromethane | 524.1 |
| 1,2-Dichloropropane | 524.1 |
| 1,3-Dichloropropane | 524.1 |
| 2,2-Dichloropropane | 524.1 |
| 1,1-Dichloropropane | 524.1 |
| 1,3-Dichloropropane | 524.1 |
| Ethylbenzene | 524.1 |
| Ethylenedibromide | 504,524.1 |
| Flurotrichloromethane | 524.1 |
| Hexachlorobutadiene | 524.1 |
| Isopropylbenzene | 524.1 |

* "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", September, 1986, EMSL-CI, U.S.EPA Cincinnati, Ohio 45268.

METHODS USED BY COMPUCHEM®
FOR POTABLE WATER ANALYSIS
(continued)

| Volatile Organic Contaminants/THMS | Method Used * |
|---|---|
| n-Propylbenzene | 524.1 |
| Styrene | 524.1 |
| 1,1,1,2-Tetrachloroethane | 524.1 |
| 1,1,2,2-Tetrachloroethane | 524.1 |
| Tetrachloroethylene | 524.1 |
| 1,1,1,-Trichloroethane | 524.1 |
| Trichloroethylene | 524.1 |
| Toluene | 524.1 |
| 1,1,2-Trichloroethane | 524.1 |
| 1,2,3-Trichloropropane | 524.1 |
| Vinyl chloride | 524.1 |
| o-Xylene | 524.1 |
| m-Xylene | 524.1 |
| p-Xylene | 524.1 |
| Chlorodane | 608 |
| Polychlorinated Biphenyls | 608 |

| Inorganic Contaminants | Method Used ** |
|---|---|
| Iron | 236.2 |
| Manganese | 243.2 |
| Arsenic | 206.2 |
| Barium | 208.2 |
| Cadmium | 213.2 |
| Chromium | 218.2 |
| Fluoride | 340.2 |
| Lead | 239.2 |
| Mercury | 245.1 |
| pH | 150.1 |
| Selenium | 270.2 |
| Silver | 272.2 |
| Sodium | 273.2 |

* "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", September, 1986, EMSL-CI, U.S.EPA Cincinnati, Ohio 45268.

** "Methods for Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. For approved analytical procedures for metals, the technique applicable to total metals must be used.

# APPROVED METHODOLOGY FOR ORGANIC CONTAMINANTS

| Additional Organic Contaminants | Method Used |
|---|---|
| Chlorinated Hydrocarbons | 1 |
| Endrin | 1 |
| Lindane | 1 |
| Methoxychlor | 1 |
| Toxaphene | 1 |
| Chlorophenoxy, Acids | 2 |
| 2,4,-D | 2 |
| 2,4,5-T | 2 |

1 : "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. (pp. 1-19)

2 : Ibid. (pp. 20-35)

# 10.0 Corrective Action

## 10.1 Introduction

Generally, there are two types of corrective actions that may be required when data quality falls below specified limits. The first type, and the simplest to implement and document, is corrective action required because routine data quality assessments are out-of-control. Surrogate and spike standard recoveries, relative percent differences between duplicates, internal standard response variations, and unacceptable blank contamination are some of these assessments in the first category. These are all performed on a sample-by-sample and/or batch basis, and corrective action is limited to evaluating the data with respect to SOP criteria, and accepting or rejecting the sample/batch. The decision that is made is clearly indicated on analytical worksheets, and unless a trend is observed during the course of data validation, additional corrective action or documentation is not necessary.

The second type of corrective action is that required when other, more global QC/QA assessments, are made. The assessments might typically indicate systematic deficiences or those affecting data useability for more than one batch (i.e., glassware contamination checks, standards preparation errors, etc.). In most cases, assessments of this nature are made by reviewing peripheral QC/QA documentation, observing procedures for comparison with SOPs or GLPs, or receiving feedback from data reviewers, management or those external to the organization (clients, auditors).

The following sections describe the QA reporting and feedback channels designed to ensure that early and effective corrective action is taken in such instances.

In many cases, depending on the nature of the deficiency and the urgency for remedial action, a Corrective Action Report (following this section) will be completed. The report serves to document the deficiency, the required corrective action, and accountability for the action.

For observations made over longer periods of time, the QA Department issues formal summary reports to management on a monthly or quarterly basis. Following is a brief discussion of the types of reports issued to management to assess the overall effectiveness of the QA Program and to reinforce the application of Good Laboratory Practices (GLPs).

# CORRECTIVE ACTION REPORT

DATE:_____

PROBLEM / DEFICIENCY:

_____

_____

_____

_____

_____

IDENTIFIED BY: _____

REFERRED TO: _____ (QA)

CORRECTIVE ACTION TO BE TAKEN: TARGET DATE:_____

_____

_____

_____

_____

_____

_____

FOLLOW-UP AUDIT FINDINGS:

_____

_____

_____

_____

RESOLVED? _____ DATE: _____

SOP REQUIRED TO BE WRITTEN/MODIFIED? YES [ ] NO [ ] TARGET DATE:_____

This form to be filed with the Quality Assurance Clerk for permanent record.

## 10.2 Routine QC Check Reports

The following routine quality control checks (also discussed in section 9.2 of the QA Plan) are performed to verify that samples are not contaminated during transportation, preparation, analysis or storage, and that standards prepared internally are traceable to certified sources.

- -- Vendor-Supplied Glassware Checks
- -- Glassware Decontamination Checks
- -- Water Purification Systems Checks
- -- Glassware Storage Cabinet Checks
- -- Refrigerated Storage Systems Checks
- -- Reagent Purity Checks
- -- Standards Prepartion and Traceability Checks

The criteria for these QC checks and corrective action steps are detailed in the QA SOP Manual. Results are tabulated and/or plotted on control charts, and records reviewed by the QA staff. A series of quarterly reports to management summarize this information and the status of these programs.

## 10.3 Monthly QA Activity Reports

These reports are produced by all members of the QA staff, and summarize key QA activities during the previous month. The reports are distributed to the Director of QA, and are provided as an attachment and referenced in the Director's report to the CEO, the Executive Staff and senior laboratory management.

Included in these reports is a summary of significant quality problems observed during the period, and the corrective actions taken to remove deficiencies. The report stresses proactive measures that are being taken to improve quality or ensure compliance with QA program requirements.

Laboratory management uses the report to quantitatively measure monthly performance in terms of the number of samples processed, the frequency of repeated sample analyses due to unacceptable QC performance, and the cause of the unacceptable performance. These data are all presented in tables, Pareto control charts or attribute control charts, based on the characterization of each analysis in the Computerized Laboratory Management System (CLMS) using a system of analytical "condition codes."

The Condition Code System is used to monitor sources of data failures. Condition code definitions are provided in an SOP to data generators and reviewers who are responsible for assigning the appropriate code to each analysis (see Appendix D). Each two-letter code is used to characterize the cause of a sample failure or the final status of the data package prior to release to the client.

Various computer programs may be used to sort condition code data according to sample matrix and method. This system is used to pinpoint sources of error, provide feedback to management, reinforce good laboratory practices, and document laboratory performance over time. The QA staff also note in the Monthly QA Activities Report any corrective actions taken or necessary procedural changes, based on the application of condition codes.

Other items included in this report are:

- -- Summary of any changes in certification/accreditation status
- -- Involvements in resolution of quality issues with clients or agencies
- -- QA organizational changes
- -- Notice of the distribution of revised documents controlled by the QA Department (i.e., SOPs, QA Plan)
- -- Training and safety issues, if not already covered in audit reports during the period
- -- Performance of subcontractor laboratories (also communicated in separate, detailed subcontractor audit report to management)
- -- Positive feedback for acceptable performance on interlaboratory or intralaboratory tests or successful completion of audits.

## 10.4 Laboratory Performance Reports

This quarterly report presents a statistical and graphical summary of the laboratory's performance on batch-associated quality control samples analyzed over the period. Included are tables, Shewhart control charts and I-charts (for individual data points) for all surrogate and spike standard recoveries. Additionally, a monthly report to the Director of QA presents control charts and tables for all Laboratory Control Sample (Blank Spike) and Blank recoveries. The charts and tables are used primarily to document historical performance, update recovery control limits, and monitor long-range trends that might not be apparent to data reviewers evaluating data on a sample/batch basis.

## 10.5 Laboratory Audit Reports

Quarterly audit reports are written by a member of the QA staff and distributed to management, and summarize the results of internal laboratory Performance Audits, Systems Audits and Security/Access Audits. When external auditors are involved in Performance or System Audits, a report is written within the next week by the QA staff member coordinating the audit. The report, summarizing audit results as discussed in the debriefing as well as other observations, is distributed to the CEO and senior lab management. The report includes corrective actions required as a result of the audit, and a schedule for implementation. A follow-up audit, usually within three weeks of the distribution of this report, is conducted to verify that corrective actions have been implemented.

### Performance Audits

Performance Audits are checks made by a QA staff member or other independent auditors to evaluate the quality of the data produced by the analytical system. These audits are performed independent of an in addition to routine quality control checks, and reflect as closely as possible lab performance under normal operating conditions.

These audits involve the review of approximately 10% of all analytical data reports generated by the lab for calculation and data validation procedures, and overall data quality. Errors observed during the audit are characterized as "critical" or "correctable" and tabulated. If necessary, based on audit findings, an amended data report may be sent to the customer. Following this section is a copy of the QA Audit Summary used by auditors to tabulate the data

for summary into the Quarterly Performance Audit report. A thorough discussion of these audits is included in the QA SOPs. The reports are used by laboratory managers to provide feedback to staff members and establish goals for improved performance.

A number in interlaboratory and intralaboratory tests are conducted routinely at CompuChem®, and the results are included in individual Performance Audit reports specific to each test. When new methods are available to the laboratory or new personnel are being trained, Laboratory Proficiency Tests are performed. These tests consist of quadruplicate blank spikes, containing a full complement of tests parameters to be analyzed by the method. The replicate results are analyzed by a QA staff member, who generates a summary report to the Director of QA. This report includes the standard deviation and mean recovery for each of the replicate parameters, and the data are used to statistically validate method and/or personnel proficiency. For a thorough discussion of the method validation procedures used, refer to Appendix A of the QA Plan.

On a quarterly basis, blind intralaboratory check samples are introduced into the system by the QA Department. Parameters and methods are chosen for these studies based upon independent (interlaboratory) tests from certifying agencies (including the U.S. EPA and various state agencies), Laboratory Proficiency Test results, Method Validation studies, or results from routine batch-related QC samples. The existence of these check samples in the system is known only to those personnel involved in preparing the samples and scheduling the analytical requirements into the CLMS. A thorough report, detailing the entire data generation and support functions, is completed by the QA staff and reviewed by

the Director of QA before distribution to the CEO and senior laboratory management.

CompuChem® also participates in a number of external, interlaboratory performance studies. These are required as part of various agencies' certification/accreditation programs. As a member of the USEPA's Contract Laboratory Program (CLP), the laboratory is required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP program also requires an annual on-site inspection by principals from the USEPA (and their contracted agents). These audits generally follow the same format described below, Systems Audits.

CompuChem® also participates in a number of state certification programs, including those for North Carolina, New Jersey, New York and Florida. All of these programs require the laboratory to submit to annual on-site inspections in order to maintain certification to perform testing on samples originating in the state. All states also require successful performance on interlaboratory check samples, submitted at least annually, though some reciprocity with the two NC programs (one for drinking water and one for wastewater certification) and USEPA-CLP is allowed under certain circumstances.

Several states utilize the laboratory's performance on the annual Water Supply (WS) and Water Pollution (WP) proficiency testing series, orginating out of the EPA Environmental Monitoring and Support Laboratory's performance on all interlaboratory and intralaboratory check samples, tabulated by parameter and method, so negative performance trends can be readily pinpointed.

System Audits

A System Audit is an on-site inspection and review of the QA Program for the total laboratory. While Performance Audits are a quantitative appraisal, System Audits are for the most part qualitative in nature. The System Audit may be either scheduled or unannounced before it is conducted, but occurs routinely on at least a quarterly basis. The auditor reviews the laboratories' SOPs to verify compliance with procedures and activities actually in place. Personnel and facilities are also evaluated during the System Audit. The auditor is required to investigate anything which seems in conflict with the QA Plan, the laboratory or QA SOPs, or Good Laboratory Practices.

If deficiencies are observed during a Performance Audit, and if deemed necessary, the QA Department initiates a System Audit. The audit emphasizes the actions necessary to correct deficiencies noted in the Performance Audit. A Corrective Action Report is completed, detailing all remedial actions taken, and reviewed by the Director of QA. The report must indicate the proposed implementation date and the individual(s) responsible for the action.

Many of the objectives of a routine System Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring the following:

1. The quality control, including necessary corrective actions , are being applied
2. Adequate facilities and equipment are available to perform the client's required scope-of-work
3. The personnel are qualified to perform the assigned tasks
4. Complete documentation is available, including sample chain-of-custody

5. Proper analytical methodology is being applied

6. Acceptable data handling techniques are being used

7. Corrective actions identified in any previous on-site visits have been implemented, and

8. The Laboratory Management continues to demonstrate a commitment to quality.

These objectives may be documented by completing an EPA-approved Laboratory Evaluation Checklist. In response to System Audits, any corrective actions taken are noted with reference to the auditor's deficiency report and the lab's Standard Operating Procedures.

| | SAMPLE NUMBER | FRACTION | REJECT LOCATION/CAUSE | | | | | | COMMENTS | SUMMARY corr* Failure | crit** Failure | status |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | | | | | | | | | | |
| 2 | | | | | | | | | | | | |
| 3 | | | | | | | | | | | | |
| 4 | | | | | | | | | | | | |
| 5 | | | | | | | | | | | | |
| 6 | | | | | | | | | | | | |
| 7 | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | |
| 9 | | | | | | | | | | | | |
| 10 | | | | | | | | | | | | |
| 11 | | | | | | | | | | | | |
| 12 | | | | | | | | | | | | |
| 13 | | | | | | | | | | | | |
| 14 | | | | | | | | | | | | |
| 15 | | | | | | | | | | | | |
| 16 | | | | | | | | | | | | |
| 17 | | | | | | | | | | | | |
| 18 | | | | | | | | | | | | |
| 19 | | | | | | | | | | | | |
| 20 | | | | | | | | | | | | |
| 21 | | | | | | | | | | | | |
| 22 | | | | | | | | | | | | |
| 23 | | | | | | | | | | | | |
| 24 | | | | | | | | | | | | |
| 25 | | | | | | | | | | | | |
| 26 | | | | | | | | | | | | |
| 27 | | | | | | | | | | | | |
| 28 | | | | | | | | | | | | |

*correctable failures

**critical failures

DATE:

# QA AUDIT CODES

## Missing/Incorrect:

| | |
|---|---|
| CAM/I | Calculations missing/incorrect |
| CCM/I | Condition code missing/incorrect |
| DFM/I | Data footnote missing/incorrect |
| CFM/I | Correction factor missing/incorrect |
| DWI | Dry weight/percent moisture incorrect |
| FNI | Filename incorrect |
| FFM/I | Form 4 missing/incorrect |
| LSM/I | Library search missing/incorrect |
| QNM/I | QA Notice missing/incorrect |
| RRM/I | Reportable run missing/incorrect |
| SPM/I | Spectrum missing/incorrect |
| SRM/I | Sample receiving information missing/incorrect |
| SSM/I | Surrogate Summary Form missing/incorrect |
| STM/I | Standard package missing/incorrect |
| TFM/I | Tuning Form missing/incorrect |
| UNM/I | Units missing/incorrect |
| WSM/I | Worksheet missing/incorrect/incomplete |
| OAM/I | OADS missing/incorrect/incomplete |

## Qualitative/Quantitative Errors:

| | |
|---|---|
| HNR | Hit not reported, but should have been |
| HRE | Hit reported in error, should not have been reported |
| HAI | Hit amount reported incorrectly |
| CFN | Correction factor not applied to hit |
| SFI | Significant figures (or rounding off) incorrect |
| TRE | Transcription error |

## Miscellaneous Errors:

| | |
|---|---|
| ISF | Internal standard area monitor indicates failure |
| ODI | OWA date or time incorrect |
| RNL | RIC not labeled |
| SOL | Surrogate(s) actually outside limits |
| WOU | Whiteout used on documents (deliverables) |
| NSO | Not signed off |
| CNI | Change not initialed |

## Condition Code Applications:

| | |
|---|---|
| CS | Carryover suspected |
| CT | Contamination evident |
| RU | Repeated unnecessarily |
| SF | Spikes failed |
| UN | Unacceptable, not needed |

## 6.4 Instrument Maintenance

Analytical instruments are maintained by experts employed by CompuChem® on a full-time basis. Preventative maintenance as well as major instrument repairs can be accomplished on-site. An extensive in-house stock of spare parts allows for rapid repair. CompuChem® maintains service agreements with instrument manufacturers to further assure the operational viability of all in-house equipment.

The operational condition of instruments is one of the keys to successful completion of analytical tasks. This requirement is further magnified by the necessity to complete large programmatic requirements in a limited period of time. CompuChem's commitment to instrument maintenance assures clients that equipment will be available to generate the required data.

In discussing instrument maintenance services at CompuChem®, a distinction between GC/MS instruments and other hardware is required. In the case of the GC/MS instrumentation, CompuChem® staff have full maintenance and repair responsibility. These staff have been trained by the instrument manufacturer and are fully qualified to perform the required work. For other instruments, we have service contracts for periodic maintenance visits by the vendor, although maintenance personnel do assess whether repairs can be made in-house before outside vendors are called.

All GC/MS instrument repair logs and instrument service records are maintained in individual instrument files in the instrument repair shop.

EXAMPLE 1

COMPUCHEM SERVICE REPORT

R-1234 A

INSTRUMENT NO 1 2 DATE 0 7 2 3 8 6 TIME 1 1 5 1 OPERATOR: Susan Bass

GC oven not heating

| START DATE | 0 7 | 2 3 | 8 6 | END DATE | 0 7 | 2 3 | 8 6 | RESPONSE TIME | 5 | S/E Lee Gregory | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| START TIME | | 1 0 | 1 5 | END TIME | | 1 0 | 5 7 | TOTAL DOWNTIME | 1 2 | JOB COMPLETE? YES | X | NO |

| ASSEMBLY NO. | REV LEVEL | REPAIR CODE | FAILURE CODE | COMPONENT DESIGNATOR | PART NO. | DESCRIPTION | QTY | COST |
|---|---|---|---|---|---|---|---|---|
| 7 0 1 7 8 9 1 4 3 2 0 1 | 0 A | 0 4 | 0 6 | F 4 | 3 A 0 4 A | Fuse | 1 | 1 35 |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

COMMENTS: Oven not heating, 24 volt relay not engaging found fuse F4 blown in power supply. Replaced fuse F4, checked +24 volts, checked operation of GC, gave back to operator L.G.

OPERATOR ACCEPTANCE: DATE 0 7 2 3 8 6 TIME 1 1 0 0 REV07298216

EXAMPLE 1 (CONTINUED)

## INSTRUCTIONS

1. INSTRUMENT NO. - LIST AS 05 FOR OWA05, 12 for OWA12, ETC. THE 4021 GC/MS/DS IS INSTRUMENT 00. ALL STANDALONE DATA SYSTEMS ARE INSTRUMENT 99.

2. DATE & TIME - ENTER DATE AS MM/DD/YY; AUGUST 28, 1985 IS 08/28/85. ENTER TIME BY 24-HOUR CLOCK. 9:25AM IS 0925 AND 9:25PM IS 2125. THE TIME AND DATE SHOULD BE WHEN A PROBLEM IS DISCOVERED AND REPORTED VIA THIS FORM.

3. OPERATOR - WHO YOU ARE.

4. PROBLEM CODE & DESCRIPTION - USE THE 3 DIGIT PROBLEM CODE THAT MOST APPROPRIATELY DESCRIBES YOUR PROBLEM. PLEASE DETAIL THE PROBLEM AS FULLY AS YOU CAN.

5. USE BLACK INK ONLY & WRITE OR PRINT LEGIBLY.

| PROBLEM CODES (CAUSE & EFFECT) | |
|---|---|
| P.M. | 000 |
| CANNOT MEET TUNE | 001 |
| IDOS ERRORS - LIST AND FULLY DESCRIBE WHAT THE DATA SYSTEM WAS DOING | 002 |
| POOR SENSITIVITY | 003 |
| DRIFTING RET. TIMES | 004 |
| NO SPECTRA OR NO MS RESPONSE | 005 |
| SOFTWARE ANOMALIES | 006 |
| GAS CHROM. | 007 |
| DISC DRIVE | 008 |
| PRINTER | 009 |
| PURGE & TRAP | 010 |
| VACUUM FAULT | 011 |
| AIR LEAKS | 012 |
| TERMINAL | 013 |
| DATA SYSTEM | 014 |
| CANNOT BOOT | 015 |
| UNKNOWN | 016 |

| REPAIR ACTION CODES | |
|---|---|
| PIRATE PARTS | 100 |
| ADJUSTMENT - ELEC | 102 |
| ADJUSTMENT - MECH. | 104 |
| REPLACED ASSY. | 106 |
| RETURNED TO VENDOR REPAIR | 108 |
| RETURNED TO VENDOR WARRANTY | 110 |
| REQUESTED IN-HOUSE VENDOR SERVICE | 112 |
| WAITING FOR PARTS (NOTE P.O. #) | 114 |
| CLEANED SEPARATOR | 116 |
| CLEANED MASS FILTER | 118 |
| CLEANED SOURCE | 120 |
| REPLACE PART | 122 |
| REPAIR IN-HOUSE | 124 |
| UNABLE TO REPRODUCE | 126 |

| FAILURE ANALYSIS CODES | |
|---|---|
| UNKNOWN | 200 |
| MISCELLANEOUS | 202 |
| OPERATOR ERROR | 204 |
| SOFTWARE | 206 |
| HEADCRASH | 208 |
| MECH. DEFECT | 210 |
| OUT OF ADJUSTMENT | 29* |
| INTERMITTANT | 29* |
| EXCESSIVE NOISE | 29* |
| EXCESSIVE WEAR | 212 |
| SHORTED COMPONENT | 214 |
| OPEN COMPONENT | 216 |
| FAULTY CRIMP | 218 |
| POOR CONTACT | 220 |
| POOR SOLDER JOINT | 222 |
| DIRTY/DUSTY | 224 |
| LEAKING | 226 |
| REPLACE * WITH | |
| 1 = ELECTRICAL | |
| 2 = MECHANICAL | |
| 3 = VACUUM | |
| 4 = SOFTWARE | |

REV07298216OO

PREVENTIVE MAINTENANCE - 3 MONTH INTERVAL

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GU

| ITEMS TO BE INSPECTED | PROBABLE SYMPTOM | SERVICE INTERVAL | | | | | | SE IN |
|---|---|---|---|---|---|---|---|---|
| | | daily | bi-monthly | 3 months | 6 months | yearly | as required | NOTE: are pr Operat other |
| Sigma 3 GC | | | | | | | | |
| 1. Line fuses for the GC | Inactive GC, blown fuse | | | | | | X | repla |
| 2. Injector for packed columns | | | | | | | X | |
| 3. Splitless injection for capillary columns | | | | | | | X | |
| 4. Injector septum in the GC | obstruction, leaks | X | | | | | X | clear Inspe |
| 5. Carrier gas connections/couplings | leakage | | | | | | X | repl requ |
| 6. Carrier gas filter in the GC | replace when new gas cylinder is installed | | | | | | X | |
| 7. Filter, flow controller | dirty filter | | | X | | | | repl clea |
| 8. Capillary column | | | | | | | X | Insp repl |
| 9. Packed column (glass type) | excessive usage, leaks at injection and interface port of the zone-heating block | | | | | | X | neec |
| 10. Packed column (metal type) | | | | | | | X | |
| 11. Detector port to GC/MS interface | | | | | | | X | |
| 12. GC cool down fan | | X | | X | | | | Ins or |

*These maintenance procedures meet or exceed Finnigan's recommended preventive mai checks and services.

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

| ITEMS TO BE INSPECTED | PROBABLE SYMPTOM | SERVICE INTERVAL: daily | bi-monthly | 3 months | 6 months | yearly | as required | SERVICE INTERNAL | PROCEDURE |
|---|---|---|---|---|---|---|---|---|---|
| **Mass Spectrometer** | | | | | | | | | |
| 1. Glass jet separator | obstruction or glass | | | | | | X | clean or replace | |
| 2. Glass jet separator ferrules | breakage | | | | | | X | replace | |
| 3. Mass analyzer head assembly (in the vacuum manifold) | gross leaks, presistent pressure due to degasing of trapped gases in the vacuum system | | | | | | X | inspect | |
| •magnet well flange assy | leakage, faulty CAL gas | | | | | | X | inspect | |
| •CAL gas valve assy | pressure (see Pirani | | | | | | X | inspect | |
| •vent valve assy | gauge) | | | | | | X | inspect | |
| •water flow sensor switch | faulty switch | | | | | | X | | |
| 4. Quadrupole mass analyzer | | | | X | | | | inspect and/or replace | |
| 5. Electron multiplier | | | | | | | X | | |
| 6. Alcatel vacuum pumps (2) | | | | X | | | | purge weekly and replace oil | |
| 7. Pfeiffer turbo pump | dirty oil | | | | | | | | |
| 8. Balzer turbo pump | | | | X | | | | | Balzer Manual pg...23 |
| 9. Vacuum system filter/drier | excessive use, dirty filter | | | | | | X | clean & inspect | |
| 10. Ion Source | | | | X | | | | | |
| •ion source filament assy | lack of sensitivity, faulty peak shape, no autotune | | | X | | | | replace | |
| •collector | | Service concurrently with every filament assembly replacement | | | | | | clean, inspect or replace as required | |
| •lens | | | | | | | | | |
| •aperture | | | | | | | | | |
| •ion volume | | | | | | | | | |
| **GC/MS Interface Oven** | | | | | | | | | |
| 1. Capillary interface tubing | plugged | | | | | | X | clean, inspect and/or replace | |
| 2. Separator divert fitting | | | | X | | | X | | |
| 3. Vacuum divert valve | | | | X | | | X | | |
| **Power Module** | | | | | | | | | |
| 1. MS power supply | | | | X | | | X | measure & verify PCB | |
| 2. Turbo power supply | | | | X | | | X | | |
| **Card Cage Module** | | | | | | | | | |
| 1. Air filter at bottom of cage | dirty filter, obstruction of air flow | | | X | | | | clean and/or replace | |
| 2. Fan | burned out fan | | | X | | | X | | |
| 3. Signal cable on Digital I/O PCB | | | | | | | X | inspect for secure fit | |

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

| ITEMS TO BE INSPECTED | PROBABLE SYMPTOM | SERVICE INTERVAL | | | | | | SERVICE INTERNAL | PROCEDURE |
|---|---|---|---|---|---|---|---|---|---|
| | | daily | bi-monthly | 3 months | 6 months | yearly | as required | | |
| Nova Computer | | | | | | | | | |
| 1. Fan | faulty fan rotation | | | x | | | | inspect and/or replace | |
| Perkin-Elmer Disk Drive | | | | | | | | | |
| 1. Output signal | | | | x | | | | check and verify | |
| 2. Adjustable DC voltages (+5V, +13V, -13V) | | | | x | | | | | |
| 3. Brushes | | | | | | | | clean and/or replace | |
| 4. Positioner carriage guide rails | | | | | x | | | clean and inspect | |
| 5. Spindle chuck and cone | | | | | x | | | clean and inspect | P/ECEM Manual |
| 6. Read-write heads | | | | x | | | | inspect and repair | P/ECEM Manual |
| 7. Fixed disk | | | | x | | | | | |
| 8. Air filter | | | | | | x | | | P/ECEM Manual |
| *prefilter | | | | x | | | | replace | |
| *main filter | | | | x | | | | replace | |
| 9. Blower ground brush | | | | | | x | | replace | |
| 10. Spindle ground brush | | | | | | x | | replace | |
| 11. Blower drive belt | | | | | | | | replace | P/ECEM Manual |