Golder Associates Inc.

305 Fellowship Road, Suite 200
Mt. Laurel, NJ USA 08054
Tel: (609) 273-1110
Fax (609) 273-0778

Golder Associates

WORK PLAN

REMOVAL ACTION

NEASE CHEMICAL SITE

SALEM, OHIO

VOLUME 2 OF 2

REVISION #2

Submitted to:

Ruetgers-Nease Corporation
201 Struble Road
State College, Pennsylvania 16801

DISTRIBUTION:

3 Copies	-	USEPA Region 5
2 Copies	-	Ohio EPA
2 Copies	-	B & V Waste Science and Technology Corp.
1 Copy	-	Michael L. Hardy, Esq., Thompson, Hine & Flory
2 Copies	-	Ruetgers-Nease Corporation
2 Copies	-	Golder Associates Inc.

April 1994

EPA Region 5 Records Ctr.
231132

Project No.: 933-6158

WORK PLAN

REMOVAL ACTION

RUETGERS-NEASE SALEM, OHIO SITE

CONTENTS OF VOLUME 2

SECTION I	-	FIELD SAMPLING PLAN
SECTION II	-	QUALITY ASSURANCE PROJECT PLAN
SECTION III	-	HEALTH AND SAFETY PLAN

REVISION #2

April 1994

Project No.: 933-6158

VOLUME 2

SECTION I - FIELD SAMPLING PLAN

REVISION #2

April 1994

Project No.: 933-6158

FIELD SAMPLING PLAN
TABLE OF CONTENTS

Table of Contents i

SECTION PAGE

1.0 INTRODUCTION 1
1.1 Facility Information 2
1.1.1 Site Location 2
1.1.2 Site Operations History 3
1.1.3 Site Regulation History 4
1.1.4 Previous Investigations 5
1.1.5 Previous Remedial Actions 6

2.0 FIELD ACTIVITIES SUMMARY 8
2.1 Investigation Activities 8
2.1.1 Treatment Plant Evaluation Activities 8
2.1.2 Field Activities 9
2.1.2.1 Preliminary Activities 9
2.1.2.2 Field Investigation Activities 9
2.2 Sampling and Analysis Program 10
2.3 Project Organization 12
2.3.1 Project Coordinator 12
2.3.2 Project Director 12
2.3.3 Project Manager 13
2.3.4 Field Operations Manager 13
2.3.5 Health and Safety Officer 14
2.3.6 Project QA/QC Manager 14
2.3.7 Site Safety Manager 14
2.3.8 U.S. EPA Remedial Project Manager and OEPA Project Coordinator 15
2.3.9 U.S. EPA/OEPA QA/QC Manager 16

3.0 FIELD INVESTIGATION ACTIVITIES 17
3.1 Treatment Plant Performance Evaluation 17
3.1.1 Air Monitoring 17
3.1.1.1 Operation of the HNU Model P1-101 Photoionizer Instrument 18
3.1.2 Treatment Plant Sampling 18
3.1.2.1 Leachate Collection 19
3.1.2.2 Gas Sampling 21

TABLE OF CONTENTS (Cont'd)

SECTION PAGE

3.2 Field Sampling 22
3.2.1 Air Monitoring 22
3.2.2 Surface Water and Sediment Sampling Protocols 22
3.2.3 Groundwater Purging and Sampling Protocols 24
3.3 Quality Assurance/Quality Control Samples 25
3.3.1 Treatment Plant QA/QC 25
3.3.2 Field Quality Control Checks 26
3.3.3 Laboratory Quality Control Checks 28
3.3.3.1 Trace Organic Analysis: Volatiles, Semivolatiles, Pesticides, Mirex, Photomirex, and Kepone 28
3.3.3.2 Trace Metals Analysis 29
3.3.3.3 Control Limits 30

4.0 EQUIPMENT AND CALIBRATION 31

5.0 SAMPLE HANDLING 32
5.1 Chain-of-Custody 32
5.2 Sample Identification 33
5.3 Sample Packaging 33
5.4 Sample Shipping 34

6.0 FIELD DOCUMENTATION 36
6.1 Log In/Log Out Record 36
6.2 Field Notebooks 36
6.3 Photo-Documentation 37
6.4 Correspondence/Communications 38

7.0 EQUIPMENT DECONTAMINATION 39
7.1 General Consideration 39
7.2 Heavy Equipment 40
7.2.1 Drilling Equipment 40
7.3 Sampling Equipment 41
7.3.1 Soil and Sediment Sampling Equipment 41
7.3.2 Groundwater and Surface Water Sampling Equipment . 42
7.3.3 pH, Temperature, Dissolved Oxygen, and Depth to Water Probes 42
7.4 Electronic Equipment 43

TABLE OF CONTENTS (Cont'd)

In Order
Following
Page 43

LIST OF TABLES

Table 1a Performance Evaluation Sampling and Analysis Plan Summary: Target Analytes, Analytical Methods, and Quality Assurance Samples
Table 1b Field Sampling and Analysis Plan Summary: Target Analytes, Analytical Methods, and Quality Assurance Samples
Table 2a Sample Locations and Laboratory Analyses
Table 2b Sample Locations and Field Analyses
Table 3 Analytical Methods, Sample Containers, Preservation and Analytical Hold Times for Aqueous and Gaseous Samples
Table 4a Sample and Data Quality Objective Summary: Treatment Plant Evaluation
Table 4b Sample and Data Quality Objective Summary: Field Sampling

LIST OF FIGURES

Figure 1 Site Location Plan
Figure 2 Interim Remedial Measures
Figure 3 Removal Actions Work Plan Schedule
Figure 4 Project Organization Chart

LIST OF APPENDICES

Appendix A Sampling and Field Testing Procedures

1.0 INTRODUCTION

On November 17, 1993 Ruetgers-Nease Corporation (RNC) entered into a Administrative Order by Consent (Removal AOC) with the U.S EPA concerning removal actions to take place at the Nease Chemical Site, Salem, Ohio.

The Removal AOC "requires the Respondent to undertake and complete emergency removal activities to abate conditions which the U.S. EPA has determined may present an imminent and substantial endangerment to the public health or welfare or the environment because of an actual or threatened release of hazardous substances at the Site."

Based upon the findings and determinations of the U.S. EPA contained therein, and pursuant to Section 106(a) of CERCLA, 42 U.S.C. Section 9606(a), the Respondent is required to undertake certain Removal Actions at the Nease Chemical Site.

This Field Sampling Plan was developed by Golder Associates at the request of RNC to meet the requirements of the Removal Actions mandated in the Removal AOC for the Nease Chemical Site (Site) in Salem, Ohio. An objective of the Removal Actions is to gather data of adequate technical content, quality, and quantity to complete a performance evaluation of the existing groundwater treatment plant which will be used to determine the ability of the treatment plant to meet the allowable discharge criteria.

The purpose of this FSP is to describe the sampling program rationale and procedure that will result in data of suitable quality and quantity to achieve the Removal Action objectives.

The FSP is organized into the following sections:

- Section 1.0 - Introduction
- Section 2.0 - Field Activities Summary
- Section 3.0 - Field Investigation
- Section 4.0 - Equipment Calibration
- Section 5.0 - Sample Handling
- Section 6.0 - Field Documentation
- Section 7.0 - Equipment Documentation
- Appendix A - Sampling and Field Testing Procedures.

Analytical methods and Quality Control/Quality Assurance (QA/QC) Procedures are provided in the Quality Assurance Project Plan (QAPjP), which is found in Section II of this Volume of the Work Plan.

1.1 Facility Information

This section provides an overview of the operational and administrative history of the Site. It describes the site location, operation and regulatory history, past investigations, and past remedial actions at the Site.

1.1.1 Site Location

The Site is located approximately 2.5 miles northwest of the City of Salem, Ohio in northern Columbiana County, near the southern border of Mahoning County (Figure 1). The Site, covering approximately 44 acres, is situated on the north side of State Route 14 and west of Allen Road. It is bordered by a large field and a

residential area on the south, a large wooded area to the north, an industrial facility (Crane-Deming Company) to the northeast, agricultural/residential areas to the northwest, and a residential area to the east. There are a few homes immediately to the east and southeast of the Site. The Site is dissected by Conrail railroad tracks that run southeast-northwest.

The Site is located on a topographic high, the axis of which runs southeast-northwest. The majority of the site slopes to the northeast and drains to the Middle Fork of the Little Beaver Creek (MFLBC). The geology is surficial Wisconsin Age glacial deposits (till, loam, sands) of variable thicknesses and extent, overlying a thick sequence of Middle Pennsylvanian Age sandstones and shales, with limestone and coal interbeds.

1.1.2 Site Operations History

A comprehensive history of site operations can be found in the RI Report (Ruetgers-Nease, 1993). Briefly, from 1961 through 1977, Nease Chemical Company owned and operated a chemical manufacturing plant at the site. At various times during the period of 1961 through 1973, Nease produced a variety of chemical compounds, including household cleaning compounds, fire retardants,pesticides and chemical intermediates used in agricultural, pharmaceutical, and other chemical products. Some of these chemicals included mirex, diphenylsulfone, chloramine B, benzene, sulfonic acid, methoxychlor, and hexachloroethylene.

Products and chemical intermediates were produced in batch processes. Waste generated was neutralized and treated on-site. Five unlined ponds (designated 1, 2, 3, 4, and 7) were used for treatment and storage of either acidic plant waste or lime slurries from neutralization of acidic wastes. After final settling, the

neutralized liquid was discharge to the Salem Waste Water Treatment Plant (WWTP) from the late 1960s to 1975. In addition, prior to 1977, some drummed wastes were buried in an area, on-site, which is referred to as Exclusion Area A (Figure 1). The migration of contaminants via the groundwater and overland via surface water and sediments has been documented.

In 1973, Nease discontinued manufacturing operations at the site and subsequently, decided to close the facility permanently. In 1974 and 1975, under OEPA supervision, all buildings and manufacturing equipment were removed from the site, except for a warehouse and two small block buildings. The five ponds were decommissioned (under OEPA supervision) by Nease in December 1975 pursuant to the 1973 Consent Judgment.

As of December 30, 1977, Nease Chemical Company, Inc. (including the site) was acquired by and merged with Ruetgers Chemical, Inc. The company resulting from the merger is Ruetgers-Nease Chemical Company, Inc. In 1983, the site was placed on the National Priorities List (NPL). The company officially changed its name to Ruetgers-Nease Corporation (RNC) on October 1, 1993.

1.1.3 Site Regulation History

The Remedial Investigation/Feasibility Study (RI/FS) AOC of January 1988 required RNC to conduct a remedial investigation (RI) "to determine fully the fact, nature and extent of any release or threatened release of hazardous substances, pollutants or contaminants at and/or from the Facility and to perform a Feasibility Study (FS) to identify and evaluate alternatives for the appropriate extent of remedial action to achieve in offer to comply with applicable or relevant and appropriate requirements, standards, limitations, criteria or goals and/or to prevent or mitigate

the migration or release or threatened release of hazardous substances, pollutants, or contaminants from the facility, in accordance with Section 121 of CERCLA."

On April 5, 1991 RNC submitted a partial RI report to the agencies addressing the Middle Fork Little Beaver Creek.

On July 6, 1993, RNC submitted to the Agencies a Remedial Investigation Report. That report presents information obtained from existing data and from the data gathered during the RI field work conducted under the 1988 AOC.

1.1.4 Previous Investigations

A detailed account of past Response Actions at the Nease Chemical site can be found in the RI Report (Ruetgers-Nease, 1993). Briefly, RNC undertook programs for remediating constituents of concern at the site prior to the 1988 RI/FS AOC as follows:

Fall 1982: Soil borings taken, deep and shallow monitoring wells installed, geophysical investigation, collection and analysis of surface water, soil, and sediment, progress reports to OEPA.

Fall 1983: Additional monitoring wells, depth-discrete and composite surface soil samples, and sediment and surface water samples collected from the MFLBC and analyzed.

Spring 1984: Additional monitoring wells installed.

Summer 1984: Additional monitoring wells installed, deep groundwater test wells installed, aquifer testing for hydraulic properties and hydraulic interconnectedness of aquifers.

Spring 1985: Fish, sediment, surface water, and benthic organism sampling and analysis of MFLBC.

Winter 1985: Additional soil borings.

Spring 1986: Additional monitoring wells installed, additional soil borings.

April 1982, December 1982, April, September, and December 1983, February and July 1984, January, May, and December 1985, April 1986, March 1987: Groundwater sampling and analysis.

The findings of these studies have been incorporated into the Remedial Investigation Report submitted by RNC on July 6, 1993.

1.1.5 Previous Remedial Actions

RNC initiated remedial activities with a remediation plan submitted to the OEPA on March 12, 1983. The plan called for Phase 1 removal of buried drums in Exclusion Area A, and removal of affected soil from Exclusion Areas A and B; and Phase 2 assessment of constituents of concern in surface water, groundwater, soil, and sediment.

In the fall of 1983, RNC performed Phase 1 work as described above, and implemented various steps to control soil erosion at the site, including seeding to

establish ground cover, installation of geotextile fabric barriers across drainages and ditches, installation of rock dams, and installation of barriers around the Exclusion Areas. Additionally, a leachate collection system was installed on the south side of the railroad tracks to mitigate the migration of groundwater seepage. The leachate has been intermittently collected and disposed of at a permitted off-site waste treatment facility since 1983.

Beginning in late 1991 and concluding in September of 1993, RNC voluntarily installed equipment at the Site, that was approved by U.S. EPA and OEPA, to reduce the potential for off-site transport and mitigation of certain constituents prior to implementing any permanent remedies deemed necessary in the future. The equipment was designed for surface water management and sediment control, and seep control and treatment.

2.0 FIELD ACTIVITIES SUMMARY

The Removal Actions of Paragraph 4a., 4b., 4c., 4d., 4e., and 4f. of the Removal AOC will include field sampling and subsequent chemical analysis. This section of the FSP summarizes the work that will be conducted during 1) the commencement of operation and testing of the on-site leachate treatment system plant (Figure 2), and 2) any future field sampling that may be required to meet the objectives of the Removal AOC. Figure 3 shows the planned phasing of tasks that will be completed during this work.

2.1 Investigation Activities

2.1.1 Treatment Plant Evaluation Activities

Tasks to be completed include:

- Collecting influent (to the treatment plant) leachate samples.
- Collecting post unit-process, in-line samples from the treatment plant system.
- Collecting effluent (from the treatment plant) wastewater samples.
- Collecting air samples prior to and after the air-phase granulated activated carbon (GAC) filters of the treatment plant.
- Collection of a core sample of activated carbon from each of the two (2) GAC liquid-phase filters of the treatment plant.

As planned, these activities will be conducted according to the sequence shown in Figure 3.

2.1.2 Field Activities

Field sample collection and subsequent physical and chemical analysis may be required under the actions of the Removal AOC. This section of the FSP summarizes work that may be conducted during future field sampling.

2.1.2.1 Preliminary Activities

Preliminary field activities will include:

- Coordinating arrangements with RI subcontractors and investigation personnel.
- Confirming access approvals (and permits if required).
- Staging equipment to the Site.
- Conducting an on-site orientation meeting with all subcontractor and project staff.
- Completing a site reconnaissance and initial walkthrough air monitoring survey.
- Establishing site exclusion zones, contaminant reduction zones, and the support zones previously identified in the Health and Safety Plan.
- Constructing the decontamination pad and area.

2.1.2.2 Field Investigation Activities

After completion of the preliminary activities, field efforts will begin. Tasks which may be completed include:

- Air monitoring investigation.
- Sampling groundwater.
- Collecting surface and subsurface soil samples.
- Collecting sediment samples and surface water samples.

These activities will be conducted according to the sequence shown in Figure 3, or new or revised schedules prepared as part of the Work Plan Addendum.

2.2 Sampling and Analysis Program

The sampling and analytical methodologies to be performed during the implementation of this work plan are described below and presented in Tables 1a and 1b. Standard Operating Procedures (SOP) for each field method are provided in Appendix A of this FSP.

For the treatment plant evaluation, samples for laboratory analysis will be collected at all locations approximately one-half hour to three (3) hours after start-up on Day 1 of the leachate treatment system trial run, at the mid-way point of the test, and just prior to shutdown on Day 5. Table 2 summarizes the samples that will be collected from each media and location, and identifies the analysis that will be completed for the samples. At each sampling point throughout the treatment plant, samples will be taken directly from in-line sample ports into the respective containers. For future field investigative sampling, samples for laboratory analysis will be collected on a "To Be Determined" basis, to adequately address leachate releases or seeps found to occur at the Site. In all cases, appropriate preservatives will be added to the containers before or after the samples have been collected, depending on laboratory preferences or protocol.

To provide control and tracking of sample results, specific sample location and identification procedures will be observed. Field sample identification labeling will include, at a minimum:

- site name
- sample identification
- analysis to be performed
- collection date
- collection time
- sampler's initials.

Each sampling location in the treatment plant will be marked prior to the test. In the unlikely event that field conditions necessitate moving a planned sample location, the U.S. EPA shall be notified for approval and the new location will be described in the sampling team's field log book. Sample containers, preservatives, and holding times which apply during sampling are summarized in Table 3.

Table 3 also summarizes the analytical methods that shall be followed for samples taken as described above. All liquid-phase TCL analyses (volatiles and semi-volatiles), pesticides, filtered and unfiltered metals will follow U.S. EPA SW846 Methodology (U.S. EPA, 1986). Analysis for air-phase volatiles will follow U.S. EPA TO-14 Methodology. Analysis for mirex, photomirex, and kepone shall follow the Ruetgers-Nease Method (Pulsed Positive Negative Chemical Ionization, Version 4.1) developed and validated for this Site. Wet chemistry analysis for ammonia, total organic carbon (TOC), chemical and biological oxygen demand (COD and BOD, respectively), total dissolved and total suspended solids (TDS and TSS,

respectively), total phenolics, and total oil and grease shall follow standard U.S. EPA methodologies, specifically as described in Table 3.

The analytical program is discussed in detail in the Quality Assurance Project Plan (QAPjP) provided in Section II of this Volume of the Work Plan for the Removal AOC.

2.3 Project Organization

Figure 4 is the project organization chart illustrating the structure for field activities. Specific responsibilities of personnel are described below.

2.3.1 Project Coordinator

Ralph M. Pearce, P.E. of RNC is the Project Coordinator for this investigation. The alternative Project Coordinator is Ms. Sonya F. Napier, Esq. of RNC. The responsibilities of the Project Coordinator include:

- provide an interface with the U.S. EPA Remedial Project Manager (RPM), the OEPA Project Coordinator, the Project Director, and the Project Manager.
- approving on-site activities.
- initiating modification requests.
- ensuring that the terms of the November 17, 1993 Removal AOC are met.

2.3.2 Project Director

P. Steven Finn, C.Eng. of Golder Associates is the Project Director for this investigation. The responsibilities of the Project Director include:

- providing an interface with the Project Coordinator and the Project Manager.
- committing Golder Associates resources to performing for the Site investigation.
- coordinating technical direction of the project.

2.3.3 *Project Manager*

Geoffrey R. Forrest, C.P. Eng. of Golder Associates is the Project Manager for this investigation. The responsibilities of the Project Manager include:

- providing an interface between the Project Coordinator and the Site Manager.
- implementing project plans.
- coordinating project activities.
- coordinating project personnel and staffing.
- completing project deliverable reviews.
- providing input on technical direction.

2.3.4 Field Operations Manager

Joseph E. Cavanagh of Golder Associates will be the Field Operations Manager for this investigation. The responsibilities of the Field Operations Manager include:

- managing field operations.
- reviewing and evaluating field data.

- implementing FSP, HSP, and QAPjP protocols.
- enforcing safety procedures.

2.3.5 Health and Safety Officer

William E. Harris of Golder Associates will be the Health and Safety Officer for this investigation. The responsibilities of the Health and Safety Officer include:

- selecting proper clothing and equipment to ensure the safety of on-site personnel.
- confirming each field team member's suitability for work based on a physician's recommendation.
- monitoring on-site hazards and conditions.
- monitoring the effectiveness of the Health and Safety Plan.

2.3.6 Project QA/QC Manager

Robert M. Glazier of Golder Associates will be the Project QA/QC Manager for this investigation. The responsibilities of the Project QA/QC Manager include:

- conduct performance and system audits (if required).
- review all documents with respect to adherence to QA/QC procedures in the QAPjP.
- preparation of analytical data tables and quality assurance reviews.
- recommend and institute corrective actions based on any reviews and audits which may be conducted during this work.

2.3.7 Site Safety Manager

Joseph E. Cavanagh of Golder Associates will be the Site Safety Manager for this investigation. The Site Safety Manager is responsible for the safety of all field personnel at the Site, which includes determining the hazards associated with individual phases of the investigation, reviewing safety matters during field operations, and notifying the Health and Safety Officer of any unsafe conditions or practices noted. The Site Safety Manager will report to the Health and Safety Officer, and will be responsible for the following:

- assuring all safety equipment and protective clothing availability at the Site.
- completing project safety briefings and reports.
- investigating accidents and implementing appropriate corrective actions.
- conducting daily safety briefings.

2.3.8 U.S. EPA Remedial Project Manager and OEPA Project Coordinator

Ms. Sheila Sullivan and Mr. Joseph Trocchio are the Remedial Project Manager and Project Coordinator, respectively, for U.S. EPA Region 5 and the OEPA for this investigation. Their responsibilities include:

- technical review and approval of all plans and data submitted as part of this Work Plan.
- coordination of Work Plan activities with the Project Coordinator.
- authority vested in an On-Scene Coordinator and a Remedial Project Manager by the National Contingency Plan, 40 CFR Part 300, as amended, including the authority as provided therein to halt conduct, or direct any work described in the Work Plan, or to direct

any response action undertaken by the U.S. EPA when conditions at the facility may present an imminent and substantial endangerment to the public health and welfare or the environment. The Project Coordinator's actions shall, at all times, be controlled and limited by provisions of the National Contingency Plan, 40 CFR Part 300.

2.3.9 U.S. EPA/OEPA QA/QC Manager

The U.S. EPA/OEPA QA/QC Manager for this investigation will be responsible for assuring that all laboratory activities and analytical data are of sufficient quality to meet the objectives of the investigation.

The external system performance audits of the project laboratories are the responsibility of U.S. EPA Region 5 CRL. Although neither U.S. EPA Region 5 or OEPA are responsible for data validation/data evaluation, both agencies reserve the right to perform data validation/data evaluation on any of the data generated from this investigation.

3.0 FIELD INVESTIGATION ACTIVITIES

This section describes in detail the Removal Action field investigation activities to be performed during the 1) leachate treatment system performance evaluation (Section 3.1) and 2) any future investigative field sampling necessary to perform the actions of the Removal AOC.

During the proposed five (5) day performance evaluation trial run air monitoring, and liquid-phase and vapor-phase leachate collection will be performed. During field sampling, air monitoring, surface water, sediments, and/or groundwater may be sampled at the Nease Chemical Site under the sampling protocols provided herein.

3.1 Treatment Plant Performance Evaluation

3.1.1 Air Monitoring

In order to evaluate the performance of the on-site leachate treatment system (a dual, liquid-phase/vapor-phase removal system), both air monitoring and air sampling shall be conducted during the performance evaluation trial run of the leachate treatment system. Air monitoring within the "metal building" that houses the treatment system shall be performed to determine the nature and magnitude of the ambient air within the building, both for safety reasons and background level measurements.

A survey of the metal building housing the on-site leachate treatment system using an organic vapor analyzer (OVA) or an HNU Photoionizer Instrument (HNU) or equivalent will be completed prior to the initiation of any field work. The survey will identify areas with elevated levels of volatile organic compounds (VOCs) that

may require inclusion as an exclusion area or to establish the presence of background levels of VOCs that could effect the results of air sampling analyses to be performed on the treatment system proper. If no elevated levels of volatiles are detected during the walk through, the commencement of operation of the treatment system shall begin.

3.1.1.1 Operation of the HNU Model P1-101 Photoionizer Instrument

In general, the operation of the HNU instrument or equivalent will be as described in the owners manual. Briefly these procedures are as follows: Charge the instrument battery for sixteen (16) hours prior to use. Attach the probe to the readout module. Turn the function switch to the battery position, listen to ascertain that the fan is operating, and very briefly observe that the lamp is glowing. Turn the function switch to the standby position and zero the instrument. Connect the instrument to the calibration canister which contains isobutylene in an air matrix. Turn the function switch to the 0-200 ppm range (X10) and adjust the calibration. The meter should be operated on the 0-20 ppm range for greatest sensitivity. Simply turn the instrument to the off position once sampling is completed.

3.1.2 Treatment Plant Sampling

Leachate and gas samples will be collected during the leachate treatment system performance evaluation trial run. Media to be collected and analysis to be performed are listed in Table 1. Sample location description and estimated sampling times are listed in Table 2. Table 3 lists the analytic methods to be used for sample analysis. (These are included in this field sampling plan for completeness of documentation.) The Data Quality Objectives (DQOs) of this Work Plan are listed in Table 4. This section describes the sampling procedures,

and data gathering methodologies which will be utilized to achieve the DQOs which are necessary to support the evaluation of the on-site leachate treatment system.

If at any time during the leachate treatment system performance evaluation additional data needs are recognized that may require additional field investigations not specified herein, the FSP will be amended accordingly. Any such revisions or amendments to the FSP will be submitted for review and approval by the Respondents Project Manager and the U.S. EPA Project Manager prior to implementation. Upon approval, the amended FSP can be implemented. Minor changes may be made in the field with the concurrence of U.S. EPA or their on-site representative.

3.1.2.1 Leachate Collection

The performance evaluation will commence after a brief start-up period. The evaluation will be conducted over an anticipated time of five (5) days. The duration of the test will allow for evaluation of variations in the influent quality and quantity, and the ability of the treatment system to handle these variations. Samples will be collected on the first (1st), third (3rd), and fifth (5th) days , or their hourly equivalents, during the test. Sample locations (Figure 2) have been determined for their use in the evaluation of the plant performance.

At the commencement of the performance evaluation operation, influent samples will be collected from the surge tank, T-1 (see Figure 2). The residence time throughout the plant has been estimated based on the hydraulics of the system. At pre-determined time intervals based on residence time of each unit process, and

piping of the system, samples will be collected at each of the following locations throughout the plant:

Sample	Description	Estimated Collection Time (min)
T-1	Surge Tank (influent)	0
SP-1	Influent to Air Stripper	2
SP-2	Influent to Bag Filter 2	24
SP-3	Influent to GAC 1	26
SP-4	Influent to GAC 2	43
SP-5	Effluent	60

During the performance evaluation operation period, the pressure gauges on the GAC units will be monitored to assess the potential of metals build-up on the GAC causing the GAC to clog.

Aqueous Sample Collection Protocol

At each sampling location throughout the treatment plant, samples will be collected directly from the sample port of the plant into the appropriate container. Sample containers are supplied directly from laboratories in a state ready for sampling. The appropriate preservatives will be added to the containers before the samples have been collected as described in Table 3.

The analytical procedures for this performance evaluation are described in detail in the Quality Assurance Project Plan (QAPjP). Conventional parameters such as pH, specific conductance, etc., will be analyzed in the field by Hach Testing Kits or the appropriate field instrument as described in Table 2A, and described in the

Treatment Plant Performance Evaluation Work Plan. Where necessary, field testing equipment will be decontaminated prior to use between sample locations according to the procedures described in Section 7.0.

3.1.2.2 Gas Sampling

To evaluate the air stripper and vapor-phase GAC unit processes of the leachate treatment system, five (5) gas samples (four primary and one duplicate) will be taken prior to (i.e. the gas effluent from the air stripper) and after the vapor-phase GACs as described in Table 2A. The samples will be collected using Summa Passivated Cylinders (Summa cylinders), and analyzed for VOCs. Sampling will be performed on the first (1st) and fifth (5th) days of the performance evaluation trial run.

Gas Sampling Collection Protocols

This technical procedure is to be used to establish a uniform procedure for collection of gas samples for laboratory quantification of constituents. This procedure is based upon U.S. EPA Method TO-14, which is widely considered to be one of the most reliable and sensitive test methods for gas toxics.

Equipment is to include:

- Summa passivated cylinders equipped with vacuum flow regulators. The cylinders will have been cleaned and evacuated before and after sampling by the analytical laboratory.
- Teflon or stainless steel tubing and connections to the Summa canisters.
- Shipping containers and chain-of-custody documentation.

The procedure for collection of gas samples using the Summa cylinders is described in detail in Appendix A of the FSP. Briefly, the gas samples will be collected at existing ports (Figure 2) of the vapor-phase portions of the leachate treatment system. Teflon tubing will be connected to the ports and to the canisters, and the canisters (under negative pressure) will be opened to draw-in gas from the treatment system. The canisters will come to equilibrium and the valves will be closed on completion of the sampling.

3.2 Field Sampling

3.2.1 Air Monitoring

For both health and safety reasons and background level measurements, air monitoring shall be performed in the field to determine the nature and magnitude of the ambient air whenever any future field sampling is performed.

The monitoring shall be performed using an organic vapor analyzer (OVA) or an HNu Photoionizer Instrument (HNu) or equivalent. The monitoring will identify areas with elevated levels of organic compounds that may require inclusion as an exclusion area. If no elevated levels of organics are detected during monitoring, commencement of field sampling activities shall begin.

Operation of the HNu is described above, in Section 3.1.1.1.

3.2.2 Surface Water and Sediment Sampling Protocols

Sediment samples, if needed for future investigative work, shall be collected using clean stainless steel utensils and dewatered as much as possible. Samples will be collected from the stream bottom to a depth of six to eight inches. Samples for volatile organic analysis will be placed directly in the appropriate bottleware. The

remaining sample aliquot will be placed in a stainless steel bowl, thoroughly homogenized and placed into the appropriate bottleware.

Sediment samples collected from the bottom of a pond will be obtained using a clean Ponar[R] dredge sampler from a small boat or by personnel wearing waders if the pond is sufficiently shallow. This dredge is a clam shell scoop and is lowered into the pond on dedicated nylon rope until it reaches the bottom. The dredge closes and collects the sediment on contact with the pond bottom. The dredge is retrieved, opened and the sediment is transferred to the appropriate bottleware using a clean stainless steel spatula filling the containers for volatile organic analysis first.

At some of the sediment sampling locations or other areas of standing and/or flowing surface water, a water sample may be collected. Surface water sampling will proceed from the farthest downstream location and proceed to upstream locations to minimize potential cross contamination from suspended material in the stream from sampling activities. Surface water samples will be collected before sediment samples. Dependent upon depth and stream velocity, samples will be either collected directly into the appropriate sample containers at a point upstream of the samplers position or through the use of a long-handled polyethylene grab sampler from the bank of the stream.

Stream velocity and flow characteristics, pH, conductivity, DO and temperature will be noted at each sampling location. Stream velocity will be measured using a velocity meter (e.g., Pygmy). Flow will be determined based on the calculated cross-section area at the stream sampling station.

A water sample collected from a pond will be obtained using a Kemmerer sampler. The sampler will be slowly lowered to the mid-depth of the water column and allowed to remain at that depth for several minutes. It will then be retrieved and the sample transferred to the appropriate bottleware, filling the vials for volatile organic analysis first.

3.2.3 Groundwater Purging and Sampling Protocols

Groundwater samples, if needed for future investigative work, shall be collected from monitoring wells or residential wells.

Prior to purging and sampling, the well protective casing, lock and apron will be inspected for damage or signs of tampering. Static water depth and total depth will be measured using a decontaminated probe with permanent depth markings. The static and total depth will be used to calculate the volume of standing water. One bailer of groundwater from the top of the water column and one from the bottom of the well will be collected to detect immiscible layers before purging.

Where possible, at least three volumes will be removed prior to sampling. Less than this amount will be purged only if all standing water is removed before three volumes are purged.

Bailing or pumping methods will be used during purging to ensure that all standing water is removed. Purging will take place at the top of the water column where possible. Suction hoses for centrifugal pumps will be capped with a "foot valve" to prevent purged water from flowing back into the well as the hose is removed.

Sampling whenever possible will take place on the same day after purging procedures are completed. Where the well is purged dry, purging procedures are described in Appendix A, Section 8.0.

Samples will be collected from monitoring wells using Teflon bailers dedicated to each well. The first bailer withdrawn from the well will be checked for temperature, pH and specific conductance. Subsequent bailer volumes collected from within the screened interval will be used to directly fill sample bottles. Bottles intended for volatile organic compound analysis will be filled first, followed by the extractable organic fraction and finally the inorganic analysis bottles filled last.

If artesian flow conditions exist at any of the groundwater sampling locations, the well will be allowed to flow three well volumes prior to sample collection.

3.3 Quality Assurance/Quality Control Samples

3.3.1 Treatment Plant QA/QC

Standard field sampling procedures call for preparation and submittal of three types of QC samples from the field and submitted as duplicate samples to the laboratory. Samples include:

- Trip Blank - One laboratory prepared trip blank will accompany each sample cooler containing aqueous samples to be analyzed for volatile organics. They will be prepared at the laboratory using deionized water, transported to the Site, handled like a sample, and returned to the laboratory for analysis. One trip blank will be submitted per sampling day (1st, 3rd, 5th day of performance evaluation) for liquid samples analyzed for volatile organics.

- Field Blanks (Rinsate Blanks) - Field blanks are prepared in the field to ensure a sampling device (e.g., bailer or pump) has been effectively cleaned. A rinsate blank will be collected from the metals filtering apparatus.

- Field Duplicate Samples - are samples from a single source, which are split into two (2) distinct samples. A field duplicate will be collected from the influent on Day 3 of the performance evaluation for all analysis, as described in Table 2A.

3.3.2 Field Quality Control Checks

Field Quality Control Checks will be conducted during any field investigation through the use of the following:

- Trip Blank - One laboratory prepared trip blank will accompany each sample cooler containing aqueous samples to be analyzed for volatile organics. They will be prepared at the laboratory using deionized water, transported to the Site, handled like a sample, and returned to the laboratory for analysis. One trip blank will be submitted per shipment on each day of sampling.

- Field Blanks or Rinsate Blanks - Field blanks are analyzed to check for procedural contamination at the Site which may cause sample contamination. Rinsate blanks are prepared in the field to ensure a sampling device (e.g., bailer or pump) has been effectively cleaned. The sampling device is filled with deionized water or deionized water is pumped through the device, transferred to the appropriate sample bottles, preserved and returned to the laboratory for analysis. Rinsate blanks for soil/sediments are prepared by pouring deionized water through the sampling equipment directly into appropriate bottleware and one will be submitted for every 10 soil/sediment samples collected. Field blanks for air analysis will be prepared by opening each canister/cartridge on-site and allowing any ambient contaminants to passively diffuse onto the collection media. The field blank will be collected at an upwind location. One field blank will be submitted for the air samples. One field blank will be collected for every 10 groundwater samples. As each well has dedicated sampling equipment, a rinsate blank will not be collected.

However, to meet Region V requirements, field blanks for groundwater will be collected at a frequency of one per every 10 or fewer samples. One field blank will be collected for every 10 surface water samples or everyday of sampling (whichever is more frequent).

- Field Duplicates or Replicates - Two sets of samples from a single sample location are obtained (replicates) or prepared (duplicates), labeled with unique sample numbers, and submitted to the laboratory to determine analytical precision and sample representativeness. Field duplicates/replicates will be collected at a frequency of 1 per 10 samples per matrix.

- Matrix Spike/Matrix Duplicate Samples - Matrix Spike (MS) and Matrix Spike Duplicate (MSD) samples will be submitted as further QC checks. One MS and MSD in every twenty (20) groundwater, surface water, soil, sediment and fish samples will be collected and submitted for organic analysis. These will allow accuracy to be determined by the recovery rates of compounds (the matrix spike and/or surrogate spike compounds defined in the analytical methods). The purpose of these laboratory spikes is to monitor any possible matrix effects specific to samples collected from the site. The addition of known concentrations of compounds/constituents into the sample also monitors extraction/digestion efficiency.

  The analysis of MS and MSD samples checks the precision by comparison with the respective spiked recoveries. In addition, any non-spiked analytes present in an unspiked sample will be compared with the results of the non-spiked compounds in the MS and MSD. Therefore, data will be assessed for precision for this triplicate analysis. The analysis of laboratory duplicates (inorganics) is used as an assessment of analytical precision.

Duplicate/replicate and matrix spike sample aliquots will be acquired for groundwater and surface waters by collecting sequential grab samples after the collecting of the initial sample aliquot. Therefore, the specific sample location which will be used for matrix spikes and duplicates/replicates will be chosen by the Field Operations Manager. Multiple air samples will be collected

simultaneously at a given sample location for replicate samples. Soil/sediment duplicate or replicate and matrix spike samples will be collected by splitting the sample between the sample container, duplicate container, and matrix spike container. Homogenizing of soil/sediment samples will not be performed for the aliquots designated for volatile organics compounds; therefore, they are designated as sample replicates. If insufficient soil/sediment samples are present at a particular location to collect the necessary volumes, a single sample bottle will be split at the laboratory for MS and MSD analysis.

3.3.3 Laboratory Quality Control Checks

3.3.3.1 Trace Organic Analysis: Volatiles, Semivolatiles, Pesticides, Mirex, Photomirex, and Kepone

General QC protocols for trace organic analyses include:

- Field blanks, when applicable to detect contamination introduced during sampling, shipping and handling.
- A minimum of one procedural blank (method blank) in every 20 samples analyzed to detect contamination during analysis.
- One matrix spike and one matrix spike duplicate per every 20 samples, or per case, whichever is more frequent; to determine recovery, precision, and the presence of matrix effect.
- Surrogate standards and internal standards to quantitate results, determine recoveries and to account for sample-to-sample variation.
- Routine analysis of performance evaluation samples and blind spikes to document method accuracy.
- Multilevel initial calibrations of instruments to establish calibration curves.

- Continuing calibration standards at least once every 12 hours of instrumental analysis for accurate quantitation, and recalibration if these do not meet method criteria.

- Calibration of GCs and GC/MSs according to the appropriate methods.

- Tuning of GC/MS systems every 12 hours to meet method criteria using BFB (bromofluorobenzene) for volatile organics analysis, and DFTPP (decafluorotriphenylphosphine) for semivolatile analysis, as described in Section 8.0.

3.3.3.2 Trace Metals Analysis

General QC protocols for trace metals analyses include:

- Analysis of moderate-to-high concentration levels by ICP (inductively coupled plasma spectroscopy) or flame atomic absorption spectroscopy.

- Analysis of low-level metal concentrations by graphite furnace atomic absorption spectroscopy.

- Calibration blanks and method blanks prior to and between the analysis of samples.

- Multilevel calibration curves generated by analyses of individual standards (AA) or mixed standards (ICP).

- Initial calibration verification (ICV) and Continuing Calibration Verification (CCV) at a minimum of one every ten samples to verify instrument calibration.

- ICP Interference Check Standards after initial calibration and after samples are analyzed.

- Recalibration and reanalysis of applicable samples if check standard response deviates by more than 10% (15% for mercury) from the initial calibration.

- A minimum of one matrix spike in every 20 samples or one for each sample matrix type.
- A minimum of one duplicate in every 10 samples analyzed.
- A minimum of one Laboratory Control Spike for every 20 samples analyzed.
- A minimum of one method blank for every 20 samples.
- Digestion of solid or sludge samples according to respective methodologies.

3.3.3.3 Control Limits

Control limits used will be those defined by the respective methodology.

For the five non-routine analytes (MPK, diphenyl sulfone, and 3,4-dichloronitrobenzene, control limits will be established from the method validation data. The control limits will be ±3 SD from the mean.

4.0 EQUIPMENT AND CALIBRATION

To ensure that measurements made in the field have been performed with properly calibrated instruments, field personnel will follow the procedures described in the Equipment Calibration and Maintenance Owners Manual. All field equipment will be calibrated (at a minimum, twice daily, prior to and after use), maintained, and repaired in accordance with manufacturer's specifications. These activities will be noted in a maintenance log book. Despite even the most rigorous maintenance program, equipment failures do occur. When equipment cannot be repaired, it is returned to the manufacturer for repairs. Calibration procedures for each instrument that will be used in the field for acquisition of data are provided below:

Equipment	Maintenance/Calibration	Frequency
PID photoionization detector	Calibrate with isobutylene gas.	Start and end of each day.
pH meters	Calibrate with three pH buffer solutions.	Before and after use.
Temperature	Check against mercury thermometer.	Every ten samples.
Specific conductance	Check with two calibration solutions.	Before and after use.

5.0 SAMPLE HANDLING

After a sample has been collected, proper sample handling procedures ensure that the sample remains representative. These procedures include: 1) identification; 2) preparation for shipment; 3) proper storage of the sample; and 4) completed chain-of-custody.

5.1 Chain-of-Custody

All samples will be collected and handled in accordance with standard U.S. EPA chain-of-custody protocols. The objective of chain-of-custody is to maintain an accurate written custody record that traces the possession and handling of the sample from collection through analysis.

Custody is defined if a sample:

- Is in one's actual possession, (or)
- Is in one's view, after being in one's physical possession, (or)
- Is in one's physical possession and then locked up so that no one can tamper with it, (or)
- Is kept in a secured area, restricted to authorized personnel only.

One member of the project sampling team will be appointed field custodian. The field custodian will assign a unique chain-of-custody number to each sample collected before it is submitted for shipment to the laboratory for analysis. Sample storage and custody is the responsibility of the field custodian. The field custodian records each sample on a Chain-of-Custody form as the sample is collected. Upon laboratory receipt of samples, a copy of the Chain-of-Custody form will be

returned to Golder and RNC. The Chain-of-Custody form will remain with the sample until such time as the sample is destroyed or discarded.

5.2 Sample Identification

A unique designation will be used to identify individual samples for each matrix and location. Sample identification numbers will be assigned in the field and will be used to identify the sample on the chain-of-custody log. Numbers will consist of a site name, sample identification, analysis to be performed, collection date, collection time, and sampler's initials.

For the performance evaluation, sample identification shall be notated by the sample locations as used in Tables 1, 2A, 2B, and 4. These sample identification numbers will be used in field log books, chain-of-custody forms, laboratory results, and the final IRM Report. Analysis to be performed are described as Parameters of Analysis as used in Table 4. Collection dates, times, and sampler's initials are evident. Each sample will be labeled using waterproof ink. Labels will be filled out at the time of collection. All sample identifications will be entered into the sample log books as described in Section 6.3.

5.3 Sample Packaging

After labels are checked, sample containers will be placed in a transportation case (i.e. cooler, cooled to 4°C using "blue ice") along with the appropriate chain-of-custody record form. The transportation case will be sealed and locked. The following packaging procedures will be followed:

- Using duct tape, secure the drain plug at the bottom of the transportation case to ensure that water from sample container breakage does not leak out of the case.

- Line the bottom of the case with a layer of cushioning absorbent material such as vermiculite or foam pellets.

- Place sample container properly labeled and with a sealed lid in a sealed plastic bag in transportation case.

- Place all sample containers in the case.

- "Blue Ice" will be placed in the case to keep samples cooled. Prior to sample packaging, samples will be kept in on-site refrigerators and/or coolers provided for temporary sample storage.

- For large glass containers, pieces of carved out plastic foam or sheets of bubble plastic will be used to help keep containers in place and to prevent breakage. Additional absorbent material will be added as necessary and appropriate.

- Small containers such as 40 millimeter vials will be placed in small sandwich bags and wrapped with bubble plastic. Prior to sample packaging, samples will be kept in on-site refrigerators and/or coolers provided for temporary storage.

- The documents accompanying the samples will be placed in a plastic bag attached to the inside of the case lid.

- The lid of the case will be closed and fastened. Duct tape will be used to seal between the lid and the body of the case. The tape will be wrapped in two directions around the case to ensure that the lid does not open if the latch becomes unfastened. Custody seals will be signed and attached to the cooler prior to shipment.

- The following information will be attached to the outside of the transportation case: name and address of receiving laboratory with return address, arrows indicating "This End Up" on all four sides, and "This End Up" label on the top of the lid.

5.4 Sample Shipping

Each shipping container will be accompanied by a packing slip that contains the following information:

- Laboratory address and sample custodian identification.
- Date shipped.
- Return address and Site Manager's identification.
- Total number of containers included in shipment.

All samples will either be shipped by direct-courier or a 24-hour delivery courier. Upon receipt of shipment, the laboratory will check the packing slip to verify that all the containers have arrived, and each container will be inspected for evidence of any tampering. The laboratory sample custodian will then remove each sample and verify the condition of the sample and compare sample bottle information to the chain-of-custody sheet to ensure the accuracy and completeness of all documentation. If any inconsistencies are present, they will be documented on the Chain-of-Custody Form. The laboratory sample custodian will inform the Site Manager by phone upon receipt of sample shipment, and of any problems encountered. Written verification of sample receipt, condition, and analyses request form will be sent to the Project Manager by the laboratory.

6.0 FIELD DOCUMENTATION

In order to ensure that all pertinent information and data collected during the performance evaluation are documented completely and correctly, the following procedures and protocols described in the following sections will be implemented.

6.1 Log In/Log Out record

A sign-in/sign-out log will be kept at the office trailer for use by authorized personnel. Unauthorized personnel will not be granted access on site unless approved by RNC in advance. The record will contain at a minimum: date, name, organization, and entrance/exit times.

6.2 Field Notebooks

All information pertinent to the field investigation will be recorded in bound and numbered field notebooks. Each team member will be assigned an individual notebook. Field records should at a minimum contain the following information:

- Date
- Time of each data entry.
- Description of work being performed that day.
- Names and affiliations of all personnel at location.
- Weather conditions on site.
- Location and type of activity.
- Visual observations.
- Pertinent field data (pH, specific conductance, etc., and any other measurements).

- Serial numbers, if any, on seals and transportation cases.
- Name of field custodian.
- Photographs taken, including date, time, direction faced, description of subject or activity, sequential number of the photo and film roll number will be recorded in the field notebook.

Specific sample information will be compiled into the field log notebook. The following information will be included:

- Unique sample number.
- Sample date.
- Sampler's initials.
- Number of samples.
- Analysis performed.
- Further analysis required.
- Date shipped to the lab.
- Method of shipment.

All field notebooks will be standard engineering hardbound books. All field notebooks will be photocopied so that copies of field notes can be kept.

6.3 Photo-Documentation

All photographs will record time, date, site location, general direction faced, sequential number of photograph and roll number, and brief description of the subject in a field notebook.

6.4 Correspondence/Communications

All correspondence received or sent from the field office will be dated and labeled with a project filing identification number. All telephone conversations will be documented and filed.

7.0 EQUIPMENT DECONTAMINATION

This section describes procedures for decontaminating field sampling equipment. Detailed personnel decontamination procedures are discussed in Section 9.3 of the Health and Safety Plan. Decontamination protocols will be strictly adhered to in order to minimize the potential for cross-contamination between sample locations and contamination of areas off-site.

7.1 General Consideration

The following general considerations will be adhered to concerning decontamination efforts:

- All decontamination and subsequent use of decontaminated equipment will be documented in a field notebook.
- If visual signs such as discoloration indicate that decontamination was insufficient, the equipment will again be decontaminated. If the situation persists, the equipment will be taken out of service until the situation can be corrected.
- All spent wash and rinse waters will be collected, and transferred to and stored in an on-site tank pending proper disposal.
- Verification of the sampling equipment cleaning procedures will be documented by the collection of rinsate blank samples.
- Drill cuttings from off-site locations will be containerized and handled according to procedures described in Section 9.4 of the Health and Safety Plan (Section III, Vol. 2).
- All properly decontaminated equipment will be stored in plastic bags (if possible) when not in use.
- All fluids and solids generated from sample location decontamination will be transported on-site and disposed of

according to the Hazardous Materials Handling procedures described in the Health and Safety Plan, Section 9.4.

- Personnel and equipment will proceed directly to the decontamination pad between sample locations and at the end of each day.

7.2 Heavy Equipment

Drill rigs, backhoe buckets, and appropriate other heavy equipment will be decontaminated prior to the commencement of field activities, between each sample location, and after the completion of field activities at the on-site decon pad. Decontamination procedures will be as follows:

- Remove all loose soil from equipment with a brush.
- Steam or high pressure wash using non-phosphate soap.
- Potable water rinse.
- All wash water will be stored in an on-site tank pending proper disposal. All soil will be containerized and stored in the on-site warehouse.

7.2.1 Drilling Equipment

Drilling equipment (i.e., rods, auger flights, bits, casing) will be cleaned between each boring location and sample. Decontamination procedures will be as follows:

- Remove all loose soil.
- Steam or high pressure wash using non-phosphate soap.
- Potable water rinse.

- All wash water will be stored in an on-site tank pending proper disposal. All soil will be containerized and stored in the on-site warehouse.

7.3 Sampling Equipment

Equipment used to collect environmental samples will be cleaned prior to its use and between each sample location and after the final use. All equipment will be transported and stored on plastic sheets.

7.3.1 Soil and Sediment Sampling Equipment

Soil/sediment sampling equipment will be decontaminated at the on-site decon pad. Equipment that will be cleaned will include: split spoons, Shelby Tubes, hand augers, stainless steel scoops/trowels, and compositing containers. Specific procedures are as follows:

Inorganics	Organics
1. Removal loose soil	1. Remove loose soil
2. Non-phosphate soap wash	2. Non-phosphate soap wash
3. 0.1 N HCL	3. Tap water rinse
4. Tap water rinse	4. Deionized/Distilled water rinse
5. Rinse with Deionized water	5. Methanol rinse
6. Air dry	6. Pesticide grade hexane rinse
	7. Methanol rinse
	8. Four rinses with deionized/distilled water
	9. Air dry

7.3.2 Groundwater and Surface Water Sampling Equipment

Equipment used for groundwater and surface water sampling will be decontaminated before sampling activities begin and between each sample location if dedicated equipment is not used. This equipment will include: pumps, hoses, glass beakers and bailers. The following procedures will be used for decontaminating equipment:

Inorganics	Organics
1. Removal loose soil/solid	1. Removal loose soil/solid
2. Non-phosphate soap wash	2. Non-phosphate soap wash
3. 0.1 N HCL	3. Tap water rinse
4. Tap water rinse	4. Deionized/Distilled water rinse
5. Rinse with Deionized water	5. Methanol rinse
6. Air dry	6. Pesticide grade hexane rinse
	7. Methanol rinse
	8. Four rinses with deionized/distilled water
	9. Air dry

7.3.3 pH, Temperature, Dissolved Oxygen and Depth to Water Probes

These probes used during groundwater and surface water sampling will be decontaminated via the procedures specified below.

- Wash with non-phosphate detergent solution.
- Potable water rinse.
- Deionized water rinse.

All equipment will be transported and stored in plastic sheeting.

7.4 Electronic Equipment

Electronic equipment such as PIDs, FIDs, explosimeters, and portable air pumps will be decontaminated prior to their initial use and at the end of the each day. The procedure for decontaminating this equipment is as follows:

- Remove particulate contamination.
- Wipe down with clean damp cloth (deionized water).
- Air dry.

Equipment will be wrapped in plastic and stored in the office trailer when not in use.

TABLE 1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
Aqueous				
T-1	Volatile Organics	SW846 Method 8240	3	Primary
			1	Field Duplicates
			1	Trip Blank (1)
	Semi-Volatile Organics (2)	SW846 Method 8270	3	Primary
			1	Field Duplicate
	Pesticides	SW846 Method 8080	3	Primary
			1	Field Duplicate
	MPK	R-N Method (3)	3	Primary
			1	Field Duplicate
	Filtered Metals	SW846 Methodologies(6)	3	Primary
			1	Field Duplicate
	Unfiltered Metals	SW846 Methodologies(6)	3	Primary
			1	Field Duplicate
	Conventional Parameters	(4)	3	Primary
			1	Field Duplicate
SP-1	Volatile Organics	SW846 Method 8240	2	Primary
	Filtered Metals	SW846 Methodologies(6)	3	Primary

TABLE 1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
	Unfiltered Metals	SW846 Methodologies(6)	3	Primary
	Conventional Parameters (TSS Only)	EPA 160.2	3	Primary
SP-2	Volatile Organics	SW846 Method 8240	2	Primary
	Filtered Metals	SW846 Methodologies(6)	3	Primary
	Unfiltered Metals	SW846 Methodologies(6)	3	Primary
SP-3	Filtered Metals	SW846 Methodologies(6)	3	Primary
	Unfiltered Metals	SW846 Methodologies(6)	3	Primary
SP-4	Semi-Volatile Organics	SW846 Method 8270	2	Primary
	Pesticides	SW846 Method 8080	2	Primary
	MPK	R-N Method (3)	2	Primary

TABLE 1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
	Filtered Metals	SW846 Methodologies(6)	2	Primary
	Unfiltered Metals	SW846 Methodologies(6)	2	Primary
SP-5	Volatile Organics	SW846 Method 8240	3	Primary
			1	MS/MSD
			1	Trip Blank (1)
	Semi-Volatile Organics (2)	SW846 Method 8270	3	Primary
			1	MS/MSD
	Pesticides	SW846 Method 8080	3	Primary
			1	MS/MSD
	MPK	R-N Method (3)	3	Primary
			1	MS/MSD
	Filtered Metals	SW846 Methodologies(6)	3	Primary
			1	MS/MSD
	Unfiltered Metals	SW846 Methodologies(6)	3	Primary
			1	MS/MSD
	Conventional Parameters	(4)	3	Primary
			1	MS/MSD
	NPDES Parameters	(5)	1	Primary
			1	MS/MSD

TABLE 1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
A-1	Volatile Organics	EPA Method TO14	2	Primary
			1	Field Duplicate
A-2	Volatile Organics	EPA Method TO14	2	Primary

NOTES:

1) One trip blank to be analyzed per day of sample shipment (3 Total).
2) Semi-volatile organic analysis will include 3,4-dichloronitrobenzene and diphenyl sulfone.
3) Mirex, photomirex, and kepone will be analyzed according to the Ruetgers-Nease Method as described in the approved RI Work Plan.
4) Conventional parameters include pH, specific conductance, TDS, TSS, TOC, COD, BOD, and ammonia. The respective methodologies are presented in Table 9.
5) NPDES parameters include total phenolics, total oil and grease, TUa ceriodaphnia, TUc ceriodaphnia, TUa pimephales promelas, and TUc pimephales promelas. The respective methods are presented in Table 9.
6) The SW846 Methodologies for metals analyses are as follows:
   Mercury - EPA SW846 Method 7470
   Arsenic - EPA SW846 Method 7060
   Lead - EPA SW846 Method 7421
   Selenium - EPA SW846 Method 7740
   Thallium - EPA SW846 Method 7841
   EPA SW846 Method 6010: Aluminum, Antimony, Barium, Beryllium, Calcium, Cadmium, Chromium, Cobalt, Copper, Iron, Magnesium, Manganese, Nickel, Potassium, Silver, Sodium, Vanadium, and Zinc.

TABLE 1b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

FIELD SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
Aqueous				
Field Samples	Volatile	SW846	TBD	Primary
	Organics	Method 8240	TBD	Field Duplicates
			TBD	Rinsate Blank
			TBD	Trip Blank (1)
			TBD	MS/MSD
	Semi-Volatile	SW846	TBD	Primary
			TBD	Rinsate Blank
	Organics (2)	Method 8270	TBD	Field Duplicate
			TBD	MS/MSD
	Pesticides	SW846	TBD	Primary
		Method 8080	TBD	Field Duplicate
			TBD	Rinsate Blank
	MPK	R-N Method (3)	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
	Filtered	SW846	TBD	Primary
	Metals	Methodologies(5)	TBD	Field Duplicate
			TBD	Rinsate Blank
	Unfiltered	SW846	TBD	Primary
	Metals	Methodologies(5)	TBD	Field Duplicate
			TBD	Rinsate Blank

TABLE 1b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

FIELD SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
	Conventional Parameters	(4)	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
Soils/Sediments Field Samples	Volatile Organics	SW846 Method 8240	TBD	Primary
			TBD	Field Duplicates
			TBD	Rinsate Blank
			TBD	Trip Blank (1)
	Semi-Volatile Organics (2)	SW846 Method 8270	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
	Pesticides	SW846 Method 8080	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
	MPK	R-N Method (3)	TBD	Primary
	Unfiltered Metals	SW846 Methodologies(5)	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
	Conventional Parameters	(4)	TBD	Primary
				Field Duplicate

TABLE 1b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

FIELD SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
Air Ambient Air Monitoring	Volatile Organics	Field Measurement	TBD	Primary

NOTES:

1) One trip blank to be analyzed per day of sample shipment (3 Total).
2) Semi-volatile organic analysis will include 3,4-dichloronitrobenzene and diphenyl sulfone.
3) Mirex, photomirex, and kepone will be analyzed according to the Ruetgers-Nease Method as described in the approved RI Work Plan.
4) Conventional parameters include pH, specific conductance, TDS, TSS, TOC, COD, BOD, and ammonia. The respective methodologies are presented in Table 9.
5) NPDES parameters include total phenolics, total oil and grease, TUa ceriodaphnia, TUc ceriodaphnia, TUa pimephales promelas, and TUc pimephales promelas. The respective methods are presented in Table 9.
   TBD - To be determined.
6) The SW846 Methodologies for metals analyses are as follows:
   Mercury - EPA SW846 Method 7470
   Arsenic - EPA SW846 Method 7060
   Lead - EPA SW846 Method 7421
   Selenium - EPA SW846 Method 7740
   Thallium - EPA SW846 Method 7841
   EPA SW846 Method 6010: Aluminum, Antinomy, Barium, Beryllium, Calcium, Cadmium, Chromium, Cobalt, Copper, Iron, Magnesium, Manganese, Nickel, Potassium, Silver, Sodium, Vanadium, and Zinc.

TABLE 2A
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE LOCATIONS AND LABORATORY ANALYSES

SAMPLE LOC.	DESCRIPTION	ESTIMATED COLLECTION TIME (MIN.)(10)	VOCS(1)			SVOCS(2)			PEST.(3)			MPK(4)			METALS(5)			CONV.(6)			NPDES(8)		
			Day			Day			Day			Day			Day			Day			Day		
			1	3	5	1	3	5	1	3	5	1	3	5	1	3	5	1	3	5	1	3	5
T-1	Surge Tank (Influent)	0	X	X (9)	X	X	X (9)	X	X	X (9)	X	X	X (9)	X	X	X (9)	X	X	X	X			
SP-1	Influent to Air Stripper	2	X		X										X	X	X	X(7)	X(7)	X(7)			
SP-2	Influent to Bag Filter 2	24	X		X										X	X	X						
SP-3	Influent to Liquid GAC 1	26													X	X	X						
SP-4	Influent to Liquid GAC 2	43				X		X	X		X	X		X	X	X	X						
SP-5	Effluent (Liquid)	60	X	X*	X	X	X*	X	X	X*	X	X	X*	X	X	X*	X	X	X	X			X
A-1	Influent to Vapor GAC 1		X		X(9)																		
A-2	Effluent from Vapor GAC 2		X		X																		

Notes:
(1) Volatile organics will be analyzed by SW846 Method 8240 (liquid-phase) and Method TO14 (gaseous-phase).
(2) Semi-volatile organics will be analyzed by SW846 Method 8270 and will include 3,4-Dichloronitrobenzene and Diphenyl Sulfone.
(3) Pesticides will be analyzed by SW846 Method 8080.
(4) Mirex, photomirex, and kepone will be analyzed by Ruetgers-Nease Method as described in the Approved RI Work Plan.
(5) TAL Metals (unfiltered and filtered) will be analyzed by appropriate SW846 Methodolgies.
(6) Conventional analytes are pH, specific conductance, TDS, TSS, TOC, COD, BOD, and ammonia.
(7) This sample will be analyzed for TSS only.
(8) NPDES parameters include: oil and grease (total); Phenols (total), TUa ceriodaphnia (acute toxicity); TUc ceriodaphnia (chronic toxicity); TUa pimephales promelas (acute toxicity); and TUc pimephales promelas (chronic toxicity).
(9) A field duplicate will be collected from the influent on Day 3 for all analyses.
(10) Samples will be collected following the estimated hydraulic retention times from the influent (T-1) to the point of sample locations.
(*) Sample location where sufficient samples will be collected for MS/MSD analyses.

TABLE 2B
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE LOCATIONS AND FIELD ANALYSIS (1)

SAMPLE LOC.	DESCRIPTION	IRON(2)	pH(3)	SPEC. COND.(3)	VOCs(4)
T-1	Surge Tank (Influent)	X	X	X	
SP-1	Influent to Air Stripper	X	X	X	
SP-2	Influent to Bag Filter 2	X	X	X	
SP-3	Influent to Liquid GAC 1	X	X	X	
SP-4	Influent to Liquid GAC 2	X	X	X	
SP-5	Effluent (Liquid)	X	X	X	
A-1	Influent to Vapor GAC 1				X
A-2	Effluent from Vapor GAC 2				X

NOTES:
(1) Samples will be collected from each location at least three times on each of the days during the performance evaluation.
(2) Iron will be analyzed by a Hach Kit (or equivalent).
(3) Specific conductance, temperature, and pH will be analyzed by an appropriate field instrument.
(4) Air-phase VOCs will be monitored by an OVA.

TABLE 3
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION AND ANALYTICAL HOLD TIMES FOR AQUEOUS AND GASEOUS SAMPLES

PARAMETER	METHODOLOGY	CONTAINER		MINIMUM SAMPLE	PRESERVATION (a)	HOLD TIME (b)
Volatile Organics (Aqueous)	SW846 Method 8240	(3)-40 ml	G	(3) - 40 ml (d)	Cool 4 deg C;HCl,pH<2	14 days (c)
Volatile Organics (Gaseous)	EPA Method TO14	(1) 5 l	Summa Can.	5 l	None	14 days (c)
Semi-Volatile Organics	SW846 Method 8270	(2)-1000 ml	Amber G	1000 ml (d)	Cool 4 deg C	7 days (e)
Pesticides	SW846 Method 8080	(2)-1000 ml	Amber G	1000 ml (d)	Cool 4 deg C	7 days (e)
MPK	R-N Method(g)	(2)-1000 ml	Amber G	1000 ml (d)	Cool 4 deg C	7 days (e)
Total Cyanide-TAL	SW846 Methodologies	(2)-1000 ml	P or G	1000 ml (d)	Cool 4 deg C;NaOH,pH>12	14 days
Total Metals-TAL	SW846 Methodologies	(1)-1000 ml	P or G	1000 ml (d)	Cool 4 deg C;HNO3,pH<2	180 days (f)
Ammonia	EPA 350.2	(1)-1000 ml	P or G	400 ml	Cool 4 deg C;H2SO4,pH<2	28 days
Total Organic Carbon	EPA 415.1	(1)-500 ml	P or G	250 ml	Cool 4 deg C;HCl,pH<2	28 days
Chemical Oxygen Demand	EPA 410.4	(1)-125 ml	P or G	50 ml	Cool 4 deg C;H2SO4,pH<2	28 days
Total Phenolics	EPA 420.1	(1)-1000 ml	G	1000 ml	Cool 4 deg C;H2SO4,pH<2	28 days
Biochemical Oxygen Demand	EPA 405.1	(2)-1000 ml	P or G	1000 ml	Cool 4 deg C	48 hours
Total Dissolved Solids	EPA 160.1	(1)-250 ml	P or G	100 ml	Cool 4 deg C	7 days
Total Suspended Solids	EPA 160.2	(1)-250 ml	P or G	100 ml	Cool 4 deg C	7 days
Total Oil and Grease	Gravimetric	(1)-1000 ml	G	1000 ml	Cool 4 deg C	7 days
TUa ceriodaphnia	Note h	5 gal.	P	5 gal.	Cool 4 deg C	7 days
TUc ceriodaphnia	Note i	5 gal.	P	5 gal.	Cool 4 deg C	7 days
TUa pimephales promelas	Note h	5 gal.	P	5 gal.	Cool 4 deg C	7 days
TUc pimephales promelas	Note j	5 gal.	P	5 gal.	Cool 4 deg C	7 days
Specific Conductance	Electrode		P or G	NA	None	Field Measurement
pH	Electrode		P or G	NA	None	Field Measurement
Temperature	Thermometer		G	NA	None	Field Measurement

TABLE 3
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION AND ANALYTICAL HOLD TIMES FOR AQUEOUS AND GASEOUS SAMPLES

NOTES:

(a) Sample Preservation is performed by sampler immediately upon sample collection. Dissolved metals samples will be filtered first and then preserved. Separate trial bottles will be used to assess the amount of preservative required to achieve pH<2 or pH>12.

(b) Hold time based upon day of sample collection not verified time of sample receipt.

(c) If preservation is not possible due to foaming, an unpreserved sample will be collected. The hold time will be 7 days for unpreserved sample. Preservation of VOCs in Water: Adjust the pH of the sample to less than 2 by carefully adding 1:1 hydrochloric acid (HCl) drop by drop to the required (3) 40 ml VOA sample vials. The number of drops of 1:1 HCl required should be determined on a fourth portion of sample of equal volume. Cool to 4°C. If acidification of the sample causes effervescence the samples must be submitted without preservation except for cooling to 4°C. This sample property should be appropriately noted when present, and the reduced holding times of seven days alerted to the laboratory.

(d) Triple sample volume is required for MS/MSD analysis of organics and metals at a rate of one per twenty field samples (excludes blank samples).

(e) 7 days for extraction, 40 days for analysis after commencement date of extraction.

(f) Hold time for Mercury is 28 days.

(g) Mirex, Photomirex, and Kepone will be analyzed by a Pulsed Positive Negative Chemical Ionization detection system.

(h) Acute Toxicity Test - 48-hour Static Non-Renewal Definitive Bioassay.

(i) Chronic Toxicity Test - 7-day Ceriodaphnia dubia survival and reproduction test.

(j) Chronic Toxicity Test - 7-day fathead minnow, Pimephales promelas, survival and growth test.

1. EPA "Methods for Chemical Analysis of Water and Waste", 1979, revised 3/83.
2. SM "Standard Methods for Water and Wastewater," 16th edition.
3. SW846 "Test Methods for Evaluating Solid Waste", November 1986.

ABBREVIATIONS

P = Polyethylene

G = Glass

TABLE 4a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE AND DATA QUALITY OBJECTIVE SUMMARY
TREATMENT PLANT EVALUATION

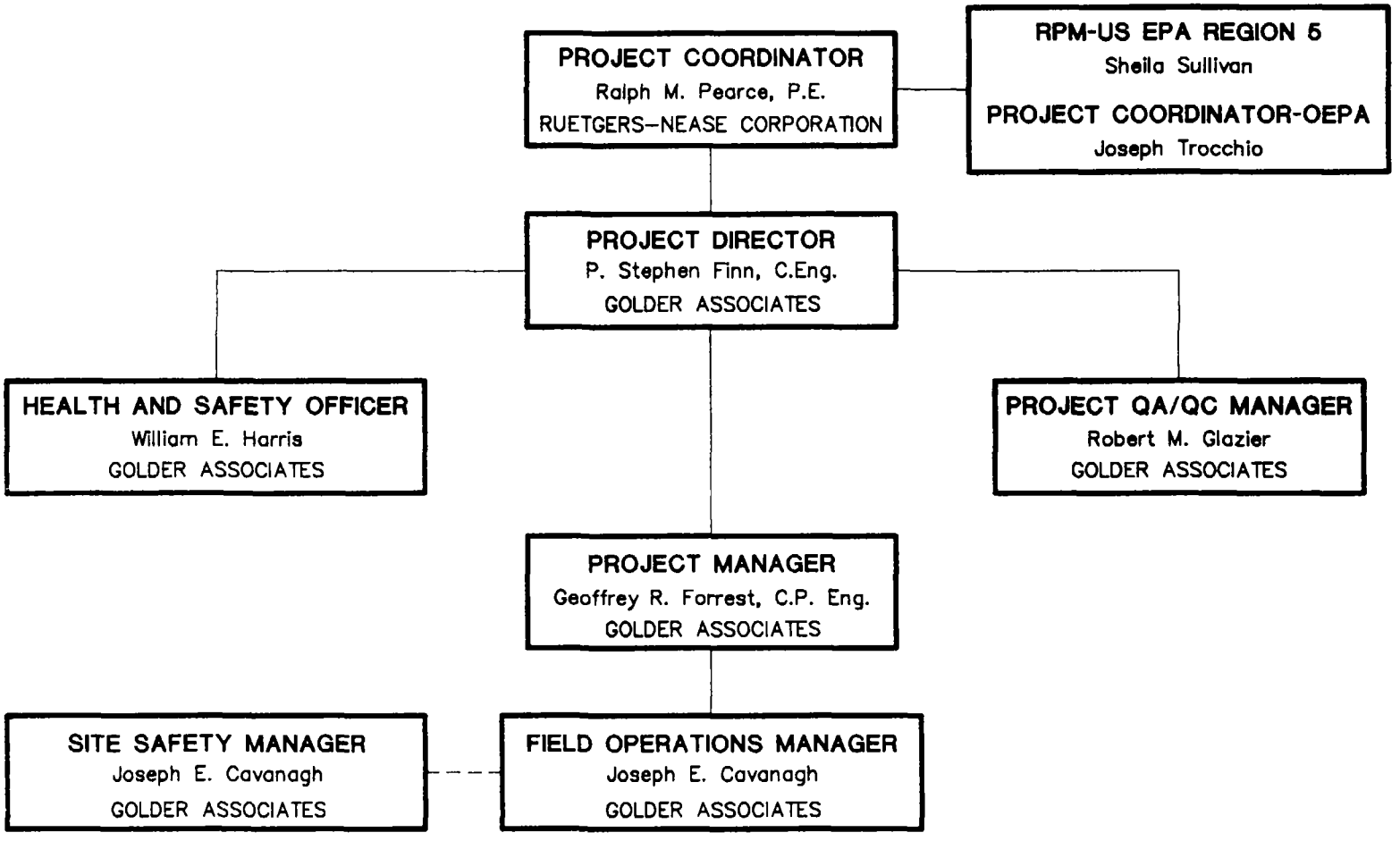
Media	Sample Point Designation	Number of Samples	Data Quality Level	Parameters of Analysis	Objective
Aqueous	T-1	3	III	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters	Establish influent constituent concentrations
		3	V	MPK	
	SP-1	2	III	VOCs	Assess removal efficiency of metals and solids by Bag Filter 1; also, establish VOC concentration entering the air stripper
		3	III	TSS and Metals	
	SP-2	2	III	VOCs	Assess removal efficiency of VOCs by the air stripper; assess metals precipitation potential
		3	III	Metals	
	SP-3	3	III	Metals	Assess removal efficiency of metals by Bag Filter 2
	SP-4	2	III	SVOCs and Pesticides	Assess the removal efficiency of organics and metals by Aqueous GAC 1
		3	III	Metals	
		2	V	MPK	
	SP-5	3	III	VOCs, SVOCs, Pesticides, Metals, and Conventional Parameters	Assess the overall effluent quality in terms of discharge limits
		3	V	MPK	
		1	III	NPDES Parameters	
Air	A-1	2	III	VOCs	Establish vapor phase VOC concentrations
	A-2	2	III	VOCs	Assess removal efficiency of the vapor phase carbon

TABLE 4b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE AND DATA QUALITY OBJECTIVE SUMMARY
FIELD SAMPLING

Media	Sample Point Designation	Number of Samples	Data Quality Level	Parameters of Analysis	Objective
Aqueous	On- and off-site surface water bodies	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	On- and off-site groundwater	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
Soils/Sediments	On- and off-site surface soils	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	On- and off-site subsurface soils	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	Pond materials	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	On- and off-site sediments	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
Air	On- and off-site air monitoring	TBD	I	VOCs	Establish contaminant concentrations for health and safety during sampling

TBD - To Be Determined



DEC 15 1993

JOB No.: 933-6158		SCALE: N/A	
DR BY: WME		DATE: 12/15/93	
CHK BY: THR 12/15/93		FILE No.: OH01-242	
REV BY: GRF 12/15/93		DR SUBTITLE: 01	

Golder Associates

REMOVAL ACTION WORK PLAN
SALEM, OHIO SITE
PROJECT ORGANIZATION CHART

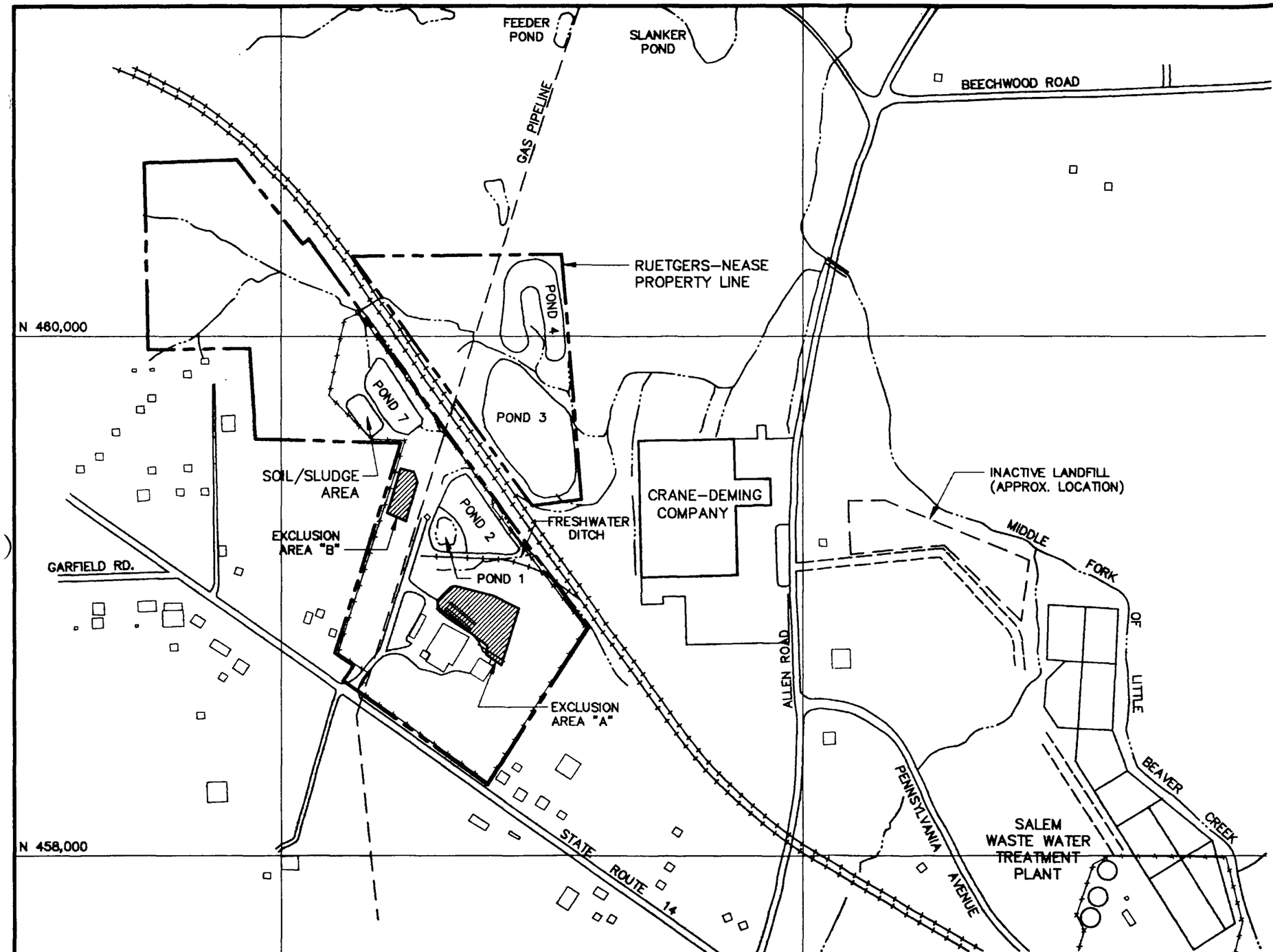
RUETGERS–NEASE CORPORATION

FIGURE 4

SECTION I - FIELD SAMPLING PLAN

APPENDIX A

SAMPLING AND FIELD TESTING PROCEDURES



REFERENCE

1.) BASE MAP TAKEN FROM ERM-MIDWEST DRAWING FILE No. 07FG2-14, PROJECT No. 202-01-07.

2.) EXCLUSION AREAS TAKEN FROM SMC MARTIN, INC. DRAWING TITLED "EXISTING AND PROPOSED MONITOR WELL LOCATIONS MAP", FIGURE 3, PROJECT No. 9061-94004, DATED JULY 1987.

DEC 15 1993

400 0 400
scale feet

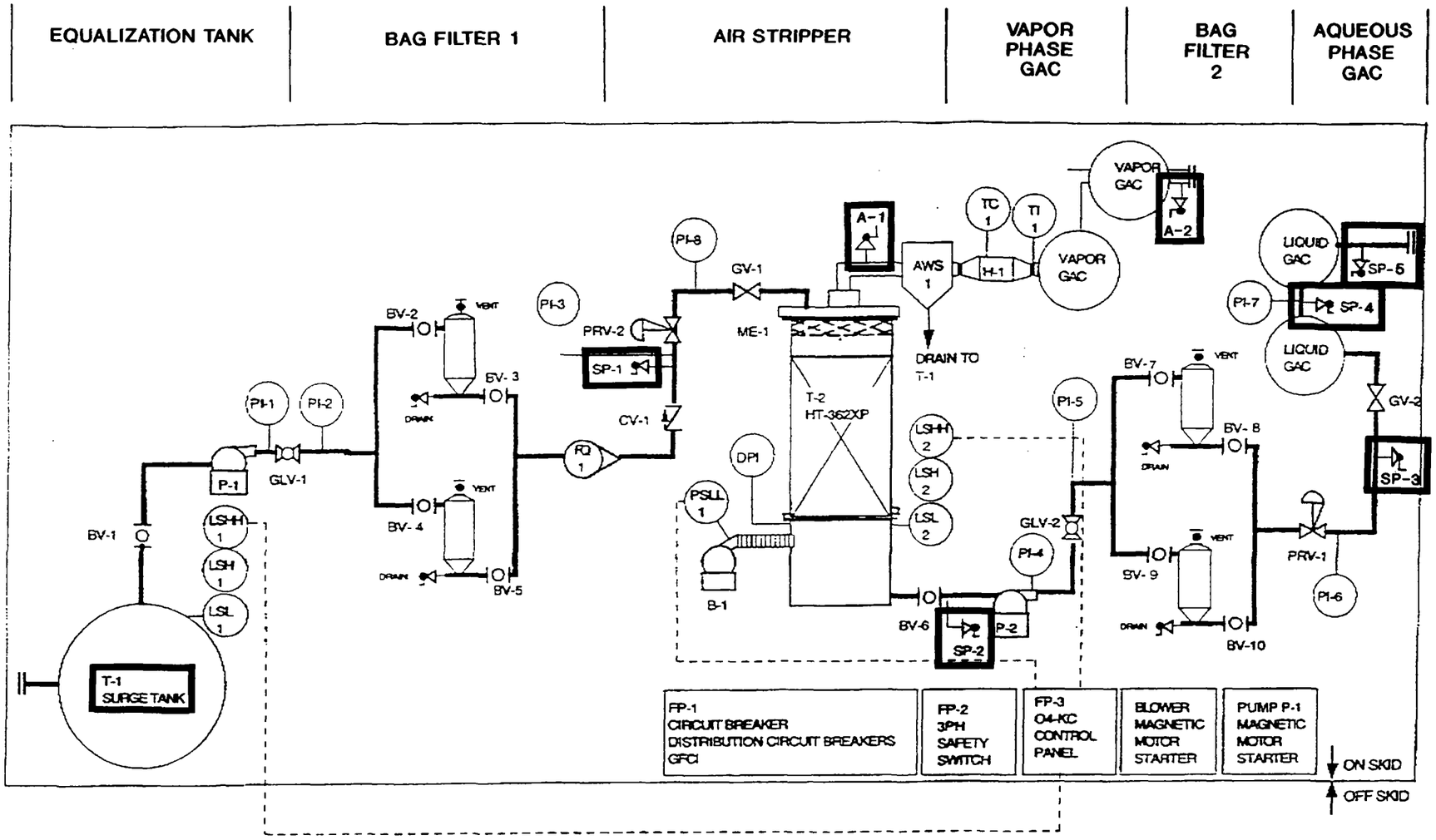
JOB No.:	933-8158	SCALE:	AS SHOWN
DR BY:	MRM	DATE:	10/01/93
CHK BY:	THR	FILE No.:	OH01-230
REV BY:	CRF	DR SUBTITLE:	01

Golder Associates

SITE LOCATION PLAN
SALEM OHIO SITE

RUETGERS-NEASE CORP.

FIGURE 1



LEGEND

SAMPLING LOCATION / PORT

SAMPLE	DESCRIPTION
T-1	SURGE TANK (INFLUENT)
SP-1	INFLUENT TO AIR STRIPPER
SP-2	INFLUENT TO BAG FILTER 2
SP-3	INFLUENT TO LIQUID GAC 1
SP-4	INFLUENT TO LIQUID GAC 2
SP-5	EFFLUENT
A-1	INFLUENT TO VAPOR GAC 1
A-2	EFFLUENT FROM VAPOR GAC 2

DEC 15 1993

JOB No.:	933-6158	SCALE:	N/A
DR BY:	EAM	DATE:	11/12/93
CHK BY:	THR	FILE No.:	OH01-220
REV BY:	GRF	DR SUBTITLE:	01

Golder Associates

SALEM IRM

TREATMENT PLANT SAMPLING LOCATIONS

RUETGERS-NEASE CORP.

FIGURE 2

1.0 INTRODUCTION

The performance evaluation objectives are to collect data of adequate technical content, quality, and quantity to complete a performance evaluation of the existing groundwater treatment plant which will be used to determine the ability of the treatment plant to meet the allowable discharge criteria.

The purpose of this FSP is to describe the sampling program rationale and procedures that will result in data of suitable quality and quantity to achieve the performance evaluation objectives.

To achieve these objectives efficiently, specific field procedures have been developed for the collection of samples from potentially affected media in the study area. These procedures are described in the following sections.

This Appendix contains the following sections that detail the field methods employed for in the Treatment Plant Performance Evaluation Work Plan:

1.0 Introduction
2.0 PID Photoionization Detector
3.0 Water Temperature
4.0 pH
5.0 Specific Conductance
6.0 Iron
7.0 Gas Samples

2.0 STANDARD OPERATING PROCEDURE FOR MEASURING VOCs IN AMBIENT WITH PHOTOIONIZATION DETECTOR, PID

1. PURPOSE

The purpose of this technical procedure is to establish methodology for the calibration, operation in both the survey and gas chromatograph (GC) modes, and routine maintenance of the Foxboro Century Systems Portable Organic Vapor Analyzer (hereafter referred to as the OVA).

2. APPLICABILITY

This procedure applies to all personnel engaged in the use of the Foxboro OVA. The OVA will typically find applications in hazardous site investigations and air monitoring for health and safety. In the survey mode, the OVA serves to detect total volatile organic vapors and gases in the low parts per million (volume/volume) concentrations. In the GC mode, the OVA can be used to tentatively identify and quantify specific volatile organic compounds in air (ppm range) and indirectly in soil and water (ppb range). In health and safety applications, even though one may not know exactly what is there, the OVA may be especially useful in the GC mode for establishing that certain compounds with very low allowable exposure limits, such as benzene, are not present.

3. DEFINITIONS

3.1 Organic Vapor Analyzer (OVA)

The OVA is a highly sensitive electronic instrument designed to measure trace quantities of organic materials in air by means of a flame ionization detector.

3.2 Gas Chromatograph (GC) Mode

Operating the OVA in the gas chromatographic or "GC" mode (as opposed to the direct read-out or "survey" mode) whereby a 250 μl volume of sample air is injected onto a GC column on the OVA with hydrogen as a carrier gas.

3.3 Calibration Gas

Methane or specific gas(es) of interest in air at known, certified concentrations are used for calibrating the OVA.

4. REFERENCES

4.1 Century Systems, 1985, "Operating and Service Manual for Century Systems: Portable Organic Vapor Analyzer (OVA) Model OVA-128 and Optional Accessories," Century Systems Corporation, Arkansas City, Kansas.

4.2 Linear Instruments Corporation, "Operating Instructions 141/142 Recorders," Linear Instruments Corporation, Stockton, California.

4.3 Golder Associates' Procedure P-12.0-1, "Calibration and Maintenance of Measuring and Test Equipment."

5. DISCUSSION

The Century Systems Portable Organic Vapor Analyzer (OVA) is designed to detect and measure a wide spectrum of volatile organic gases and vapors, but is most effective and finds the widest applications in the detection of petroleum hydrocarbons.

The instrument utilizes the principle of flame ionization for detection and measurement of organic gases and vapors. Figure 1 illustrates the gas handling system of the OVA.

In the survey mode, a continuous flow of sample air is drawn into the probe and conveyed to the detection chamber by an internal diaphragm pump. The sample flow rate is metered and passed through particle filters before reaching the detector chamber. Inside the detection chamber, the sample is passed through a hydrogen flame.

The carbon atoms which are present if the sample air is contaminated with organic hydrocarbons are ionized in the flame and subsequently collected by highly charged collector electrodes in the chamber. An electric field exists which drives the ions to the collecting electrodes. As the ions are collected, a current proportional to the ion flow rate is generated through the "circuit." The instrument "readout" is actually the voltage (greatly amplified) associated with the current (ion) flow measured across a resistor in the circuit.

6. RESPONSIBILITIES

6.1 Project Manager

The Project Manager shall be responsible for the following:

- Identifies situations where an OVA may be appropriate (project applications) and/or necessary (health and safety applications).

- Specifies the methodology, frequency of use, location of use, and measurement criteria of the OVA;
- Verifies appropriate calibrations, surveys, and maintenance activities of the OVA.

6.2 Task Leader

The Task Leader shall be responsible for the following:

- Assigns personnel to calibrate, operate, and maintain the OVA in the field;
- Ensures that the OVA is calibrated, used, and maintained correctly in the field and that these activities are documented;
- Ensures that the OVA is functioning properly at all times; and
- Informs the Equipment Manager when the OVA is not functioning properly.

6.3 Field Technicians

Field Technicians shall be responsible for the following:

- Being competent in the calibration, operation, and maintenance of the OVA;
- Documenting all calibrations, survey results, and maintenance activities; and
- Informing the Project Managers or Task Leaders when the OVA is not functioning properly.

6.4 Equipment Manager

The Equipment Manager shall be responsible for the following:

- Performs all OVA check out and check in procedures on equipment before it goes to the field and when it returns, respectively;
- Follows factory recommended calibration and maintenance schedules of the OVA;

- Maintains a file of periodic routine OVA calibration, operation, and maintenance information in accordance with procedure P-12.0-1; and
- Ensures that the OVA is properly maintained and stored when not in the field.

6.5 Health and Safety Officer

The Health and Safety Officer shall have the following responsibilities:

- Oversite of in-house personnel who calibrate, operate, and maintain the OVA;
- Training personnel on the use of the OVA;
- Reviewing the proposed use and measurement criteria of the OVA in the Health and Safety Plan.
- Reviewing calibration records and results of health and safety surveys; and
- Evaluating monitoring results and, when appropriate, adjusting Health and Safety Plan to reflect new information.

7. EQUIPMENT

- Portable Organic Vapor Analyzer (OVA) Model-128 Side Pack Assembly including the operating controls and indicators, the electronic circuitry, detector chamber, hydrogen gas supply, gas chromatographic column and electrical power supply.
- Probe/Readout Assembly
- Replacement sintered metal particulate filters
- Instrument carrying case 1
- A.C. battery charger
- Calibration gas (methane) or other specific gases
- regulator(s) for compressed gas bottles
- Air Monitoring Data Sheet (Exhibit A)
- Slotted screwdrivers (large & jewelers sizes)

- Phillips screwdriver
- 12 and 6 inch Crescent wrenches
- OVA Allen wrench kit
- Spare ignition element
- Single channel strip chart recorder (for use in GC mode), such as the Linear Model 142 or equivalent, spare chart paper and pens, power cord and patch cord. 1
- Tedlar bags
- Thermometer for use in the GC mode especially if temperature is expected to be variable.

8. PROCEDURES

8.1 Safety Precaution

The OVA Model 128 has been tested and certified by Factory Mutual Research Corporation as intrinsically safe for use in Class I, Division 1, Groups A, B, C & D hazardous atmospheres. However, the OVA uses hydrogen as a fuel source. Mixtures of hydrogen and air are flammable over a wide range of concentrations. Precautions should be taken during hydrogen filling or tank emptying to ensure there are no sources of ignition in the immediate area.

A general understanding of the operation of the OVA and the fact that it indicates an "integrated average" of concentrations of total volatile organic gases present is necessary for its use as a monitoring device. If other conditions suggest that the air may be unsafe for breathing (such as unusual odors, visible contamination, etc.), appropriate measures must be taken to protect the individuals in the field regardless of the instrument reading.

8.2 Equipment Check Out and Check In

The following activities are the responsibility of the Equipment Manager for check out and check in of the OVA to and from Golder Associates Inc. personnel:

- Periodic "Bench Top" calibration (Section 8.3)
- Checking air sampling line probe tip and side pack assembly filters (Section 8.6.3.4 and 5) 1

- Cleaning exhaust flame arrestor (Section 8.6.6)
- Seal maintenance (Section 8.6.7)
- Adjusting refiller valve (Section 8.6.8)
- Refueling H_2 tank (Section 8.6.1)
- Battery charging (Section 8.6.2)

8.3 Calibration

The OVA is capable of responding to a greater or lesser extent, to nearly all organic compounds in a gaseous or vapor state. However, the response may vary considerably depending on the compound. For precise analysis it is necessary to calibrate the instrument directly to the specific compound of interest.

The instrument is factory calibrated to a methane-in-air standard. However, it can be easily and rapidly calibrated to a variety of organic compounds. A GAS SELECT (span) control is incorporated on the instrument panel which may be set to give a direct reading for a particular organic compound.

Internal "span pots" (potentiometers) are provided to calibrate and align the three sensitivity ranges. There are four adjustments located on the internal electronics board. One adjustment potentiometer, R-38, is used to set the power supply voltage and should not be changed. The remaining three adjustments, R-31, R-32, and R-33 are used to set the electronic amplifier gain (span) for the x1, x10, and x100 sensitivity scales, respectively.

8.3.1 Field Calibration to Methane

At a minimum, the calibration of the OVA shall be checked and documented against a 9 ppm methane-in-air standard at the beginning and end of each day of use.

Gain Adjustment

1. After a sufficient warm-up period (5 - 10 minutes), place instrument in normal operational mode (inject switch "up") with CALIBRATE Switch set to x1 and GAS SELECT control set to 3.0.

2. Use the CALIBRATE ADJUST (zero) Knob and adjust the meter reading to zero.

3. Introduce methane calibration gas (9 ppm methane-in-air) from a tedlar bag and adjust the Gas Select Knob until the instrument reads 9 ppm. Document this span setting. Switch to the x10 scale and verify that the instrument read-out is approximately (0.70-1.1) ".9" (.9 x 10 = 9 ppm). If the instrument is used to quantify higher concentrations of organic vapors (i.e., x10 or x100 scale) the instrument calibration should be checked in each of these ranges as well using 90 ppm and 900 ppm methane-in-air, respectively as discussed in item 4. below.

4. Verify that the instrument reads between 80 and 100 ppm in the x10 range and between 800 and 1000 ppm in the x100 range in response to 90 ppm and 900 ppm methane, respectively. If the instrument readings are outside of this acceptable range in the x10 and x100 scales, adjust and record the span (Gas Select) settings required to obtain readings of exactly 90 ppm and 900 ppm, respectively. Return the instrument to the x1 scale and span setting identified in step 3 above. The Gas Select (span) setting should be adjusted to the appropriate value (determined during calibration in the x10 and x100 scales as discussed above) whenever quantitative readings are required in the x10 or x100 sensitivity range.

8.3.2 "Bench" Calibration to Methane

The OVA shall be subject to the controls specified in procedure P-12.0-1, "Calibration and Maintenance of Measuring and Test Equipment." Whenever there is any indication that the instrument is not within acceptable calibration limits (i.e., readings outside of 8-10 ppm, 80-100 ppm, and 800-1000 ppm in response to 9, 90, and 900 ppm methane, respectively, with "Gas Select" set at 3.0), but at least once every three months, the OVA should be bench calibrated in all three ranges. Calibration required by procedure P-12.0-1 shall be performed as follows:

1. Disconnect the air sample line and umbilical cord, and remove the ignitor button guard. Remove the OVA from its case by loosening the 4 quarter turn mounting screws in the faceplate and gently pulling the body of the instrument out and up.

2. Re-connect the air sampling line and umbilical cord and turn instrument on.

3. After a sufficient warm-up period, set the Gas Select to 3.0, and set the "Adjust" knob such that the instrument reads "zero" in clean air in the x1 scale. Change the scale setting to x100.

4. Introduce 900 ppm methane-in-air to the instrument from a Tedlar bag. Do not introduce gas directly into the probe from a cylinder or regulator. Adjust the internal span pot "R33" in the ceramic circuit board until the instrument reads exactly 900 ppm (a reading of "9" on the read-out probe x100) with the gas select setting on 3.0.

5. Disconnect the 900 ppm methane and introduce 90 ppm methane while the instrument is still in the x100 scale. Verify that the instrument reads between 80 and 100 ppm (.8 to 1 on the read-out probe) in the x100 scale. Switch to the x10 scale and adjust the interval span pot R32 until the instrument reads exactly 90 ppm in the x10 scale (a reading of "9" on the read-out dial x10).

6. Disconnect the 90 ppm methane and introduce 9 ppm menthane while the instrument is still in the x10 scale. Verify that it reads between 8 and 10 ppm (.8 to 1 on the read-out dial). Switch to the x1 scale. Adjust the interval span pot R31 until the instrument reads exactly 9 ppm in the x1 scale.

7. Verify that the Gas Select is set to 3.0. Verify that the instrument is reading 9 ppm in the x1 scale in response to 9 ppm methane. Switch to the x10 scale and verify that the instrument reads approximately .9 on the read-out dial.

8. Reintroduce 90 ppm methane and verify that the instrument reads 90 ppm (9 on the read-out dial) in the x10 scale. Readjust span pot R32 if necessary. Switch to the x100 scale and verify that the instrument reads approximately .9 on the read-out dial.

9. Reintroduce 900 ppm methane and verify that the instrument reads 900 ppm in the x100 scale. Adjust span pot R33 if necessary.

10. Replace instrument in case. Replace ignition button guard, and reconnect air sampling line and umbilical cord. 1

    Document the calibration and indicate the date of last calibration on the instrument case in accordance with procedure P-12.0-1.

8.3.3 Calibration of Other Organic Gases

1. Perform the field calibration check for methane.

2. Draw a sample of the desired calibration gas into the instrument.

3. Adjust the GAS SELECT Knob on the panel to shift the readout meter indication to correspond to the actual concentration of the calibration gas mixture.

4. The instrument is now calibrated to the organic gas being used. Document this "Gas Select" setting. Whenever the "Gas Select" is set to this value, the instrument will give direct readings for this gas.

8.4 *Operating Procedure*

8.4.1 Initial Preparation For Use

1. Connect the appropriate probe tip to the Readout Assembly with the captive locking nut. The short, non-telescoping probe tip is recommended for most applications. The telescoping probe is appropriate for broad, sweeping area wide surveys. Ensure that the probe is seated firmly in the Readout Assembly.

2. Connect the air sample hose to the Side Pack Assembly. The nut should be tightened by hand and then secured with at most an eighth to a quarter turn using a small crescent wrench. DO NOT OVER TIGHTEN. Connect umbilical cord. 1

3. Hydrogen fueling should be accomplished using the Foxboro high pressure filling hose. See Section 8.6.1 below.

4. Move INSTR/BATT Test Switch to the BATT position and verify that the battery is charged as indicated on the readout meter.

8.4.2 Turn On Procedure

1. Move the INSTR SWITCH to ON and allow several minutes for warm up.

2. Move the CALIBRATE Switch to x1 and adjust the meter reading to or slightly above zero with the CALIBRATE ADJUST (zero) .

3. Turn the PUMP Switch to ON and observe the SAMPLE FLOW RATE Indicator. Flow rate should be approximately 2 units.

4. Open H2 TANK VALVE several turns and observe the reading on the H2 TANK PRESSURE Indicator. The tank pressure should be at or above 1800 psi for a full day of operation. Note that the instrument will continue to operate down to approximately 200 psi of hydrogen if it is not turned off, but will become increasingly difficult to start (relight) below 500 psi.

5. Open H2 SUPPLY VALVE 1/2 to 1 turn and observe the reading on the H2 SUPPLY PRESSURE indicator. Depending on the GC column in use, this reading should be between 9 and 13 psi, longer columns will require higher H_2 supply pressures.

6. Confirm that the meter is still reading approximately zero.

7. Depress ignition button and hold. The readout needle should "peg" and there will be a slight "pop" as the hydrogen ignites. Immediately after ignition, release the ignition button. If burner does not ignite, let instrument run for several seconds and try again. Allow several minutes for the instrument to stabilize after ignition. If necessary, re-zero the instrument with the CALIBRATE ADJUST (zero) Knob. Do not hold ignition button down for more than five seconds.

8. Move instrument to an area that is representative of clean air. Move the CALIBRATE Switch to x1 and adjust the meter to read 0-1 ppm with the CALIBRATE ADJUST (zero) Knob.

9. Use of the alarm is not recommended. However, if the alarm level is to be used, set the read out to the desired alarm level using the zero adjust knob. Be sure the alarm volume control is turned up to an audible level and adjust the alarm level adjustment knob on the back of the read-out assembly until the alarm just begins to sound to the level indicated on the read-out assembly. Rezero the read-out assembly using the zero adjust knob.

10. The instrument is now ready for calibration check as described in Section 8.3, and use as described below.

8.4.3 Operating Procedure in the Survey Mode

1. Set the CALIBRATE Switch to the desired range. Using one hand operation, survey the areas of interest while observing the meter. For ease of operation, carry the Side Pack Assembly positioned on the side opposite the hand which holds the Probe/Readout Assembly. Always keep the cover on the instrument when using it for field measurements in the survey mode. Once the instrument is properly zeroed and calibrated the operator will require access to the controls only occasionally to correct zero drift or to change sensitivity scales in the event of off-scale readings. This can be easily accomplished even with the cover on the instrument once the user becomes familiar with the location of the cover latches and controls. For broad survey outdoors, the pickup fixture should be positioned several feet above ground level. When making quantitative readings or pinpointing areas of contamination, the pickup fixture should be positioned at the point of interest (but not actually in the mud, waste, water, etc.).

2. When organic gases are detected, the meter pointer will move upscale.

3. If the nature of the organic vapors being detected in unknown, nothing can be said about the exact concentration or nature of the contaminants other than that the meter reads "X ppm" relative to methane (if the instrument is, in fact, calibrated to methane). If, on the other hand, the identity of the compound being monitored is known, it can be quantified by calibrating the instrument directly to that compound as discussed in 8.3.3 above, or the actual concentration may be estimated by calibrating the instrument to methane and incorporating the relative response factors (relative to methane) identified in Table 1. For example, Table 1 indicates that the response of trichloroethylene relative to methane is 70%. If the OVA is calibrated to methane and reads 70 ppm in an atmosphere known to be contaminated with trichoroethylene, the 70 ppm reading is only 70% of the actual concentration of trichloroethylene. The actual concentration is approximately 100 ppm. 1

4. If the readout drifts to a negative (below zero) value, use the adjust knob to bring the reading back to zero and verify that the instrument is still "lit" by very briefly holding a permanent felt tip marker in front of the probe. Introduce the marker and pull it away. Do not hold it to the probe until you observe a response. There will be a slight "lag time." If the pen is held to the probe tip until a response is observed it may "load up" (contaminate) the instrument plumbing. Flame-out may result from sampling a gas mixture that is above the lower explosive level. If this is the case, it will be necessary to relight the instrument.

8.4.4 Operating Procedure in the GC Mode

8.4.4.1 General Description

The OVA with the available GC option has two modes of operation. With the Sample Inject Valve in the "out" position the instrument is measuring total organic vapors in the survey mode as described in Section 8.4.3. The OVA is in the GC mode of operation when a sample is injected into the GC system and transported through the GC column by depressing the "Sample Inject" Valve. In the GC mode the OVA can be utilized for analysis of specific organic vapors, but will not give a continuous direct read-out of sample air. A strip chart recorder should be used when operating in the GC mode as described in Section 8.4.4.6 below. The Instruction and Service Manual for the OVA-128 (Century Systems 1985) should be reviewed for a more detailed description of the general operating principles and theory for operation in the GC mode. Troubleshooting tips are provided in Table 2 of this procedure.

8.4.4.2 Sample Flow

The flow paths of the hydrogen fuel, sample air, and GC injected sample aliquot through the OVA in the two modes of operation are shown in Figure 2. It is well worth the user's time to carefully study and understand Figure 2. By doing so the user will gain a much clearer understanding of how the instrument works and much greater insight into potential causes and solutions of problems commonly encountered in the field. In the GC mode the hydrogen acts as a carrier gas. The recommended hydrogen flow rate is 12 cc/min. for proper flame ionization detector (FID) operation and as a standard flow rate for generating GC reference/calibration data. Restrictions to hydrogen flow will vary with the length and type of packing of the GC column. The hydrogen flow rate is factory set at 12 cc/min. with a Century standard 24 inch long column, and normally no adjustment needs to be made when using columns from three (3) inches to four (4) feet long. Adjustment may be required if and when precisely controlled analysis is being conducted or when the hydrogen flow is too low to keep the flame burning. The hydrogen flow rate is adjusted by varying the hydrogen Supply Pressure. The pressure is changed by adjusting the set screw in the bonnet of the low pressure regulator, accessible by removing the battery pack from the instrument panel. The sample air flow in the OVA is not adjustable and is nominally 1.0 liter/min. This flow rate should remain relatively constant.

8.4.4.3 Column Selection

You should have at least two different columns available when using the OVA in the GC mode. Appropriate columns should be determined prior to going into the field by the Project Manager by referring to the chromatographic column selection guide provided with the Century OVA and in Table 1. This table should be referred to when selecting suitable columns and to determine approximate retention times for a variety of volatile organic compounds. In the interest of time and ideal peak configurations, columns should be selected such that the compounds of interest will elute ("come out") within a range of 15 seconds to 3-4 minutes. It is possible that a single sample may contain two or more compounds of interest which have vastly different retention times on a particular column (i.e., fluorotrichloromethane and 1,1,2,2 tetrachloroethane on a T-8 column). In this case it may be desirable to select two columns, one of which is most appropriate for each compound. It is also possible that two or more compounds may have nearly identical retention times on a given column and be difficult or impossible to distinguish on one column. If there is any question of the identity of compounds of interest that are detected, the identification should be verified by comparison of retention times to calibration standards using two different columns.

8.4.4.4 Connecting the Strip Chart Recorder

A single channel strip chart recorder (Linear Model 142 or equivalent) should be used with the OVA when operating in the GC mode. When using a recorder other than the manufacturers, any patch cord with a two pin connector can be used. Connect the patch cord to the B and D pins on the OVA and to the red (+) and black (-) terminals of the recorder. If positive deflections on the read-out probe result in negative deflection of the strip chart recorder, simply switch the leads to the red and black terminals. For normal full-scale readings, the "input voltage" of the strip-chart recorder should be in the "1 volt" range. Operation of the recorder with the OVA in the GC mode is discussed in Section 8.4.4.6 below.

8.4.4.5 GC Mode Operation

In normal GC analysis, a strip chart recorder is used to record the output from the OVA as a function of time. This record (chromatogram) is utilized for interpretation of the GC data. All GC data is evaluated by comparing chromatograms of environmental samples to chromatograms of calibration standards of known concentration. The OVA should always be calibrated (as described in Step 4) prior to analysis of environmental samples, and the strip chart recorder should be properly connected and "calibrated" to the OVA.

Operation

1) Begin with the OVA running in the normal survey mode. Turn the strip chart recorder on (as described in Section 8.4.4.6) with the pen in the down position. Make sure that the chart paper drive is "ON." Two (2) cm/min is an appropriate chart speed for most compounds of interest. Prior to introducing the sample air, the following information must be entered in a separate field notebook dedicated to the GC analytical procedures for the particular project.

   Information to be entered immediately prior to each GC run:

   - GC run ID number consistent with run number recorded on strip chart print out.
   - Date and time of run.
   - Origin of sample.
   - Strip chart paper speed.
   - Value on OVA read-out probe corresponding to full-scale deflection on strip chart paper (i.e., 1, 5, 10 or whatever is appropriate and convenient).

- Initial OVA Scale Setting (x1, x10, x100) and any changes to this setting as the run progresses.

- All observations, comments, non-routine occurrences (i.e., flame-outs) which may be of interest in interpreting results later.

Introduce the sample air, either from the ambient air, a tedlar sample bag, headspace technique, etc. If detectable organics are present, the read-out probe and the strip chart recorder will give a positive "total organics" deflection in the survey mode. Push the Sample Inject Valve "in" with a fast, positive motion. This injects a 250 microliter "slug" of contaminated sample air onto the GC column and starts the GC analysis. Immediately upon pushing the sample inject button, the total organics reading on the strip chart recorder should drop to zero. This drop on the print-out is indicative of "T_0" or the GC start time. If the read-out does not drop to zero or perhaps even increases, the activated carbon in the charcoal filter is probably contaminated and must be replaced. Note: On occasion, the flame in the FID detector may go out, which is difficult to distinguish from normal operation except that the "zero" reading may be "pinned" below zero at the bottom of the chart. If you have reason to suspect a flame-out condition, immediately reignite the flame in the normal manner (provided that you have not "missed" any potential compounds of interest during the flame-out condition), and continue the analysis. A negative "air" peak typically occurs shortly after sample injection and should not be confused as flame-out.

2) The negative air peak and various positive compound peaks will be indicated by deflections on the OVA readout meter and by corresponding peaks on the strip chart recorder. Compounds which "come out" in a few seconds will appear as narrow, sharp spikes. Compounds which come out in 30 seconds to 1 or 2 minutes will be very defined "peaks." As the retention time increases, the "peaks" become less defined and begin to resemble ill defined "hills", which is one reason that it is desirable to select a column that is appropriate for the specific compounds of interest. Peak resolution of "slow eluters" may be enhanced by using a slower chart speed.

3) After the predetermined time for analysis has elapsed (normally immediately after the peak of the last compound of concern), rapidly move the Backflush Valve to it's alternate position (in or out). Leave the instrument in this condition until the backflush peak printed on the recorder returns to baseline. If no backflush peak appears, leave the GC in the backflush condition for a period of time <u>at least</u> as long as the GC analysis time, preferably slightly longer. Pull the Sample Inject Valve to the "out" position (the OVA will not operate in the survey mode or be ready for another sample injection in the GC mode unless the Sample Inject Valve has been pulled to the "out" position). Immediately upon pulling the sample inject button to the "out" position there should be a sharp spike (jump) on the strip chart recorder. At this point record "End of Good Run [INSERT NUMBER]" on the chart. If the spike is not observed when the inject button is pulled up, it is indicative of a flame-out at

To obtain a calibration point, inject a known concentration sample into the GC system and record the resulting chromatogram peak. The peak height response should be linear up to concentrations of greater than 1,000 ppm. Therefore, a single calibration point, preferably around the concentration of most concern, is normally all that is required. If temperature variations are expected standards should be repeated throughout the day.

8.4.4.6 Operating the Strip Chart Recorder

This discussion covers general set up and operation of a Linear Model 142 (or equivalent) single channel strip chart recorder. The manufacturers operating instructions should be reviewed for general theory, field calibration procedures and tips for operation and maintenance. Troubleshooting tips are provided in Table 3.

8.4.4.6.1 Set Up Procedures

The recorder should be connected to the OVA as discussed in Section 8.4.4.4 above. The procedures, detailed below must be performed to prepare the recorder for operation.

Fuse Requirements

Check unit for proper fuse (0.15A) located on back panel of the recorder.

Chart Paper Installation

1. Pull release lever on front left side of the recorder forward to pull out chart drawer for loading.

2. Insert metal tube inside new roll of chart paper. Metal insert provides proper weight/tension to supply roll as it is expended.

3. Drop new chart roll through (directly behind paper feed sprockets) with round sprocket holes on the left side. The supply chart paper roll sits on the base of the tray, directly behind the faceplate.

4. Remove tape and pull out about 12 inches of paper. Pull paper up, over sprockets, and down across the front of the face plate.

5. Push end of chart paper through slot in bottom of face plate from front to back.

6. Push spare core on take-up post. Pull chart paper under the spare core. Roll once around take-up core and tape end of paper to core.

7. Turn core to take up slack in chart.

some point in the GC run and the entire run may have to be rejected. A sample GC run print-out indicating all appropriate information is shown in Figure 4.

Calibration in the GC Mode

4) The OVA in the GC mode is intended for applications where there are a limited number of compounds of interest and the "universe" of compounds which may be expected is well characterized. For example, an ideal application of the OVA/GC would be to identify specific contaminants on a remediation site where considerable data regarding the known contaminants already exists. In other situations, it may be desirable to screen a sample for one or two "key" contaminants. For example, at a site contaminated with petroleum hydrocarbons it may be necessary to determine the presence (or absence) and concentration of benzene to establish an appropriate level of respiratory protection. Under these types of field conditions the OVA should be calibrated in the GC mode, by testing the retention times and peak area (using peak height analysis) characteristics for the specific compound(s) of interest. Calibration should be conducted using the same column(s) to be utilized and over the concentration of concern and similar temperature conditions as will be encountered during sample analysis.

When calibrating (i.e., running "standards" through) the GC the following information should be recorded on the chromatogram and/or on a calibration chart (Figure 3):

a) Column - description and serial number as applicable

b) Temperature - column temperature, normally room or ambient

c) Chart Speed - distance/unit time

d) Sample concentration - ppm for each compound

e) Recorder Scaling - ppm per unit of deflection

f) Range - range of OVA, i.e., x1, x10, x100

g) OVA type and serial number

h) Date and sample inject time

i) Backflush time

8. Close drawer by pushing on center of chart. DO NOT push release lever.

9. Turn on chart drive at 10 cm/min for several seconds to take up slack.

Pen Installation

1. Remove the pen cap and store it where it can be easily found. Pens can be left uncapped for long periods of time, but greater pen life results from capping when not in use.

2. Lift marker scale (on front of recorder) by pushing up on center of scale; the scale should pivot 90° and raise the pen carriage.

3. Insert pen in carriage until it stops.

4. Push lever on right side of scale plate down to drop the pen onto the chart, and return scale to normal position.

Power Requirements

The Model 142 is designed to operate from a self-contained, rechargeable, 12 VDC, "sealed" lead acid battery. The battery will be charged when operating from 115 or 230 VAC, 50/60/400 Hz, 1 0. Be sure to set the conversion slide switch located on the back panel to match the correct line voltage. Note that the unit will run from AC power or the battery pack but actually always runs off of the batteries. The AC power only serves to charge the batteries continuously during use. The batteries must be sufficiently charged to use the instrument even off of external AC power.

8.4.4.6.2 Initial Recording/Calibration

With chart paper and pen installed and recorder plugged into proper AC source (or in DC battery operation) perform the following steps for routine recording:

1. Turn power switch on and chart switch off. On Model 142 the power light is on when the battery is charging and off when the battery is charged.

2. Set input scale to desired range (normally 1 volt) and attenuator to Cal position.

3. "Zero" readout on OVA with "Adjust" knob. 1

4. Push zero toggle switch on strip chart recorder.

5. Zero pen to left side of chart using zero control knob while holding down zero toggle switch.

6. Release zero toggle switch and apply input signal by increasing the OVA read-out with the "adjust" knob.

7. Adjust the attenuator knob on strip chart recorder until strip chart readout corresponds to OVA probe readout. Select a full scale deflection value on the strip chart recorder which will best display the anticipated OVA readout levels, i.e., if you don't expect any readings greater than 5 ppm, set up the strip chart such that full chart deflection corresponds to 5 ppm on the OVA. 1

8. Document the OVA readout value (i.e., 1, 5, 10) which corresponds to full chart deflection on the strip chart recorder.

9. Turn chart speed on and set chart drive for desired speed (normally 2 cm/min).

8.4.4.7 Interpretation of Results

Interpretation of the recorded chromatogram is always based on the predetermined calibration data discussed in Section 8.4.4.5. The compound(s) detected in the sample chromatogram may be tentatively identified by comparing the retention times of the observed peak(s) with those of the calibration standards. The compounds can be quantified based on the height of the peak in relation to the peak heights of the calibration standards of known concentration. Caution should be exercised in interpreting results. GC results cannot be considered positive identification with complete certainty. However, since the OVA is normally used in the GC mode when some of the compounds of interest are known, the results can be considered fairly reliable when standards of those compounds are used for calibration. 1

8.4.5 Shut Down Procedure

1. Close H_2 SUPPLY VALVE.

2. Close H_2 TANK VALVE.

3. Move INSTR Switch to OFF.

4. Wait 5 seconds and move PUMP Switch to OFF. INSTRUMENT IS NOW IN A SHUTDOWN CONFIGURATION.

8.5 Troubleshooting

The following activities are the responsibility of the Task Leader and Field Technicians to ensure that the OVA is functioning properly:

- Calibration procedure
- Filter replacements

Tables 2 shows instrument malfunctions that may occur in the field and the corresponding corrective action or remedy. Refer also to Section 8.6 below.

8.6 Maintenance Procedures

The following components of the OVA require maintenance. Maintenance personnel should be thoroughly familiar with instrument operation before performing maintenance.

8.6.1 Fuel Refilling

1. The instrument should be completely shut down during hydrogen tank refilling operations. The refilling should be done in a ventilated area. There should be no potential ignition sources or flame in the area.

2. The filling hose should be purged with clean H_2 prior to filling the instrument tank.

3. Connect the hose to the refill connection on the Side Pack Assembly. 1

4. Open the hydrogen supply bottle valve slightly. Open the REFILL VALVE and the H_2 TANK VALVE on the instrument panel and place the FILL/BLEED Valve on the filling hose assembly in the FILL position. The pressure in the instrument tank will now be indicated on the H_2 TANK PRESSURE indicator.

5. After the instrument fuel tank is filled, shut off the REFILL VALVE on the panel, the FILL/BLEED Valve on the filling hose assembly and the hydrogen supply bottle valve.

6. The hydrogen trapped in the hose should now be bled off to atmospheric pressure. CAUTION should be used in this operation as described in Step (7) below, since the hose will contain a significant amount of hydrogen at high pressure.

7. The hose is bled by turning the FILL/BLEED Valve on the filling hose assembly to the BLEED position. After the hose is bled down to atmospheric pressure, the FILL/BLEED Valve should be turned to the FILL position to allow the hydrogen trapped in the connection fittings to go into the hose assembly. Then, again, turn the

FILL/BLEED Valve to the BLEED position and exhaust the trapped hydrogen. Then turn the FILL/BLEED Valve to OFF to keep the hydrogen at one atmosphere in the hose so that at the time of the next filling there will be no air trapped in the filling line.

8. Close the H_2 Tank Valve.

9. With the H_2 TANK VALVE and the H_2 SUPPLY VALVE closed, a small amount of H_2 at high pressure will be present in the regulators and plumbing. As a leak check, observe the H_2 TANK PRESSURE indicator while the remainder of the system is shut down and ensure that the pressure indication does not go down rapidly, indicating a significant leak. If it does decrease rapidly (greater than 350 PSIG/hr.), there is a significant leak in the H_2 supply system.

8.6.2 Battery Recharging

1. Plug charger BNC connector into mating connector on battery cover and insert AC plug into 115 VAC wall outlet. Never charge in a hazardous area or environment.

2. Move the battery charger switch to the ON position. The light above the switch button should illuminate.

3. Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the right when charging. When fully charged, the pointer will be in line with "charged" marker above the scale.

4. Approximately one hour of charging time is required for each hour of operation. However, an overnight charge is recommended. The charger can be left on indefinitely without damaging the batteries. When finished, move the battery charger switch to OFF and disconnect the Side Pack Assembly.

8.6.3 Primary Filter

This filter is located behind the sample inlet connector on the Side Pack Assembly and is removed for cleaning by using a small adjustable wrench, or a thin wall socket to unscrew the Fitting Assembly. The porous stainless filter cup can be cleaned by blowing out or washing in a solvent. If a solvent is used, care should be taken to ensure that all solvent is removed by blowing out or heating the filter.

8.6.4 Particle Filter

A particle filter is located in each pickup fixture. The filter should be changed or cleaned, depending upon the OVA model number.

8.6.5 Mixer/Burner Assembly Filter

Another porous metal particle filter is incorporated in the Mixer/Burner Assembly which screws into the Preamp Assembly. This filter should not become contaminated under normal conditions but can be cleaned or the assembly replaced if necessary.

8.6.6 Exhaust Flame Arrestor

A porous metal flame arrestor is located in the exhaust port of the detector chamber. This filter may be cleaned, if required, by removing the exhaust port from the Preamp Assembly. Clean the filter with a solvent or detergent but ensure that it is dry and any solvent is completely baked out at 120° F before installation.

8.6.7 Seal Maintenance

After some time, the teflon washers under each valve packing nut can "cold flow" and allow hydrogen to leak. This leakage can usually be stopped by tightening the compression nut (adapter). This compression nut is against soft material and only a small amount of force is necessary to sufficiently compress the teflon washers. If after tightening, leakage still occurs, it is advisable to replace the two teflon washers. The replacement procedure is outlined in the Users Manual for the OVA.

8.6.8 Refiller Valve Packing Adjustment

Adjustment for the valve on the refiller can be made by loosening the set screws with a 3/32" hex key, so that the handle turns freely on the stem. Insert two 3/32" hex keys through the holes provided in the handle and turn until they engage the holes in the packing adjuster. Then tighten the packing by turning the handle.

8.6.9 Air Sampling System Maintenance

A potential problem associated with the OVA is that leaks can develop in the air sample pumping system. The OVA is equipped with a flow gauge, which provides a method to check for air leaks. To isolate a leak, remove parts of the air-sampling system, one at a time, and recheck for leaks.

8.6.10 Contamination Control and Maintenance

If instrument readings fluctuate widely either the GC column or the hydrogen fuel is contaminated. Try a replacement column or try using a heating pad to heat the column while the instrument is running. It may take an hour or longer but many times contamination on the column can be flushed out. If it is determined that the column is not the problem, the hydrogen fuel tank in the instrument is probably contaminated. Empty the hydrogen fuel tank through the refill valve. Purge the hydrogen refill hose with (hopefully) clean hydrogen from the source and flush out (i.e., refill and drain) the H_2 tank several times. Refill the tank. If the problem still exists either your fuel source is contaminated, the instrument is contaminated internally, or both. The OVA should be returned to the equipment manager and/or the manufacturer for maintenance.

8.6.11 Pump Stops Running

The pump may periodically "hang up" and stop running. It is located in the lower right hand corner of the instrument immediately below the rotameter (air flow meter). Unscrew the lower right hand case fastening screw and gently pry up the corner of the face plate. With the electronics and pump switches "on" insert a flat dull utensil (popsicle stick is ideal) into the case and give the pump a "push." It should start immediately.

8.6.12 Does Not Return to Zero When GC Inject is Pushed

If the readout fails to go to zero or increases upon hitting the GC inject button, the carbon filter bed is probably contaminated. Remove the filter tube from the instrument and pull out (DO NOT TWIST OR USE PLIERS) the end cap. Discard the old charcoal. Rinse the tube with acetone and distilled water and allow it to dry completely. It can be dried in an oven at 120°-140°F. Fill with fresh activated carbon (fish tank filter carbon will do or in a pinch open up a fresh organic vapor respirator cartridge) and replace filter on instrument.

8.6.13 Factory Maintenance

To ensure continuous trouble-free operation, Century recommends a periodic factory maintenance overhaul and recalibration every six to nine months.

9. RECORDS

Calibration of the OVA and air monitoring results are documented using the Air Monitoring Data Sheet (Exhibit A). In addition, when used in the GC mode a calibration chart (Figure 3) should be prepared and chromatograms kept of all calibration and environmental samples as described in Section 8.4.4.5.

10.0 PROCEDURE ALTERATION CHECKLIST

Variation from established procedure requirements may be necessary due to unique circumstances encountered on individual projects. All variations from established procedures shall be documented on Procedure Alteration Checklists (Exhibit B) and reviewed by the Project Manger and the QA Manager.

The Project Manager may authorize individual Task Leaders to initiate variations as necessary. If practical, the request for variation shall be reviewed by the Project Manager and the QA Manager prior to implementation. If prior review is not possible, the variation may be implemented immediately at the direction of the Task Leader, provided that the Project Manager is notified of the variation within 24 hours of implementation, and the Procedure Alteration Checklist is forwarded to the Project Manger and QA Manager for review within 2 working days of implementation. If the variation is unacceptable to either reviewer, the activity shall be reperformed or action shall be taken as indicated in the Comments section of the Checklist.

All completed Procedure Alteration Checklists shall be maintained in project records.

FIGURE 1

GAS HANDLING SYSTEM

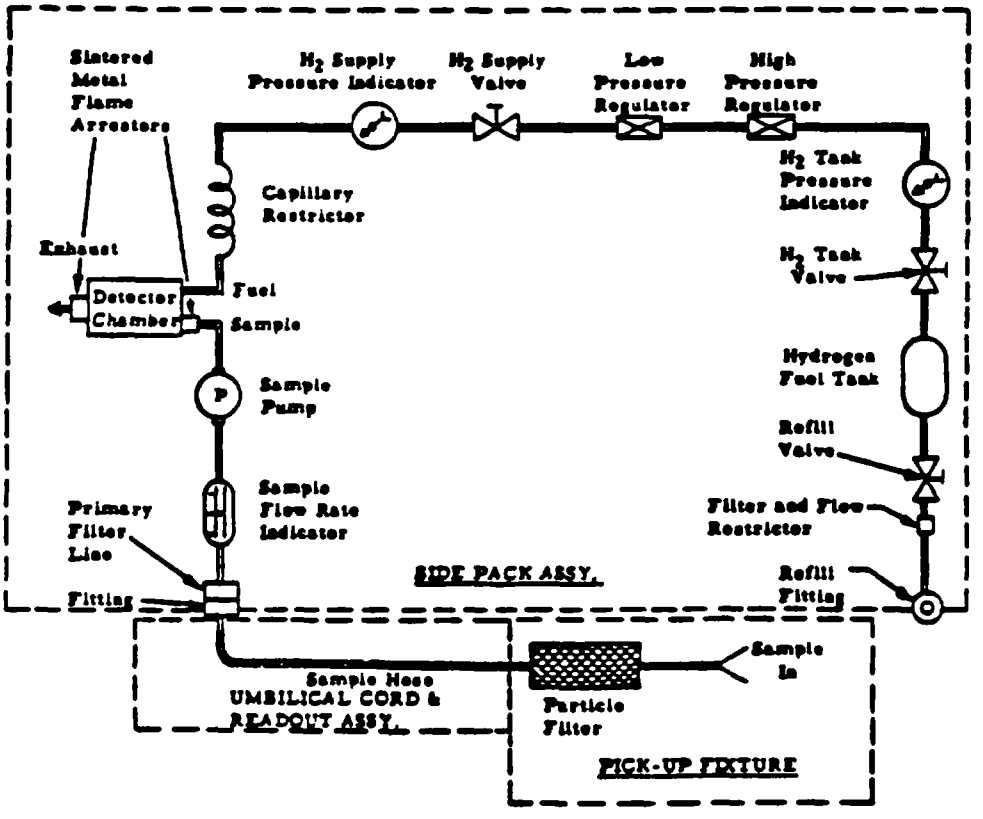


FIGURE 2

SAMPLE AND GAS FLOW SURVEY AND GC MODE

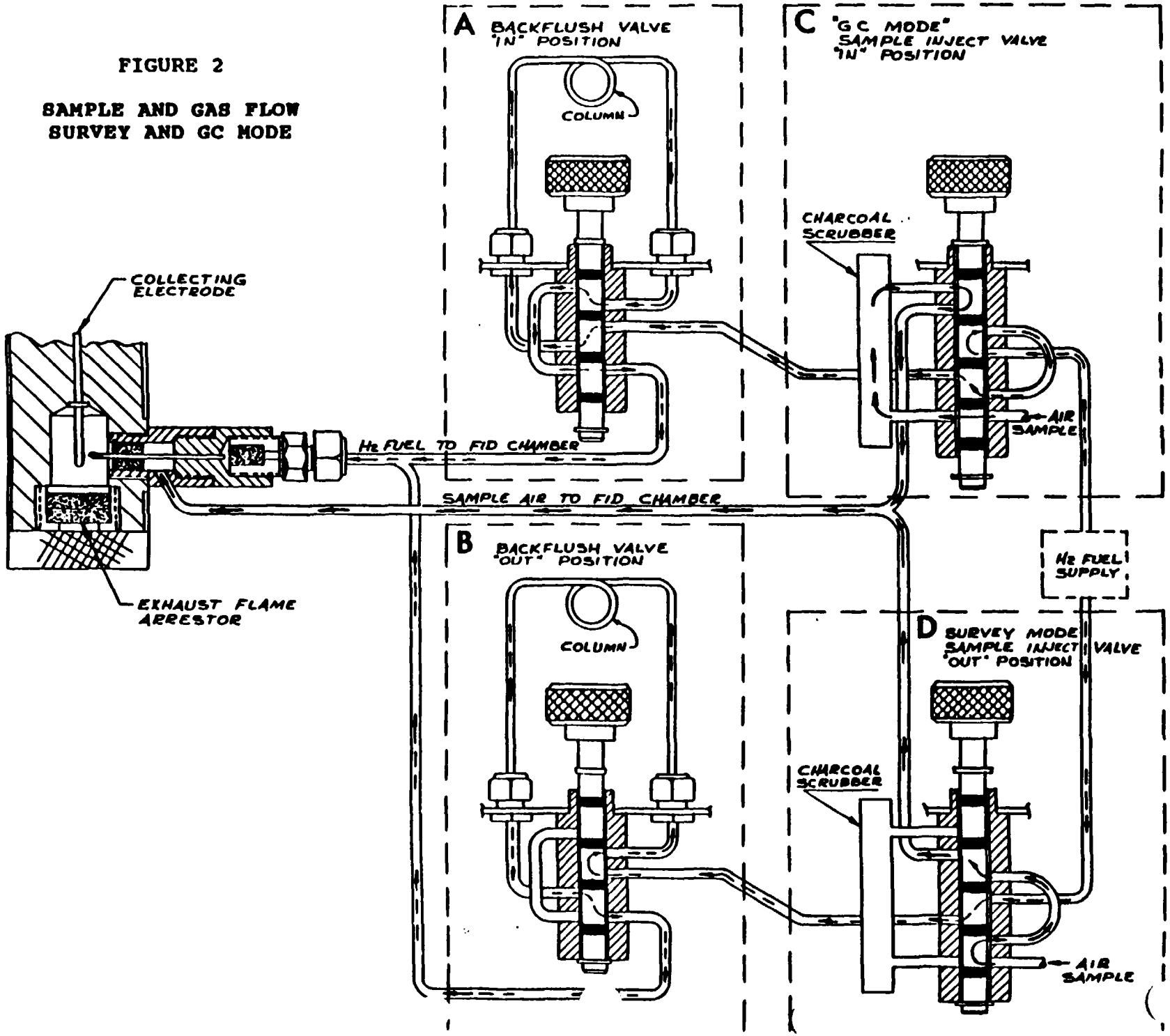
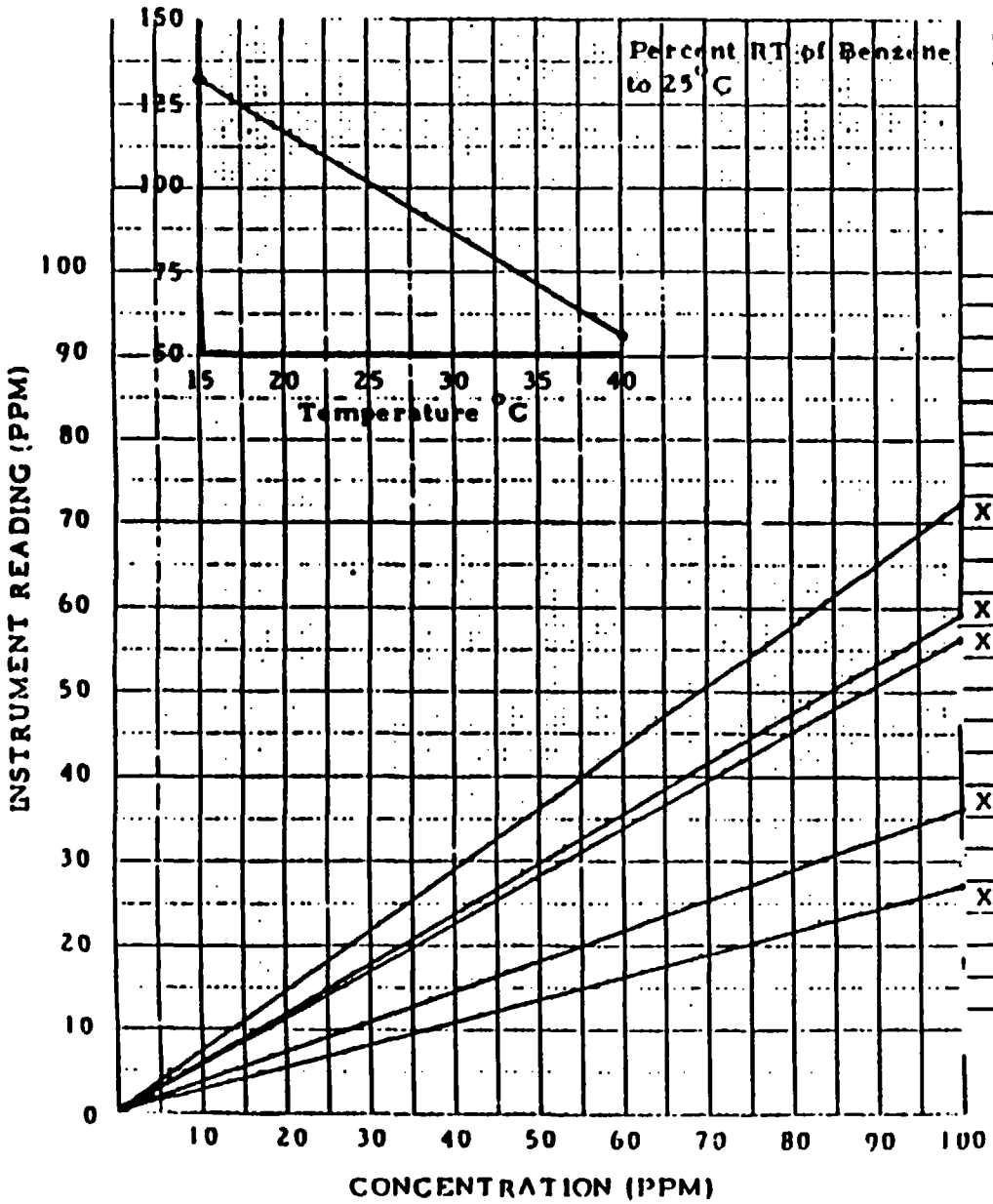


FIGURE 3 EXAMPLE CALIBRATION CHART



Column G-48 Chromatograph OVA-118
Temperature 25 °C Carrier Flow 12 ml/sec.
Injection: Volume 2.5 ml Type valve

X	Compound	R. R. (%)	RT (sec)	Date	TLV (ppm)
X4	Heptane	80	471	1-31-78	400
X2	Pentane	100	90	1-31-78	1000
X2	Hexane	90	198	1-31-78	100
X2	Benzene	150	291	1-31-78	1
X3	Toluene	120	762	1-31-78	200

NOTE: Concentration used for test is 100 ppm or as indicated by marked data points.

COMMENTS:

FIGURE 4 SAMPLE GC RUN STRIP CHART

TP-2.3-2

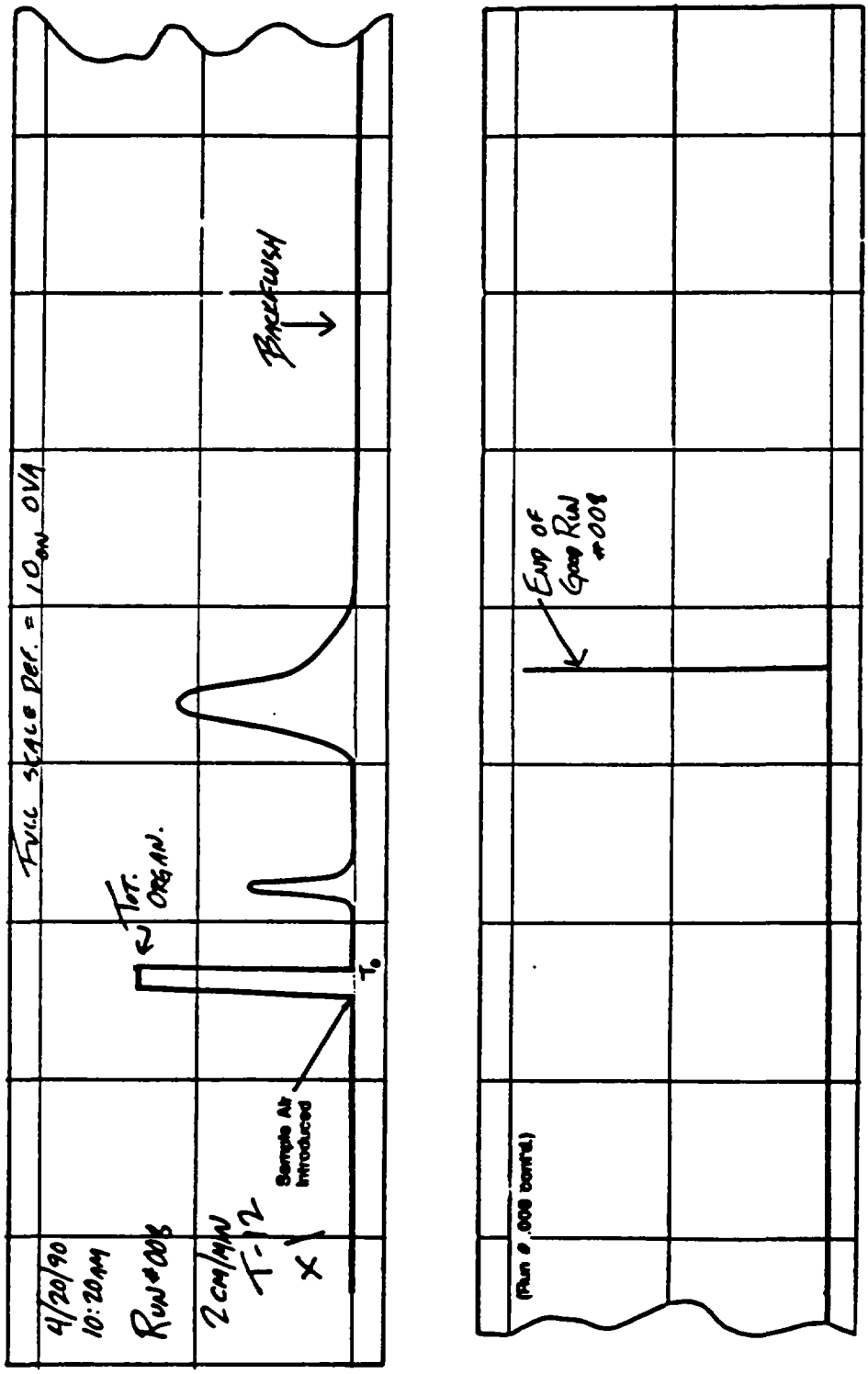


EXHIBIT A

TP-2.3-2

Golder Associates Inc.

AIR MONITORING DATA SHEET

Date _____ Sheet ____ of ____

Job Name _____ Job Number _____

Location _____

Time In _____ Time Out _____ Weather _____ Temp. _____ Wind D. ____ V _____

Instrument Type _____ Serial No. _____

Calibration Gas and Concentration _____ Instrument Reading _____ Span/Gain/RF Setting _____

** If more than one instrument is used, document calibration procedures and results for each additional instrument in recommendations section below and indicate the instrument used (eg. OVA, 361, OVM, etc.) for each observation.*

Time	Station	Instr.*	Reading	Procedure/Observations/Comments

Recommendations

_____

_____

_____

_____

_____

Printed Name _____ Signature _____

PROCEDURE ALTERATION CHECKLIST

Job/Task Number: _____

Procedure Reference: _____

Requested Variation: _____

Reason for Variation: _____

Special Equipment, Material or Personnel Required: _____

Alteration Requested By: _____ Date: _____

Title: _____

Reviewed By: _____ Date: _____

Title: GAI Project Manager

Comments: _____

Reviewed By: _____ Date: _____

Title: GAI QA Manager

Comments: _____

TABLE 1

CHROMATOGRAPHIC COLUMN SELECTION GUIDE

COMPOUND	Maximum Allowable Exposure (8 Hour Weighted Average) PPM	Relative Response (%)	Column Temperature (°C)	Column Packing (Retention Time in Minutes) 8.8	C.8	7.8
Acetone	1000	60	0	0 37	0 27	1 50
			40	0 20	0 15	0 30
Acetonitrile	40	70	0	0 40	0 30	5 24
			40	0 20	0 18	1 15
Acrylonitrile	2	70	0	0 35	0 30	3 45
			40	0 19	0 17	0 51
Allyl Alcohol	2	30	0	1:15	0.44	11 45
			40	0.37	0 26	1 35
Allyl Chloride	1	50	0	0 16	0.28	0 31
			40	0:08	0 16	0 15
Benzene	10	150	0	0:50	1 19	1 43
			40	0.22	0.25	0 32
2-Bromo-2-chloro-1,1,1-trifluoroethane (Halothane)	—	45	0	0.37	0.35	0 58
			40	0:17	0 14	0 19
Bromoethane	200	75	0	0.15	0 26	0 23
			40	0 05	0 15	0 14
1-Bromopropane	—	75	0	0 21	0 58	0 40
			40	0 08	0 22	0 19
2-Butane	—	60	0	0.15	0 15	0 10
			40	—	—	—
n-Butanol	100	50	0	4 10	2 20	15 07
			40	0 24	0 53	1 44
2-Butanol	150	65	0	1.45	0:55	6 07
			40	0.24	0:24	0 53
n-Butyl Acetate	150	80	0	4:15	7:30	11 40
			40	0:50	1:14	1 31
n-Butyl Acrylate	—	60	0	—	—	—
			40	2:30	2:15	2 40
2-Butyl Acrylate	—	70	0	—	—	—
			40	1:22	1:45	1:46
n-Butyl Formate	—	50	0	2:15	2:57	4:40
			40	0:28	0:34	0 49
2-Butyl Formate	—	60	0	1:22	2:00	3 31
			40	0.20	0.26	0 37
n-Butyl Methacrylate	—	60	0	—	—	—
			40	4:03	5:46	5.10
2-Butyl Methacrylate	—	80	0	—	—	—
			40	2 46	3.40	3 34
Carbon Tetrachloride	10	10	0	0:20	1:24	0 37
			40	0:10	0.25	0 17
Chlorobenzene	75	200	0	5:45	8:00	11.20
			40	1:08	1:24	1.35
Chlorodifluoromethane (Freon 22)	1000	40	0	0:11	0:11	0 15
			40	—	—	—
Chloroform	50	85	0	0:55	0:57	2.00
			40	0:20	0:20	0 31
1-Chloropropane	—	75	0	0.16	0:31	0.23
			40	0:05	0:16	0 14
2-Chloropropane	—	90	0	0 15	0:23	0 18
			40	0:05	0.05	0 05
2-Chloro-1,1,2-trifluoroethyl difluoromethyl ether (Ethrane)	—	150	0	0 36	0.26	1 22
			40	0.13	0.12	0 19
Cumene	50	100	0	11:00	20:00	12.45
			40	2.20	3:03	2.19
Cyclohexane	300	85	0	0 36	1 25	0 19
			40	0 18	0 26	0 14
Cyclohexanone	50	100	0	18 00	12:45	—
			40	3:00	2 00	—
n-Decane	—	75	0	—	—	—
			40	2.57	6 20	1 35
O-Dichlorobenzene	50	50	0	—	—	—
			40	8 06	10.00	11 20
Dichlorodifluoromethane (Freon 12)	1000	15	0	0 10	0:11	0 12
			40	—	—	—
1,1 Dichloroethane	100	80	0	0 17	0.37	0 45
			40	0.08	0.17	0 18

TABLE 1

CHROMATOGRAPHIC COLUMN SELECTION GUIDE

COMPOUND	Maximum Allowable Exposure (8 Hour Weighted Average) PPM	Relative Response (%)	Column Temperature (°C)	Column Packing (Retention Time in Minutes) B-S	G-S	T-S
1,2 Dichloroethane	50	80	0 40	1:14 0:23	1:08 0.22	3:50 0.43
trans 1,2-Dichloroethylene	200	50	0 40	0:16 0:05	0.35 0:18	0.31 0.16
Dichlorofluoromethane (Freon 21)	1000	70	0 40	0.16 —	0:15 —	0.23 —
Dichloromethane	500	100	0 40	0:27 0.10	0:29 0.10	1:08 0.22
1,2-Dichloropropane	75	90	0 40	0:41 0:18	1:49 0:29	2.56 0.36
1,3-Dichloropropane	—	80	0 40	1:32 0.26	4:12 0.47	4.24 1.20
1,2-Dichloro 1,1,2,2-tetrafluoro-ethane (Freon 114)	1000	110	0 40	0.12 —	0.12 —	0.14 —
Diethyl Ether	400	50	0 40	0.20 0:05	0.26 0.05	0.15 0.05
Diethyl Ketone	—	80	0 40	2:00 0.29	2.01 0.30	3.16 0.49
p-Dioxane	100	30	0 40	3.15 0.44	2.09 0.34	6.40 1.19
Ethane	—	80	0 40	0.15 —	0.15 —	0.15 —
Ethanethiol	—	30	0 40	0.18 0:13	0.24 0:12	0.25 0:14
Ethanol	1000	25	0 40	0:59 0:26	0:31 0:22	0.26 0.43
Ethyl Acetate	400	65	0 40	0.48 0:20	1:00 0.20	2.20 0:31
Ethyl Acrylate	25	40	0 40	1:40 0:30	2:10 0.30	4:08 0:50
Ethyl Benzene	100	100	0 40	6:10 1:15	9.31 1:35	7:44 1.35
Ethyl Butyrate	—	70	0 40	4:34 0:48	6.22 1:07	8.00 1.24
Ethyl Formate	100	40	0 40	0.25 0:16	0.29 0:17	1:05 0.20
Ethyl Methacrylate	—	70	0 40	4:13 0:47	6.13 1:01	6.20 1.01
Ethyl Propionate	—	65	0 40	1:40 0:25	2:48 0.35	4.10 0.50
Ethylene Dibromide	20	50	0 40	5:20 1:00	4:51 0:55	15:00 2:43
Ethylene Dichloride	50	60	0 40	1:07 0:20	1:08 0:21	3:45 0:50
Ethylene Oxide	50	70	0 40	0:10 —	0.13 —	0:20 —
Fluorotrichloromethane (Freon 11)	1000	10	0 40	0:15 —	0:18 —	0:17 —
Heptane	500	75	0 40	0:50 0:20	2:20 0:30	0:27 0.16
Hexane	500	70	0 40	0:23 0:13	0:50 0:20	0:20 0:13
Isoprene	—	50	0 40	0:10 0:05	0:23 0:05	0:20 0:05
Methane	—	100	0 40	0:05 —	0:05 —	0:05 —
Methyl Alcohol	200	12	0 40	0.37 0:22	0:21 0.14	2.23 0.45
Methyl Acetate	200	41	0 40	0:30 0:17	0.30 0.15	1.30 0.24
Methyl Acrylate	10	40	0 40	0:50 0:20	0.58 0.21	2.30 0:37
Methyl Cyclohexane	500	100	0 40	0:54 0:18	2.37 0.33	0:25 0.17
Methyl Cyclopentane	—	80	0 40	0:22 0:05	1:02 0.19	0.17 0.05

TABLE 1

CHROMATOGRAPHIC COLUMN SELECTION GUIDE

COMPOUND	Maximum Allowable Exposure (8 Hour Weighted Average) PPM	Relative Response (%)	Column Temperature (°C)	Column Packing (Retention Time in Minutes) 8.0	6.0	1.0
Methyl Ethyl Ketone	200	80	0	1.00	0.50	3.30
			40	0.22	0.20	0.43
Methyl Isobutyl Ketone	100	80	0	4.20	3.15	7.30
			40	0.42	0.40	1.25
Methyl Methacrylate	100	50	0	1.41	2.22	4.08
			40	0.27	0.31	0.55
Methyl Propyl Ketone	200	70	0	2.20	2.05	6.14
			40	0.33	0.32	0.52
Nitromethane	100	35	0	0.51	0.40	3.00
			40	0.25	0.19	1.31
1-Nitropropane	25	60	0	4.50	2.50	25.00
			40	0.46	0.41	4.05
2-Nitropropane	25	70	0	2.53	1.51	10.00
			40	0.35	0.31	1.52
Nonane	--	90	0	7.26	8.00	2.32
			40	1.08	2.40	0.42
Octane	500	80	0	2.47	7.39	1.07
			40	0.39	1.09	0.27
Pentane	1000	65	0	0.18	0.25	0.14
			40	0.12	0.12	0.12
Pentanol	–	40	0	12.00	6.00	20.00
			40	2.44	1.17	3.36
Propane	1000	80	0	0.05	0.11	0.05
			40	–	–	–
n-Propanol	200	40	0	2.50	1.00	6.30
			40	0.35	0.30	1.05
2-Propanol	400	65	0	1.13	0.30	3.43
			40	0.25	0.20	0.38
n-Propyl Acetate	200	75	0	2.04	2.52	5.52
			40	0.30	0.38	0.51
n-Propyl Ether	–	65	0	1.37	2.06	1.15
			40	0.43	0.29	0.18
n-Propyl Formate	–	50	0	0.46	1.07	2.10
			40	0.17	0.18	0.27
Pyridine	5	128	0	8.00	4.15	–
			40	–	1.28	–
Styrene	100	85	0	20.00	25.00	28.00
			40	2.06	2.26	2.39
1,1,1,2-Tetrachloroethane	–	100	0	8.25	7.48	10.00
			40	1.16	1.27	1.37
1,1,2,2-Tetrachloroethane	5	100	0	32.00	14.00	50.00
			40	4.09	2.37	7.51
Tetrachloroethylene	100	70	0	3.00	5.45	2.10
			40	0.41	1.06	0.33
Tetrahydrofuran	200	40	0	1.05	1.05	1.45
			40	0.23	0.23	0.30
Toluene	200	110	0	2.30	4.05	4.30
			40	0.38	0.47	0.53
1,1,1 Trichloroethane	350	105	0	0.31	1.10	0.47
			40	0.15	0.23	0.20
1,1,2 Trichloroethane	10	85	0	5.13	3.43	15.00
			40	0.40	0.45	2.30
Trichloroethylene	100	70	0	1.17	2.02	1.25
			40	0.23	0.28	0.28
Trichlorotrifluoroethane (Freon 113)	1000	80	0	0.15	0.30	0.15
			40	0.13	0.14	0.13
Triethylamine	25	70	0	–	2.49	–
			40	–	–	–
Vinyl Acetate	–	50	0	0.34	0.43	1.55
			40	0.15	0.17	0.30
Vinylidene Chloride	–	40	0	0.20	0.25	0.22
			40	0.13	0.14	0.14
m-Xylene	100	111	0	2.39	12.03	8.31
			40	0.39	1.42	1.17
o-Xylene	100	116	0	3.29	15.07	8.40
			40	0.48	1.58	1.45
p-Xylene	100	116	0	2.46	12.25	8.23
			40	0.39	1.42	1.19

TABLE 1

CHROMATOGRAPHIC COLUMN SELECTION GUIDE

EXPLANATION OF TABLE:

A blank in the table indicates that no data is available for the analysis.

COMPOUND NAMES: Wherever possible compounds are listed under the chemical name used in the Merck Index.

EXPOSURE LIMITS: Exposure limits are those set by the Occupational Safety and Health Administration (OSHA) and were taken from the Federal Register 40, 23072 May 28, 1975. These limits were current at the time of printing the chart—August, 1981

RELATIVE RESPONSE: CENTURY Organic Vapor Analyzers are factory calibrated to measure "total organic" vapors according to a standard (methane) Since different organic vapors interact with the flame ionization detector (FID) to varying extents, it is vital that the instrument user be aware of the magnitude of the variation in order to obtain the most accurate data Each user must determine relative responses for his individual instrument.

CHROMATOGRAPHIC RETENTION TIME: For chromatographic work, the OVA can be used with a variety of column lengths and packing materials. For highest accuracy, temperature control for the column is mandatory. This is accomplished using the Portable Isothermal Pack (PIP) kit which is supplied with three 8 inch columns packed with B, G, and T materials respectively. (See following chart for packing material description.) Isothermal control is accomplished nonelectrically using an ice/water mixture for 0°C and a seeded eutectic mixture for 40°C. The data listed are for comparison purposes only since retention time for a compound can vary due to the condition of the column packing material, packing procedure, and chemical interaction among the components of a vapor mixture.

COLUMN PACKING MATERIAL

B—3% Diisodecyl Phthalate on Anakrom A, AW, 60/80 mesh
E—20% Carbowax 400 on Anakrom C22A, AW, 60/80 mesh
G—10% OV-101 on Anakrom O, 60/80 mesh
T—10% 1, 2, 3-Tris (2-cyanoethoxy) Propane on Anakrom C22A, AW, 60/80 mesh
PT—Porapak T, 60/80 mesh

AMBIENT AIR ANALYSIS:

Potentially toxic vapors and gases do not fill an area uniformly Consequently, the measurement and control of these vapors can present a difficult problem for safeguarding the health of personnel. Foxboro manufactures a complete line of ambient air analyzers for on-the-spot testing as well as continuous area monitoring which have the sensitivity to detect and measure over 300 vapors and gases declared hazardous by OSHA. In addition, the analyzers are ideal for locating the source of the problems, such as a chemical leak or spill, and inadequate ventilation.

Foxboro Ambient Air Analyzers are currently being used for such applications as:

- Toxic gas and vapor analysis to comply with OSHA regulations
- Leak detection in chemical processes
- Analysis of anesthetic gases
- Measurement of vapor concentration in exposure chambers
- Analysis of vapor contaminants in "high purity" gases
- Analysis of industrial plant emissions
- Detecting breakthrough of vapors from respiratory cartridges or industrial adsorption systems
- Identify gases to aid in determining causes of fire
- Monitoring of hazardous waste and spill sites

TABLE 2

TROUBLESHOOTING

Trouble	Troubleshooting Procedures	Remedy
1. Low Sample flow rate on flow indicator. Nominally 2 units on flow gauge (See also 6 below).	a) Check the primary filter in sidepack and particle filters in the pickup assembly.	Replace or clean filter if clogged.
	b) Determine assembly containing restriction by process of elimination, i.e., remove probe, remove readout assembly, remove primary filter.	Investigate the assembly containing this restriction to determine cause of blockage. Clean or replace as required.
	c) If the restriction is in the Side Pack Assembly, further isolate by disconnecting the sample flow tubing at various points, i.e., pump output, chamber input, etc. NOTE: The inherent restrictions due to length of sample line, flame arrestors, etc., must be taken into account when trouble shooting.	If in the detector chamber, remove and clean or replace porous metal flame arrestors. If pump is found to be the problem, remove and clean or replace. Return to the equipment manager for repair.
2. H_2 flame will not light.	a) Check sample flow rate (see 1 above).	If sample flow rate is low, follow procedure 1 above.
	b) Check battery. If charge is low the instrument will often not light.	If battery is low, recharge. Once lit, the OVA will continue to run on a low battery but will not light. If the battery is not severely discharged, the charger can be hooked up to light the flame, then the instrument can be removed from the charger used until the battery is to low to run the pump.
	c) Check igniter by removing the chamber exhaust port and observing the glow when the IGNITE button is depressed.	If igniter does not light up, replace the igniter element. (The small "spark plug" that screws into the exhaust chamber.) If igniter still does not light, check the battery and wiring.
	d) Check for rated H_2 Supply Pressure. (Listed on calibration plate on pump bracket).	If low, remove battery pack and adjust to proper level by turning the allen wrench adjustment on the low pressure regulator cap.
	e) Check H_2 tank pressure gauge. OVA will run below 500 psi but becomes increasingly difficult to light.	Refill with H_2.

TABLE 2 (Cont.)

TROUBLESHOOTING

Trouble	Troubleshooting Procedures	Remedy
2) H_2 flame will not light. (Cont.)	f) Check H_2 flow rate by observing the PSI decrease in pressure on the H_2 Tank pressure gauge. The flow rate should be about 130 PSI decrease in pressure per hour. (Approximately 12 cc/min. at detector.) On instruments with GC Option, disconnect column and measure H_2 flow rate with a bubble meter.	The normal cause for H_2 flow restriction would be a blocked or partially blocked capillary tube. If flow rate is marginally low, attempt to compensate by increasing the H_2 Supply Pressure by one-half or one PSI. If flow rate cannot be compensated for, replace capillary tubing.
	g) Check all H_2 plumbing joints for leaks using soap bubble solution. Also, shut off all valves and note pressure decay on H_2 tank gauge. It should be less than 350 PSIG per hour.	Repair leaking joint.
	h) Check to see if H_2 supply system is frozen up by taking unit into a warm area.	If there is moisture in the H_2 supply system and the unit must be operated in subfreezing temperatures, purge the H_2 system with dry H_2 and ensure the H_2 gas used is dry.
	i) Check column connections on top of unit to make sure they are tight.	Tighten fitting.
	j) Check column for sharp bends or kinks. (Hydrogen flow through this column at all times and a sharp bend will compact packing too tightly for proper hydrogen flow.)	Replace column.
	k) Check charcoal filter fittings to make sure they are tight.	Tighten fittings.
	l) Check H_2 flow rate from the column.	Adjust hydrogen pressure to obtain 12 cc/min flow rate.
	m) Check that the Inject and Backflush Valves are both completely in or out. A partially activated valve will block the H_2 and air flow paths.	Ensure both valves are either completely in or out.
	n) If a new column was installed prior to problem identification, check for proper hydrogen flow rate through the column (should be approximately 12 cc/minute).	Increase hydrogen pressure to obtain proper hydrogen flow rate if column is excessively restrictive, replace or repack the column.

TABLE 2 (Cont.)

TROUBLESHOOTING

Trouble	Troubleshooting Procedures	Remedy
3) Ambient background reading in clean environment is too high.	a) Check for contamination in charcoal filter assembly. This can be detected if ambient reading increases when going in to the chromatographic mode.	Replace activated charcoal in charcoal filter assembly.
	b) Check for contamination in column.	Replace or clean column.
	c) Check for contamination in column valve assembly.	Remove valve stems and wipe with clean lint-free cloth. Heat valve assembly during operation to vaporize and remove contaminants.
	d) Check for contamination in H_2 fuel.	If H_2 fuel supply is available, drain H_2 tank. Bleed H_2 supply hose with clean H_2. Refill, purge and refill H_2 tank with ultrapure H_2. Relight OVA and check ambient readings.
4) Flame-out when operating either by-pass or GC inject valves.	a) Ensure valves are being operated with a quick, positive motion.	Operate valve with positive motion.
	b) Either H_2 or air may be leaking around one or more of the valve quad rings. Assess by tests and "O" ring inspection.	Remove stems and lightly coat with silicone grease, only on contact surface of the "O" ring. Wipe off excess (do not remove quad rings).
	c) Damaged or worn quad rings causing leak.	Return to equipment manage for repair.
5) Excessive peak tailing.	a) Change or clean GC column and see if problem disappears.	Ensure columns are clean prior to use. If one of a same type of column tails worse than others, repack the column or discard.
	b) Inspect GC valves for excessive silicone grease or contamination.	Excessively lubricant or foreign matter in the valve assembly can cause excessive tailing. Clean valve assemblies and lightly relubricate as required. Lubricant should be put only on the outside contact surface of the "O" right. Do not get grease into "O" ring grooves.

TABLE 3

TROUBLESHOOTING THE LINEAR 142 STRIP CHART RECORDER

ANALOG SECTION – MODEL 142 RECORDERS

SYMPTOM	PROBABLE CAUSE	REMEDY
Recorder is inactive	1. Fuse blown.	1. Replace fuse.
	2. Signal input leads disconnected.	2. Check input connections.
	3. Pulse servo amplifier circuit board loose in connector.	3. Check circuit board installation.
	4. Defective pulse servo amplifier circuit board.	4. Replace pulse servo amplifier circuit board.
	5. Defective servo motor.	5. Replace servo motor.
	6. Battery is disconnected.	6. Check battery connections.
	7. Battery is discharged.	7. Recharge battery.
	8. Battery is discharged and will not hold a charge.	8. Replace battery.
Excessive Deadband	1. Excessive common mode signal.	1. Connect ground terminal to source of common mode signal.
	2. Recorder ground not connected to signal source ground.	2. Provide a common ground for third wire (green or green & yellow) in power cords.
	3. Pen carriage dragging on slide rod.	3. Contact distributor or service rep.
Noise on pen trace	1. Noisy input signal from signal source.	1. Check signal source.
	2. Gain set too high.	2. Adjust gain potentiometer.
	3. Recorder ground not connected.	3. Provide a common ground for third wire (green or green & yellow) in power cord.
Pen trace has isolated steps	1. Pen carriage slide rod dirty.	1. Contact distributor or service rep.
	2. Defective servo motor.	2. Replace servo motor.
Pens will stop moving but will catch up if physically pushed	1. Defective brush contacts in servo motor.	1. Replace servo motor.
Pen writes poorly	1. Ink supply depleted.	1. Replace pen.
	2. Pen tip dried out.	2. Revitalize by dipping in water for a few seconds.

CHART DRIVE SECTION – MODEL 142 RECORDERS

SYMPTOM	PROBABLE CAUSE	REMEDY
Chart drive inoperative	1. Loose or defective power connections.	1. Check and repair power connections.
	2. Defective chart drive motor.	2. Replace chart drive motor.
	3. Defective stepper chart drive circuit.	3. (A) Check connections to circuit board. (B) Check DC voltages on circuit board. (C) Replace stepper chart drive circuit board.
	4. Drive belt too loose or too tight.	4. Contact distributor or service rep.
No chart drive in a particular chart speed	1. Loose or defective connections at chart speed selector and/or circuit board connector.	1. Check and repair connections.
	2. Defective component on circuit board.	2. Replace component or replace stepper chart drive circuit board.

INSTRUCTION MANUAL S/N 801468

MODEL PI 101

Portable Photoionization Analyzer

hnu

©HNU Systems, Inc. 1986

SECTION 2

OPERATION

2.1 INTRODUCTION/UNPACKING

Unpack the instrument carefully. The carton will contain the housing, straps, battery charger, additional probes, regulator and cylinder if ordered, spare parts, supplies and a manual. Be sure all items are removed before discarding the carton.

Attached to the instrument is a warranty card which should be filled out completely and returned to HNU Systems.

2.2 CONTROLS AND INDICATORS

The controls and indicators are located on the front panel of the readout assembly (see Figure 2-1) and are listed and described in Tables 2-1 and 2-2.

2.3 OPERATING PROCEDURES

The following procedures are to be used in operating the analyzer:

a. Unclamp the cover from the main readout assembly.

b. Remove the inner lid from the cover by pulling out the two fasteners.

c. Remove the probe, handle and cable from the cover. Attach the handle to the front part of the probe.

d. Connect the probe cable plug to the 12 pin keyed socket on the readout assembly panel. Carefully match the alignment slot in the plug to the key in the connector. Screw down the probe connector until a distinct snap and lock is felt.

e. Screw the probe extension into the probe end cap. The probe may be used without the extension if desired.

f. Set the SPAN control for the probe being used (10.2, 9.5, or 11.7 eV) as specified by the initial factory calibration or by subsequent calibrations.

TABLE 2-1

CONTROLS

Name	Position	Function
Function Switch	---	Controls the operation of the analyzer
	OFF	All operations OFF
	BATT (battery check)	Checks the condition of the battery. If the meter needle is in the green arc, the battery is charged. If not the battery should be recharged. Charging can be done in any position, best in OFF; see directions on charger.
	STANDBY	All electronics ON, ultraviolet (UV) light source OFF. This position conserves power and extends battery life. This position is used to set the analyzer zero position. (i.e. no UV light, no signal)
	0-2000	Sets range of meter at 0-2000 ppm.
	0-200	Sets range of meter at 0-200 ppm.
	0-20	Sets range of meter at 0-20 ppm.
ZERO	---	With the function switch in STANDBY position, this potentiometer is used to adjust the reading to zero.

NOTE: See Figure 2-1 for locations.

TABLE 2-2

INDICATORS AND DISPLAYS

Name	Function
Low Battery Indicator Light (red light) (see NOTE)	Illuminates when battery is discharged, indicates need for recharge. Do not use unit when this light is ON. Readings may be taken while battery is being recharged.
Meter (see NOTE)	Indicates concentration of measured gas.
Recorder (optional) (see Figures 2-1 And 8-3)	Provides a record of readings while analyzer operates unattended. Recorder inputs 0 to -5 V DC.

NOTE: See Figure 2-1 for locations.

n. The analyzer is now operational.

o. Hold the probe so that the extension is at the point where the measurement is to be made. The instrument measures the concentration by drawing the gas in at the end of the extension, through the ionization chamber, and out the handle end of the probe.

WARNING

The instrument measures gases in the vicinity of the operator and a high reading when measuring toxic or explosive gases should be cause for action for operator safety.

p. Take the reading or readings as desired taking into account that air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings. Change the ranges as required.

q. Check battery condition as required. If the Low Battery Indicator comes on, turn analyzer off and recharge.

CAUTION

Use only in an emergency with a low battery when on battery charge.

SECTION 3

CALIBRATION

3.1 INTRODUCTION

The PI 101 Analyzer is designed for trace gas analysis in ambient air and is calibrated at HNU with certified standards of benzene, vinyl chloride and isobutylene. Other optional calibrations are available (e.g., ammonia, ethylene oxide, H2S, etc.). Calibration data is given in the data sheet. If a special calibration has been done, the data is given in the Application Data Sheet, which notes the sample source, type of calibration (see Section 8, Appendix), and other pertinent information.

Good instrumentation practice calls for calibration on the species to be measured in the concentration range to be used. This procedure assures the operator that the analyzer is operating properly and will generate reliable data.

Some general points to consider when calibrating the PI 101 are that the analyzer is designed for operation at ambient conditions and therefore the gas standards used for calibration should be delivered to the analyzer at ambient temperatures and pressure and at the proper flow rates.

WARNING:

The PI 101 is a non-destructive analyzer; calibrations using toxic or hazardous gases must be done in a hood.

The frequency of calibration should be dictated by the usage of the analyzer and the toxicity of the species measured. If the analyzer has been serviced or repaired, calibration should be done to verify operation and performance. It is recommended that calibration be checked frequently at first (daily or every other day) and then regularly based on the confidence level developed.

The normal meter scaleplate is 0 to 20. If the scaleplate is different, refer to the Application Data Sheet. If there are questions, consult the HNU representative before proceeding with calibration check.

An accurate and reliable method of calibration check is to use an analyzed gas cylinder in a test setup as shown in Figure 3-1 and described below. Additional material on calibration is given in Section 8, Appendix.

3.2 ANALYZED GAS CYLINDER

a. Concentration - The calibration gas cylinder is to contain the species of interest made up in an air matrix at or near the concentration to be analyzed. If the component is unstable in air, another matrix is to be used. The final calibration mixture should be similar to the sample the PI 101 will analyze. If the expected concentration is not known then a concentration should be chosen that will cause a scale displacement of 50 to 80% on the X10 range. Calibration on X10 range will provide accurate values on the X1 range as well.

This insures that the PI 101 sees the calibration gas at atmospheric pressure and ambient temperature.

d. Usage - Generally, a gas cylinder should not be used below 200-300 psi as pressure effects could cause concentration variations. The cylinder should not be used past the recommended age of the contents as indicated by the manufacturer. In case of difficulty, verify the contents and concentration of the gas cylinder.

e. Alternate means of calibration are possible. For more information, contact the HNU Service Department.

3.3 PROBE

a. Identify the probe by the lamp label. If a question exists, disassemble the probe and inspect the lamp. The energy of the lamp is etched into the glass envelope.

b. Connect the probe to the readout assembly, making sure the red interlock switch is depressed by the ring on the connector.

c. Set the SPAN pot to the proper value for the probe being calibrated. Refer to the calibration memo accompanying the probe.

d. Check the Ionization Potential (IP) of the calibration gas to be used. The IP of the calibration gas must be at or below the IP of the lamp.

e. Proceed with the calibration as described in Section 3.4. Check the calibration memo for specific data. If any questions develop, call the HNU representative.

f. NOTE: The 11.7eV lamp has a special cleaning compound. Do not use water or any other cleaning compound with the 11.7 eV lamp. Do not interchange ion chambers, amplifier boards or lamps between probes. (See Section 5.2).

3.4 PROCEDURE

a. Battery check - Turn the function switch to BATT. The needle should be in the green region. If not, recharge the battery.

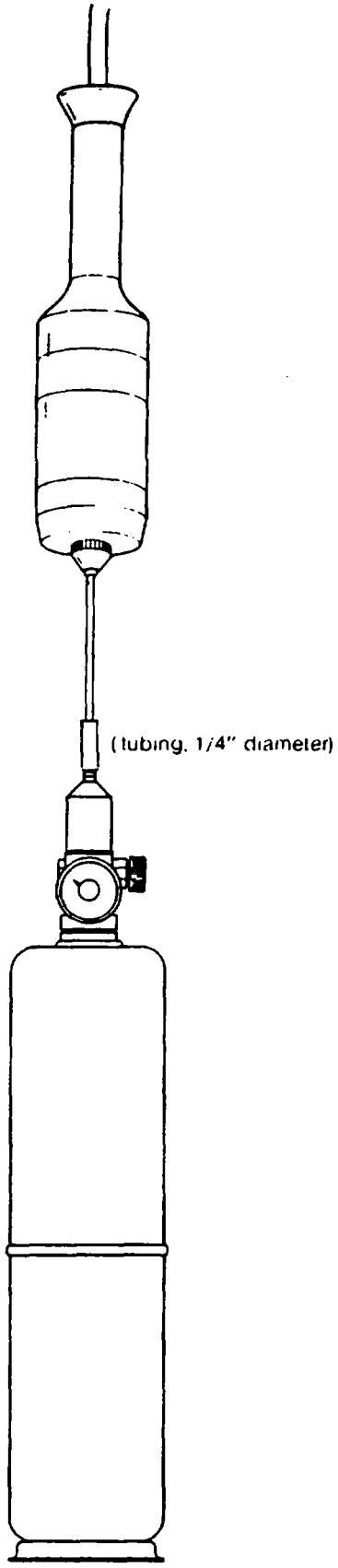


FIGURE 3-1
CALIBRATION TEST SET UP

3.0 STANDARD OPERATING PROCEDURE FOR MEASURING WATER TEMPERATURE

3.1 Calibration

- Calibration of thermometers will be performed before entering the field and checked upon return to the office.

- Thermometers will be calibrated against a National Bureau of Standards (NBS) - traceable thermometer.

- The thermometer must read within 1° - 1.5° C of the NBS - traceable thermometer. If the thermometer does not read within this range and the thermometer cannot be calibrated, then it will not be used for temperature measurements and will be disposed of in an appropriate manner. If the thermometer does not read within this range and the thermometer can be calibrated, then the thermometer will be calibrated to the NBS - traceable thermometer.

- The following information is documented in the calibration logbook at the time of calibration:

  a. Date
  b. Thermometer Identification
  c. Initials
  d. Calibration Data

3.2 Procedure

- The thermometer is immersed in water until the temperature equilibrates. The temperature is read in Celsius (°C).

- Temperature data are recorded in the field notebook, and initialed and dated.

4.0 STANDARD OPERATING PROCEDURE FOR MEASURING THE pH OF WATER SAMPLES

4.1 Calibration

- Calibration of the pH meter is to be performed prior to its use each day at the end of the day, and at least every four hours.

- Recalibration must occur if: (1) the pH of the samples being measured is outside the previous calibration range, or (2) the battery is replaced.

- Two buffer calibrations bracketing the expected pH range of samples are to be performed prior to the meters use each day. Three pH buffers (4.0, 7.0, and 10.0) are read after standardization at pH of 7.0 to evaluate the linearity and electrodes. The samples and buffers are to be measured at the same temperature.

- The following information is documented in the calibration logbook at the time of calibration:

  a. Date
  b. pH meter identification
  c. Initials
  d. Calibration results using pH standards

4.2 Procedure

- The pH electrode must be kept moist.

- The electrodes must be carefully rinsed with deionized water before each measurement.

- Follow manufacturer's operating instructions.

- The pH readings are documented in the field notebook, and initialed and dated.

- The electrodes are rinsed with deionized/distilled water and the unit stored properly. The electrodes are not to be stored in tap water or deionized/distilled water.

5.0 STANDARD OPERATING PROCEDURE FOR MEASURING THE CONDUCTIVITY OF WATER SAMPLES

5.1 Calibration

- Calibration is in accordance with the manufacturer's specific directions, and the following information is documented in the calibration logbook:

  a. Date
  b. Conductivity meter identification
  c. Calibration results
  d. Initials

- Calibration is performed at the beginning and end of the day, and at least every four hours.

5.2 Procedure

- The probe is immersed in a water sample until the meter equilibrates.

- In reading the conductivity meter scale, one or more of the following may have to be considered:

  a. The reading may have to be multiplied appropriately (e.g., the reading is expressed in x1, x10, x100 scales).

  b. If the conductivity meter is not capable of compensating for temperature differences, then note that the conductance measurements are not temperature compensated and document the temperatures of the standards and samples.

  c. If the conductivity meter can be compensated for temperature, then adjust the temperature control before reading the conductance measurement.

- Conductivity measurements and any other relevant information are recorded in the field notebook, and initialed and dated.

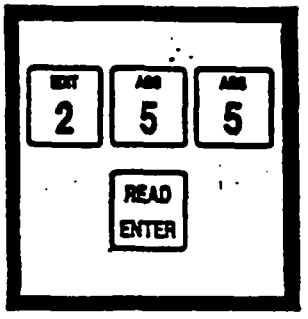
6.0 STANDARD OPERATING PROCEDURE FOR MEASURING TOTAL AND FERROUS IRON IN WATER SAMPLES

IRON, FERROUS (0 to 3.00 mg/L)

For water, wastewater and seawater

1,10 Phenanthroline Method* (Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



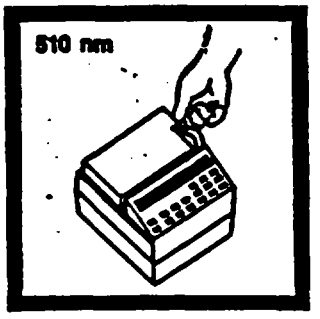
1. Enter the stored program number for ferrous iron, (Fe^{2+})–powder pillows.

Press: 2 5 5 READ/ENTER

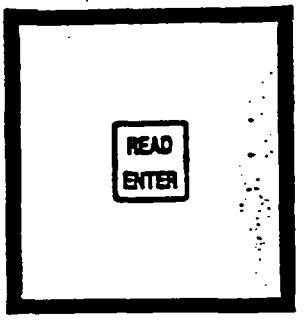
The display will show:
DIAL nm TO 510

Note: Or, use the up and down arrows to scroll the display to:
255 mg/l Fe^{2+}
and press: READ/ENTER

Note: Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.



2. Rotate the wavelength dial until the small display shows:
510 nm



3. Press: READ/ENTER

The display will show:
mg/l Fe^{2+}

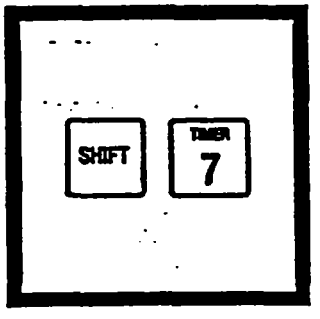
4. Fill a sample cell with 25 mL of sample.

Note: For proof of accuracy, use a 1.0 mg/L ferrous iron standard solution (preparation given in the Accuracy Check) in place of the sample.

5. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

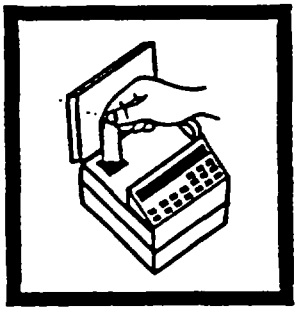
Note: An orange color will form if ferrous iron is present.

Note: Undissolved powder does not affect accuracy.



6. Press: SHIFT TIMER

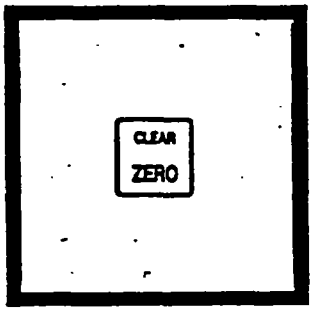
A three-minute reaction period will begin.



7. When the timer beeps, the display will show:
mg/l Fe^{2+}

Fill a second sample cell (the blank) with 25 mL of sample. Place it into the cell holder.

Note: The Pour-Thru Cell can be used with this procedure.



8. Press: ZERO

The display will show:
WAIT

then:
0.00 mg/l Fe^{2+}

ACCURACY CHECK

Prepare a ferrous iron stock solution (100 mg/L Fe) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.0 mg/L standard solution. Prepare this immediately before use.

PRECISION

In a single laboratory using an iron standard solution of 1.000 mg/L Fe^{2+} and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.006 mg/L Fe^{2+}.

In a single laboratory using a standard solution of 1.000 mg/L Fe^{2+} and two representative lots of AccuVac ampuls with the DR/2000, a single operator obtained a standard deviation of ± 0.009 mg/L Fe^{2+}.

SUMMARY OF METHOD

The 1,10 phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test. See *Chemical Procedures Explained*, Publication 7013, for more information.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
Ferrous Iron Reagent Powder Pillows	1 pillow	100/pkg	1037-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

Ferrous Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25140-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	43784-00
Beaker, 50 mL	1	each	500-41
Vial, zeroing	1	each	21228-00

OPTIONAL REAGENTS

Ferrous ammonium sulfate, hexahydrate	113 g	11256-14
Water, deionized	3.78 L	272-17

OPTIONAL APPARATUS

Flask, volumetric, 100 mL	each	547-42
Flask, volumetric, 1000 mL	each	547-53
Pipet, volumetric, 1 mL	each	515-35
Pipet filler, safety bulb	each	14651-00
Pour-Thru Cell Assembly Kit	each	45215-00

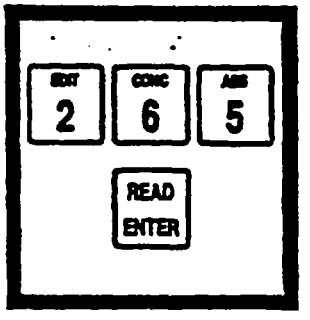
For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

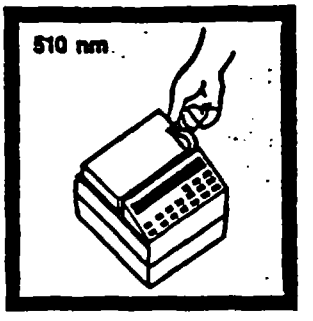
IRON, TOTAL (0 to 3.00 mg/L)

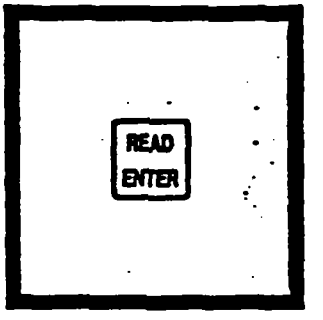
For water, wastewater and seawater

FerroVer Method* (Powder Pillows or AccuVac Ampuls), EPA Approved†—Digestion is required; see Section I.

USING POWDER PILLOWS







1. Enter the stored program number for iron (Fe), FerroVer, powder pillows.

Press: 2 6 5 READ/ENTER

The display will show:
DIAL nm TO 510

Note: Or, use the up and down arrows to scroll the display to:
265 mg/l Fe FV
and press: READ/ENTER

Note: If samples cannot be analyzed immediately, see Sampling and Storage below. Adjust pH of stored samples before analysis.

2. Rotate the wavelength dial until the small display shows:
510 nm

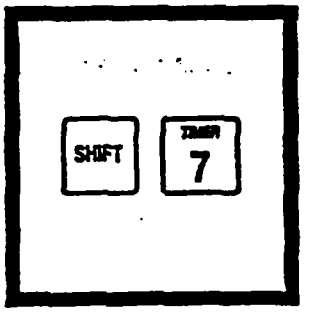
Note: Determination of total iron needs a prior digestion; use the mild, vigorous or Digesdahl digestion (Section I).

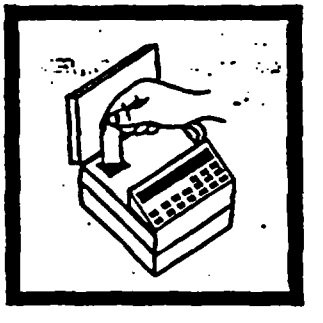
3. Press: READ/ENTER

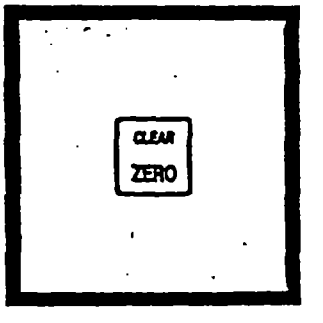
The display will show:
mg/l Fe FV

4. Fill a cell with 25 mL of sample.

Note: For proof of accuracy, use a 1.0 mg/L iron standard solution (preparation given in the Accuracy Check) in place of the sample.







5. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

Note: An orange color will form if iron is present.

Note: Accuracy is not affected by undissolved powder.

6. Press: SHIFT TIMER

A three minute reaction period will begin.

Note: Samples containing visible rust should be allowed to react at least five minutes.

7. When the timer beeps, the display will show:
mg/l Fe FV
Fill another sample cell (the blank) with 25 mL of sample. Place it into the cell holder.

Note: For turbid samples, treat the blank with one 0.2-gram scoop of RoVer Rust Remover. Swirl to mix.

Note: The Pour-Thru Cell can be used with this procedure.

8. Press: ZERO

The display will show:
WAIT
then:
0.00 mg/l Fe FV

*Adapted from *Standard Methods for the Examination of Water and Wastewater*
†*Federal Register*, 45 (126) 43459 (June 27, 1980)

SAMPLING AND STORAGE

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide before analysis. Correct the test result for volume additions; see Sampling and Storage, Volume Additions (Section I) for more information.

If only dissolved iron is to be determined, filter the sample before acid addition using the labware listed under Optional Apparatus. No acid addition is necessary if analyzing the sample immediately.

ACCURACY CHECK

a) Snap the neck off an Iron Voluette Ampule Standard Solution, 50 mg/L.

b) Use the TenSette pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL water samples and mix thoroughly. (For AccuVac Ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Prepare a 1.0 mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water. Or, use the TenSette pipet to dilute 1.0 mL of the contents of an Iron Voluette Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily.

PRECISION

In a single laboratory, using a standard solution of 1.000 mg/L Fe and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.006 mg/L.

In a single laboratory, using a standard solution of 1.000 mg/L Fe and two representative lots of AccuVac ampuls with the DR/2000, a single operator obtained a standard deviation of ± 0.009 mg/L Fe.

INTERFERENCES

The following will not interfere below the levels shown:

Chloride	185,000 mg/L
Calcium	10,000 mg/L as $CaCO_3$
Magnesium	100,000 mg/L as $CaCO_3$

A large excess of iron will inhibit color development. A diluted sample should be tested if there is any doubt about the validity of a result.

FerroVer Iron Reagent Powder Pillows and AccuVac Ampuls contain a masking agent which eliminates potential interferences from copper.

Samples containing some forms of iron oxide require the mild, vigorous or Digesdahl digestion (Section I). After digestion adjust the pH to between 2.5 and 5 with ammonium hydroxide.

Samples containing large amounts of sulfide should be treated as follows in a fume hood, or well ventilated area: Add 5 mL of Hydrochloric Acid to 100 mL of sample and boil for 20 minutes. Adjust the pH to between 2.5 and 5 with 5 N Sodium Hydroxide and readjust the volume to 100 mL with deionized water. Analyze as described above.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

REAGENT STORAGE

FerroVer Reagent Powder Pillows are stable indefinitely if stored properly. A cool, dry atmosphere is recommended. The reagent can be checked by adding the contents of a pillow to about 25 mL of water containing visual rust (such as a few drops of Rust Suspension). If the orange color does not form, the reagent should be replaced.

SUMMARY OF METHOD

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample, to produce soluble ferrous iron. This reacts with the 1,10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration. See *Chemical Procedures Explained*, Publication 7013, for more information.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
FerroVer Reagent Powder Pillows	1 pillow	50/pkg	854-66

REQUIRED REAGENTS (Using AccuVac Ampuls)

FerroVer Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25070-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac vial	1	each	43784-00
Beaker, 50 mL	1	each	500-41
Vial, zeroing	1	each	21228-00

OPTIONAL REAGENTS

Ammonium hydroxide, ACS	473 mL	106-11
Hydrochloric Acid, 6 N	500 mL	884-49
Hydrochloric acid, ACS	473 mL	134-11
Iron Standard Solution, 100 mg/L	118 mL	14175-14
Iron Voluette Ampule Standard, 50 mg/L	16/pkg	14254-10
Nitric acid, ACS	473 mL	152-11
Nitric Acid Solution, 1:1	473 mL	2540-11
RoVer Rust Remover	28 g	300-20
Rust Suspension	15 mL DB	1279-36
Sodium Hydroxide Standard Solution, 5.0 N	118 mL MDB	2450-37
Water, deionized	3.78 L	272-17

OPTIONAL APPARATUS

Cylinder, graduated, poly, 25 mL	each	1081-40
Cylinder, graduated, poly, 100 mL	each	1081-42
Filter discs, glass, 47 mm	100/pkg	2530-00
Filter holder, membrane	each	2340-00
Filter pump	each	2131-00
Flask, erlenmeyer, 250 mL	each	505-46
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Hot plate, micro	each	12067-01
pH meter, Hach One	each	43800-00
pH indicator paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, tips for 19700-01	50/pkg	21856-96
Pipet, volumetric, 1.00 mL	each	515-35
Pour-Thru Cell Assembly Kit	each	45215-00
Spoon, measuring, 0.1 g	each	511-00
Spoon, measuring, 0.2 g	each	638-00

For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

7.0 STANDARD OPERATING PROCEDURE FOR COLLECTION OF TIME WEIGHTED AVERAGE AIR SAMPLES

7.1 Purpose

This technical procedure is to be used to establish a uniform procedure for the collection of air samples for laboratory quantification of constituents.

7.2 Applicability

This technical procedure is applicable to all personnel involved with the collection of air samples.

7.3 Definitions

- Summa Passivated Cylinder: A stainless steel cylinder with an inlet valve where the surfaces which contact the sample have been conditioned to reduce reactivity of the sample.

- Vacuum Flow Regulator: A flow regulator which attaches to the Summa Cylinder and allows the air sample to enter the cylinder at a controlled rate. A vacuum gauge is attached to the flow regulator.

- Cryogenic Focusing: A method of pre-concentrating an air sample where a precise volume of the sample is pumped through a supercooled loop where the analytes are condensed and then liberated by heating into a precise volume of gas which is smaller than that of the original sample.

7.4 Discussion

This procedure is based upon USEPA Method TO-14, which is widely considered to be one of the most reliable and sensitive test methods for ambient air toxics.

7.5 Equipment and Materials

- Summa passivated cylinders (see Figure 1) equipped with vacuum flow regulators and vacuum gauges. The cylinders will have been

cleaned and evacuated, and the flow regulator will have been calibrated before and after sampling, by the analytical laboratory.

- Air ultra zero.
- Teflon or stainless steel tubing and connections to the Summa cannisters.
- Shipping containers and chain-of-custody documentation (Figure 2).

7.6 Procedure

All samples should be collected at approximately the same time. Inlet valves on the Summa cylinders will have to be opened manually and will therefore vary in the exact start and stop time. However, they can be collected at approximately the same time within a window of ±2 hours.

Upon arrival at the Site, check that all necessary sampling equipment is available. Plan to deploy the samplers in early afternoon such that the shipping time to the laboratory can be minimized.

When deploying the Summa cylinders (by opening the inlet valve) note the time and the vacuum gauge reading on the cylinder chain-of-custody form. Also note a description of the sample location, any noticeable odors, and approximate meteorological conditions such as temperature, wind speed and direction, cloud cover, and precipitation in the field book. Two cylinders should be deployed at the duplicate sample station.

Vents can be sampled using the Summa cylinders by installing a hole into the vent (sample port), inserting stainless steel or Teflon tubing into the vent sufficiently upstream of the sample port (approximately 12 inches) and sealing the sample port securing the tubing. The Summa cylinder is then connected to the tubing (see 13.6.1 below).

At the end of the sampling period (usually 8 or 24 hours) record the time and vacuum gauge reading on the chain-of-custody form, close the valve, and note any changes in the site conditions in the field book. Return the cylinder to the shipping container and record the date and time of sampling on the chain-of-custody record. Collect cylinders from all stations in the same order that the cylinders were deployed.

After all chain-of-custody documentation has been completed, seal the form in the shipping container with the samples and send them to the laboratory by overnight courier. Notify the laboratory that the samples have been shipped.

7.7 Specific Procedures for Gas Vent Sampling Using Summa Passivated Canisters

The analytical laboratory will have prepared (cleaned) the Summa canisters and calibrated the inlet flow rate prior to sampling. The vacuum pressure of the canister should be checked prior to sampling to ensure that it has not leaked during transit from the laboratory to the site. Chain-of-custody documentation should also be inspected and completed in accordance with the QAPjP.

The gas vent will have a small hole drilled in the side of the riser pipe in order to measure the gas exit velocity, temperature, and humidity and to provide access for sampling. A length of Teflon on stainless steel tubing can be inserted through this hole, or through the top of the riser pipe, and connected to the Summa canister inlet using a Swagelock (or equivalent) compression fitting. The end of the tubing should extend 12 inches upstream of the vent access port which should be sealed during sampling.

The inlet to the Summa canister should be opened to begin sampling. Sampling will proceed without the need for any operator attendance. At the end of the designated sampling period (usually 8 or 24 hours depending on the size of the

canister and the sampling flow rate) the inlet valve to the canister should be closed and the vacuum pressure in the canister recorded.

The canister should be shipped to the laboratory in accordance with the chain-of-custody procedures given in the QAPjP. Special care should be used when handling and packing the canisters to prevent damage to the valves.

Notes should be kep of the date and time that sampling was initialized and terminated at each location, weather conditions such as cloud cover, temperature, humidity, precipitation (including any recent precipitation prior to sampling), and barometric pressure. All notes and paperwork should include the project name and number, the sampler's name, and the date.

7.8 References

Bernstein, D.M, and M. Lippman. Calibration of Air Sampling Instruments.

California Air Resources Board, 1986. Testing Guidelines for Active Solid Waste Disposal Sites.

U.S. Environmental Protection Agency, 1984. Characterization of Hazardous Waste Site - A Methods Manual: Volume II. Available Sampling Methods, Second Edition.

U.S. Environmental Protection Agency, 1987. A Compendium of Superfund Field Operations Methods.

U.S. Environmental Protection Agency, 1984. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.

SECTION I - FIELD SAMPLING PLAN

APPENDIX A

SAMPLING AND FIELD TESTING PROCEDURES

1.0 INTRODUCTION

The performance evaluation objectives are to collect data of adequate technical content, quality, and quantity to complete a performance evaluation of the existing groundwater treatment plant which will be used to determine the ability of the treatment plant to meet the allowable discharge criteria.

The purpose of this FSP is to describe the sampling program rationale and procedures that will result in data of suitable quality and quantity to achieve the performance evaluation objectives.

To achieve these objectives efficiently, specific field procedures have been developed for the collection of samples from potentially affected media in the study area. These procedures are described in the following sections.

This Appendix contains the following sections that detail the field methods employed in the Removal Action Work Plan:

1.0 Introduction
2.0 PID Photoionization Detector
3.0 Water Temperature
4.0 pH
5.0 Specific Conductance
6.0 Iron
7.0 Gas Samples
8.0 Groundwater and Surface Water Sampling
9.0 Soil and Sediment Sampling

2.0 STANDARD OPERATING PROCEDURE FOR MEASURING VOCs IN AMBIENT WITH PHOTOIONIZATION DETECTOR, PID

1. PURPOSE

The purpose of this technical procedure is to establish methodology for the calibration, operation in both the survey and gas chromatograph (GC) modes, and routine maintenance of the Foxboro Century Systems Portable Organic Vapor Analyzer (hereafter referred to as the OVA).

2. APPLICABILITY

This procedure applies to all personnel engaged in the use of the Foxboro OVA. The OVA will typically find applications in hazardous site investigations and air monitoring for health and safety. In the survey mode, the OVA serves to detect total volatile organic vapors and gases in the low parts per million (volume/volume) concentrations. In the GC mode, the OVA can be used to tentatively identify and quantify specific volatile organic compounds in air (ppm range) and indirectly in soil and water (ppb range). In health and safety applications, even though one may not know exactly what is there, the OVA may be especially useful in the GC mode for establishing that certain compounds with very low allowable exposure limits, such as benzene, are not present.

3. DEFINITIONS

3.1 Organic Vapor Analyzer (OVA)

The OVA is a highly sensitive electronic instrument designed to measure trace quantities of organic materials in air by means of a flame ionization detector.

3.2 Gas Chromatograph (GC) Mode

Operating the OVA in the gas chromatographic or "GC" mode (as opposed to the direct read-out or "survey" mode) whereby a 250 μl volume of sample air is injected onto a GC column on the OVA with hydrogen as a carrier gas.

3.3 Calibration Gas

Methane or specific gas(es) of interest in air at known, certified concentrations are used for calibrating the OVA.

4. REFERENCES

4.1 Century Systems, 1985, "Operating and Service Manual for Century Systems: Portable Organic Vapor Analyzer (OVA) Model OVA-128 and Optional Accessories," Century Systems Corporation, Arkansas City, Kansas.

4.2 Linear Instruments Corporation, "Operating Instructions 141/142 Recorders," Linear Instruments Corporation, Stockton, California.

4.3 Golder Associates' Procedure P-12.0-1, "Calibration and Maintenance of Measuring and Test Equipment."

5. DISCUSSION

The Century Systems Portable Organic Vapor Analyzer (OVA) is designed to detect and measure a wide spectrum of volatile organic gases and vapors, but is most effective and finds the widest applications in the detection of petroleum hydrocarbons.

The instrument utilizes the principle of flame ionization for detection and measurement of organic gases and vapors. Figure 1 illustrates the gas handling system of the OVA.

In the survey mode, a continuous flow of sample air is drawn into the probe and conveyed to the detection chamber by an internal diaphragm pump. The sample flow rate is metered and passed through particle filters before reaching the detector chamber. Inside the detection chamber, the sample is passed through a hydrogen flame.

The carbon atoms which are present if the sample air is contaminated with organic hydrocarbons are ionized in the flame and subsequently collected by highly charged collector electrodes in the chamber. An electric field exists which drives the ions to the collecting electrodes. As the ions are collected, a current proportional to the ion flow rate is generated through the "circuit." The instrument "readout" is actually the voltage (greatly amplified) associated with the current (ion) flow measured across a resistor in the circuit.

6. RESPONSIBILITIES

6.1 Project Manager

The Project Manager shall be responsible for the following:

- Identifies situations where an OVA may be appropriate (project applications) and/or necessary (health and safety applications).

- Specifies the methodology, frequency of use, location of use, and measurement criteria of the OVA;
- Verifies appropriate calibrations, surveys, and maintenance activities of the OVA.

6.2 Task Leader

The Task Leader shall be responsible for the following:

- Assigns personnel to calibrate, operate, and maintain the OVA in the field;
- Ensures that the OVA is calibrated, used, and maintained correctly in the field and that these activities are documented;
- Ensures that the OVA is functioning properly at all times; and
- Informs the Equipment Manager when the OVA is not functioning properly.

6.3 Field Technicians

Field Technicians shall be responsible for the following:

- Being competent in the calibration, operation, and maintenance of the OVA;
- Documenting all calibrations, survey results, and maintenance activities; and
- Informing the Project Managers or Task Leaders when the OVA is not functioning properly.

6.4 Equipment Manager

The Equipment Manager shall be responsible for the following:

- Performs all OVA check out and check in procedures on equipment before it goes to the field and when it returns, respectively;
- Follows factory recommended calibration and maintenance schedules of the OVA;

- Maintains a file of periodic routine OVA calibration, operation, and maintenance information in accordance with procedure P-12.0-1; and
- Ensures that the OVA is properly maintained and stored when not in the field.

6.5 Health and Safety Officer

The Health and Safety Officer shall have the following responsibilities:

- Oversite of in-house personnel who calibrate, operate, and maintain the OVA;
- Training personnel on the use of the OVA;
- Reviewing the proposed use and measurement criteria of the OVA in the Health and Safety Plan.
- Reviewing calibration records and results of health and safety surveys; and
- Evaluating monitoring results and, when appropriate, adjusting Health and Safety Plan to reflect new information.

7. EQUIPMENT

- Portable Organic Vapor Analyzer (OVA) Model-128 Side Pack Assembly including the operating controls and indicators, the electronic circuitry, detector chamber, hydrogen gas supply, gas chromatographic column and electrical power supply.
- Probe/Readout Assembly
- Replacement sintered metal particulate filters
- Instrument carrying case
- A.C. battery charger
- Calibration gas (methane) or other specific gases
- regulator(s) for compressed gas bottles
- Air Monitoring Data Sheet (Exhibit A)
- Slotted screwdrivers (large & jewelers sizes)

1

- Phillips screwdriver
- 12 and 6 inch Crescent wrenches
- OVA Allen wrench kit
- Spare ignition element
- Single channel strip chart recorder (for use in GC mode), such as the Linear Model 142 or equivalent, spare chart paper and pens, power cord and patch cord. 1
- Tedlar bags
- Thermometer for use in the GC mode especially if temperature is expected to be variable.

8. PROCEDURES

8.1 Safety Precaution

The OVA Model 128 has been tested and certified by Factory Mutual Research Corporation as intrinsically safe for use in Class I, Division 1, Groups A, B, C & D hazardous atmospheres. However, the OVA uses hydrogen as a fuel source. Mixtures of hydrogen and air are flammable over a wide range of concentrations. Precautions should be taken during hydrogen filling or tank emptying to ensure there are no sources of ignition in the immediate area.

A general understanding of the operation of the OVA and the fact that it indicates an "integrated average" of concentrations of total volatile organic gases present is necessary for its use as a monitoring device. If other conditions suggest that the air may be unsafe for breathing (such as unusual odors, visible contamination, etc.), appropriate measures must be taken to protect the individuals in the field regardless of the instrument reading.

8.2 Equipment Check Out and Check In

The following activities are the responsibility of the Equipment Manager for check out and check in of the OVA to and from Golder Associates Inc. personnel:

- Periodic "Bench Top" calibration (Section 8.3)
- Checking air sampling line probe tip and side pack assembly filters (Section 8.6.3.4 and 5) 1

- Cleaning exhaust flame arrestor (Section 8.6.6)
- Seal maintenance (Section 8.6.7)
- Adjusting refiller valve (Section 8.6.8)
- Refueling H_2 tank (Section 8.6.1)
- Battery charging (Section 8.6.2)

8.3 Calibration

The OVA is capable of responding to a greater or lesser extent, to nearly all organic compounds in a gaseous or vapor state. However, the response may vary considerably depending on the compound. For precise analysis it is necessary to calibrate the instrument directly to the specific compound of interest.

The instrument is factory calibrated to a methane-in-air standard. However, it can be easily and rapidly calibrated to a variety of organic compounds. A GAS SELECT (span) control is incorporated on the instrument panel which may be set to give a direct reading for a particular organic compound.

Internal "span pots" (potentiometers) are provided to calibrate and align the three sensitivity ranges. There are four adjustments located on the internal electronics board. One adjustment potentiometer, R-38, is used to set the power supply voltage and should not be changed. The remaining three adjustments, R-31, R-32, and R-33 are used to set the electronic amplifier gain (span) for the x1, x10, and x100 sensitivity scales, respectively.

8.3.1 *Field Calibration to Methane*

At a minimum, the calibration of the OVA shall be checked and documented against a 9 ppm methane-in-air standard at the beginning and end of each day of use.

Gain Adjustment

1. After a sufficient warm-up period (5 - 10 minutes), place instrument in normal operational mode (inject switch "up") with CALIBRATE Switch set to x1 and GAS SELECT control set to 3.0.

2. Use the CALIBRATE ADJUST (zero) Knob and adjust the meter reading to zero.

3. Introduce methane calibration gas (9 ppm methane-in-air) from a tedlar bag and adjust the Gas Select Knob until the instrument reads 9 ppm. Document this span setting. Switch to the x10 scale and verify that the instrument read-out is approximately (0.70-1.1) ".9" (.9 x 10 = 9 ppm). If the instrument is used to quantify higher concentrations of organic vapors (i.e., x10 or x100 scale) the instrument calibration should be checked in each of these ranges as well using 90 ppm and 900 ppm methane-in-air, respectively as discussed in item 4. below.

4. Verify that the instrument reads between 80 and 100 ppm in the x10 range and between 800 and 1000 ppm in the x100 range in response to 90 ppm and 900 ppm methane, respectively. If the instrument readings are outside of this acceptable range in the x10 and x100 scales, adjust and record the span (Gas Select) settings required to obtain readings of exactly 90 ppm and 900 ppm, respectively. Return the instrument to the x1 scale and span setting identified in step 3 above. The Gas Select (span) setting should be adjusted to the appropriate value (determined during calibration in the x10 and x100 scales as discussed above) whenever quantitative readings are required in the x10 or x100 sensitivity range.

8.3.2 "Bench" Calibration to Methane

The OVA shall be subject to the controls specified in procedure P-12.0-1, "Calibration and Maintenance of Measuring and Test Equipment." Whenever there is any indication that the instrument is not within acceptable calibration limits (i.e., readings outside of 8-10 ppm, 80-100 ppm, and 800-1000 ppm in response to 9, 90, and 900 ppm methane, respectively, with "Gas Select" set at 3.0), but at least once every three months, the OVA should be bench calibrated in all three ranges. Calibration required by procedure P-12.0-1 shall be performed as follows:

1. Disconnect the air sample line and umbilical cord, and remove the ignitor button guard. Remove the OVA from its case by loosening the 4 quarter turn mounting screws in the faceplate and gently pulling the body of the instrument out and up.

2. Re-connect the air sampling line and umbilical cord and turn instrument on.

3. After a sufficient warm-up period, set the Gas Select to 3.0, and set the "Adjust" knob such that the instrument reads "zero" in clean air in the x1 scale. Change the scale setting to x100.

4. Introduce 900 ppm methane-in-air to the instrument from a Tedlar bag. Do not introduce gas directly into the probe from a cylinder or regulator. Adjust the internal span pot "R33" in the ceramic circuit board until the instrument reads exactly 900 ppm (a reading of "9" on the read-out probe x100) with the gas select setting on 3.0.

5. Disconnect the 900 ppm methane and introduce 90 ppm methane while the instrument is still in the x100 scale. Verify that the instrument reads between 80 and 100 ppm (.8 to 1 on the read-out probe) in the x100 scale. Switch to the x10 scale and adjust the interval span pot R32 until the instrument reads exactly 90 ppm in the x10 scale (a reading of "9" on the read-out dial x10).

6. Disconnect the 90 ppm methane and introduce 9 ppm menthane while the instrument is still in the x10 scale. Verify that it reads between 8 and 10 ppm (.8 to 1 on the read-out dial). Switch to the x1 scale. Adjust the interval span pot R31 until the instrument reads exactly 9 ppm in the x1 scale.

7. Verify that the Gas Select is set to 3.0. Verify that the instrument is reading 9 ppm in the x1 scale in response to 9 ppm methane. Switch to the x10 scale and verify that the instrument reads approximately .9 on the read-out dial.

8. Reintroduce 90 ppm methane and verify that the instrument reads 90 ppm (9 on the read-out dial) in the x10 scale. Readjust span pot R32 if necessary. Switch to the x100 scale and verify that the instrument reads approximately .9 on the read-out dial.

9. Reintroduce 900 ppm methane and verify that the instrument reads 900 ppm in the x100 scale. Adjust span pot R33 if necessary.

10. Replace instrument in case. Replace ignition button guard, and reconnect air sampling line and umbilical cord. 1

    Document the calibration and indicate the date of last calibration on the instrument case in accordance with procedure P-12.0-1.

8.3.3 Calibration of Other Organic Gases

1. Perform the field calibration check for methane.

2. Draw a sample of the desired calibration gas into the instrument.

3. Adjust the GAS SELECT Knob on the panel to shift the readout meter indication to correspond to the actual concentration of the calibration gas mixture.

4. The instrument is now calibrated to the organic gas being used. Document this "Gas Select" setting. Whenever the "Gas Select" is set to this value, the instrument will give direct readings for this gas.

8.4 Operating Procedure

8.4.1 Initial Preparation For Use

1. Connect the appropriate probe tip to the Readout Assembly with the captive locking nut. The short, non-telescoping probe tip is recommended for most applications. The telescoping probe is appropriate for broad, sweeping area wide surveys. Ensure that the probe is seated firmly in the Readout Assembly.

2. Connect the air sample hose to the Side Pack Assembly. The nut should be tightened by hand and then secured with at most an eighth to a quarter turn using a small crescent wrench. DO NOT OVER TIGHTEN. Connect umbilical cord. 1

3. Hydrogen fueling should be accomplished using the Foxboro high pressure filling hose. See Section 8.6.1 below.

4. Move INSTR/BATT Test Switch to the BATT position and verify that the battery is charged as indicated on the readout meter.

8.4.2 Turn On Procedure

1. Move the INSTR SWITCH to ON and allow several minutes for warm up.

2. Move the CALIBRATE Switch to x1 and adjust the meter reading to or slightly above zero with the CALIBRATE ADJUST (zero) .

3. Turn the PUMP Switch to ON and observe the SAMPLE FLOW RATE Indicator. Flow rate should be approximately 2 units.

4. Open H2 TANK VALVE several turns and observe the reading on the H2 TANK PRESSURE Indicator. The tank pressure should be at or above 1800 psi for a full day of operation. Note that the instrument will continue to operate down to approximately 200 psi of hydrogen if it is not turned off, but will become increasingly difficult to start (relight) below 500 psi.

5. Open H2 SUPPLY VALVE 1/2 to 1 turn and observe the reading on the H2 SUPPLY PRESSURE indicator. Depending on the GC column in use, this reading should be between 9 and 13 psi, longer columns will require higher H_2 supply pressures.

6. Confirm that the meter is still reading approximately zero.

7. Depress ignition button and hold. The readout needle should "peg" and there will be a slight "pop" as the hydrogen ignites. Immediately after ignition, release the ignition button. If burner does not ignite, let instrument run for several seconds and try again. Allow several minutes for the instrument to stabilize after ignition. If necessary, re-zero the instrument with the CALIBRATE ADJUST (zero) Knob. Do not hold ignition button down for more than five seconds.

8. Move instrument to an area that is representative of clean air. Move the CALIBRATE Switch to x1 and adjust the meter to read 0-1 ppm with the CALIBRATE ADJUST (zero) Knob.

9. Use of the alarm is not recommended. However, if the alarm level is to be used, set the read out to the desired alarm level using the zero adjust knob. Be sure the alarm volume control is turned up to an audible level and adjust the alarm level adjustment knob on the back of the read-out assembly until the alarm just begins to sound to the level indicated on the read-out assembly. Rezero the read-out assembly using the zero adjust knob.

10. The instrument is now ready for calibration check as described in Section 8.3, and use as described below.

8.4.3 Operating Procedure in the Survey Mode

1. Set the CALIBRATE Switch to the desired range. Using one hand operation, survey the areas of interest while observing the meter. For ease of operation, carry the Side Pack Assembly positioned on the side opposite the hand which holds the Probe/Readout Assembly. Always keep the cover on the instrument when using it for field measurements in the survey mode. Once the instrument is properly zeroed and calibrated the operator will require access to the controls only occasionally to correct zero drift or to change sensitivity scales in the event of off-scale readings. This can be easily accomplished even with the cover on the instrument once the user becomes familiar with the location of the cover latches and controls. For broad survey outdoors, the pickup fixture should be positioned several feet above ground level. When making quantitative readings or pinpointing areas of contamination, the pickup fixture should be positioned at the point of interest (but not actually in the mud, waste, water, etc.).

2. When organic gases are detected, the meter pointer will move upscale.

3. If the nature of the organic vapors being detected in unknown, nothing can be said about the exact concentration or nature of the contaminants other than that the meter reads "X ppm" relative to methane (if the instrument is, in fact, calibrated to methane). If, on the other hand, the identity of the compound being monitored is known, it can be quantified by calibrating the instrument directly to that compound as discussed in 8.3.3 above, or the actual concentration may be estimated by calibrating the instrument to methane and incorporating the relative response factors (relative to methane) identified in Table 1. For example, Table 1 indicates that the response of trichloroethylene relative to methane is 70%. If the OVA is calibrated to methane and reads 70 ppm in an atmosphere known to be contaminated with trichoroethylene, the 70 ppm reading is only 70% of the actual concentration of trichloroethylene. The actual concentration is approximately 100 ppm. 1

4. If the readout drifts to a negative (below zero) value, use the adjust knob to bring the reading back to zero and verify that the instrument is still "lit" by very briefly holding a permanent felt tip marker in front of the probe. Introduce the marker and pull it away. Do not hold it to the probe until you observe a response. There will be a slight "lag time." If the pen is held to the probe tip until a response is observed it may "load up" (contaminate) the instrument plumbing. Flame-out may result from sampling a gas mixture that is above the lower explosive level. If this is the case, it will be necessary to relight the instrument.

8.4.4 Operating Procedure in the GC Mode

8.4.4.1 General Description

The OVA with the available GC option has two modes of operation. With the Sample Inject Valve in the "out" position the instrument is measuring total organic vapors in the survey mode as described in Section 8.4.3. The OVA is in the GC mode of operation when a sample is injected into the GC system and transported through the GC column by depressing the "Sample Inject" Valve. In the GC mode the OVA can be utilized for analysis of specific organic vapors, but will not give a continuous direct read-out of sample air. A strip chart recorder should be used when operating in the GC mode as described in Section 8.4.4.6 below. The Instruction and Service Manual for the OVA-128 (Century Systems 1985) should be reviewed for a more detailed description of the general operating principles and theory for operation in the GC mode. Troubleshooting tips are provided in Table 2 of this procedure.

8.4.4.2 Sample Flow

The flow paths of the hydrogen fuel, sample air, and GC injected sample aliquot through the OVA in the two modes of operation are shown in Figure 2. It is well worth the user's time to carefully study and understand Figure 2. By doing so the user will gain a much clearer understanding of how the instrument works and much greater insight into potential causes and solutions of problems commonly encountered in the field. In the GC mode the hydrogen acts as a carrier gas. The recommended hydrogen flow rate is 12 cc/min. for proper flame ionization detector (FID) operation and as a standard flow rate for generating GC reference/calibration data. Restrictions to hydrogen flow will vary with the length and type of packing of the GC column. The hydrogen flow rate is factory set at 12 cc/min. with a Century standard 24 inch long column, and normally no adjustment needs to be made when using columns from three (3) inches to four (4) feet long. Adjustment may be required if and when precisely controlled analysis is being conducted or when the hydrogen flow is too low to keep the flame burning. The hydrogen flow rate is adjusted by varying the hydrogen Supply Pressure. The pressure is changed by adjusting the set screw in the bonnet of the low pressure regulator, accessible by removing the battery pack from the instrument panel. The sample air flow in the OVA is not adjustable and is nominally 1.0 liter/min. This flow rate should remain relatively constant.

8.4.4.3 Column Selection

You should have at least two different columns available when using the OVA in the GC mode. Appropriate columns should be determined prior to going into the field by the Project Manager by referring to the chromatographic column selection guide provided with the Century OVA and in Table 1. This table should be referred to when selecting suitable columns and to determine approximate retention times for a variety of volatile organic compounds. In the interest of time and ideal peak configurations, columns should be selected such that the compounds of interest will elute ("come out") within a range of 15 seconds to 3-4 minutes. It is possible that a single sample may contain two or more compounds of interest which have vastly different retention times on a particular column (i.e., fluorotrichloromethane and 1,1,2,2 tetrachloroethane on a T-8 column). In this case it may be desirable to select two columns, one of which is most appropriate for each compound. It is also possible that two or more compounds may have nearly identical retention times on a given column and be difficult or impossible to distinguish on one column. If there is any question of the identity of compounds of interest that are detected, the identification should be verified by comparison of retention times to calibration standards using two different columns.

8.4.4.4 Connecting the Strip Chart Recorder

A single channel strip chart recorder (Linear Model 142 or equivalent) should be used with the OVA when operating in the GC mode. When using a recorder other than the manufacturers, any patch cord with a two pin connector can be used. Connect the patch cord to the B and D pins on the OVA and to the red (+) and black (-) terminals of the recorder. If positive deflections on the read-out probe result in negative deflection of the strip chart recorder, simply switch the leads to the red and black terminals. For normal full-scale readings, the "input voltage" of the strip-chart recorder should be in the "1 volt" range. Operation of the recorder with the OVA in the GC mode is discussed in Section 8.4.4.6 below.

8.4.4.5 GC Mode Operation

In normal GC analysis, a strip chart recorder is used to record the output from the OVA as a function of time. This record (chromatogram) is utilized for interpretation of the GC data. All GC data is evaluated by comparing chromatograms of environmental samples to chromatograms of calibration standards of known concentration. The OVA should always be calibrated (as described in Step 4) prior to analysis of environmental samples, and the strip chart recorder should be properly connected and "calibrated" to the OVA.

Operation

1) Begin with the OVA running in the normal survey mode. Turn the strip chart recorder on (as described in Section 8.4.4.6) with the pen in the down position. Make sure that the chart paper drive is "ON." Two (2) cm/min is an appropriate chart speed for most compounds of interest. Prior to introducing the sample air, the following information must be entered in a separate field notebook dedicated to the GC analytical procedures for the particular project.

   Information to be entered immediately prior to each GC run:

   - GC run ID number consistent with run number recorded on strip chart print out.
   - Date and time of run.
   - Origin of sample.
   - Strip chart paper speed.
   - Value on OVA read-out probe corresponding to full-scale deflection on strip chart paper (i.e., 1, 5, 10 or whatever is appropriate and convenient).

- Initial OVA Scale Setting (x1, x10, x100) and any changes to this setting as the run progresses.

- All observations, comments, non-routine occurrences (i.e., flame-outs) which may be of interest in interpreting results later.

Introduce the sample air, either from the ambient air, a tedlar sample bag, headspace technique, etc. If detectable organics are present, the read-out probe and the strip chart recorder will give a positive "total organics" deflection in the survey mode. Push the Sample Inject Valve "in" with a fast, positive motion. This injects a 250 microliter "slug" of contaminated sample air onto the GC column and starts the GC analysis. Immediately upon pushing the sample inject button, the total organics reading on the strip chart recorder should drop to zero. This drop on the print-out is indicative of "T_0" or the GC start time. If the read-out does not drop to zero or perhaps even increases, the activated carbon in the charcoal filter is probably contaminated and must be replaced. Note: On occasion, the flame in the FID detector may go out, which is difficult to distinguish from normal operation except that the "zero" reading may be "pinned" below zero at the bottom of the chart. If you have reason to suspect a flame-out condition, immediately reignite the flame in the normal manner (provided that you have not "missed" any potential compounds of interest during the flame-out condition), and continue the analysis. A negative "air" peak typically occurs shortly after sample injection and should not be confused as flame-out.

2) The negative air peak and various positive compound peaks will be indicated by deflections on the OVA readout meter and by corresponding peaks on the strip chart recorder. Compounds which "come out" in a few seconds will appear as narrow, sharp spikes. Compounds which come out in 30 seconds to 1 or 2 minutes will be very defined "peaks." As the retention time increases, the "peaks" become less defined and begin to resemble ill defined "hills", which is one reason that it is desirable to select a column that is appropriate for the specific compounds of interest. Peak resolution of "slow eluters" may be enhanced by using a slower chart speed.

3) After the predetermined time for analysis has elapsed (normally immediately after the peak of the last compound of concern), rapidly move the Backflush Valve to it's alternate position (in or out). Leave the instrument in this condition until the backflush peak printed on the recorder returns to baseline. If no backflush peak appears, leave the GC in the backflush condition for a period of time <u>at least</u> as long as the GC analysis time, preferably slightly longer. Pull the Sample Inject Valve to the "out" position (the OVA will not operate in the survey mode or be ready for another sample injection in the GC mode unless the Sample Inject Valve has been pulled to the "out" position). Immediately upon pulling the sample inject button to the "out" position there should be a sharp spike (jump) on the strip chart recorder. At this point record "End of Good Run [INSERT NUMBER]" on the chart. If the spike is not observed when the inject button is pulled up, it is indicative of a flame-out at

To obtain a calibration point, inject a known concentration sample into the GC system and record the resulting chromatogram peak. The peak height response should be linear up to concentrations of greater than 1,000 ppm. Therefore, a single calibration point, preferably around the concentration of most concern, is normally all that is required. If temperature variations are expected standards should be repeated throughout the day.

8.4.4.6 Operating the Strip Chart Recorder

This discussion covers general set up and operation of a Linear Model 142 (or equivalent) single channel strip chart recorder. The manufacturers operating instructions should be reviewed for general theory, field calibration procedures and tips for operation and maintenance. Troubleshooting tips are provided in Table 3.

8.4.4.6.1 Set Up Procedures

The recorder should be connected to the OVA as discussed in Section 8.4.4.4 above. The procedures, detailed below must be performed to prepare the recorder for operation.

Fuse Requirements

Check unit for proper fuse (0.15A) located on back panel of the recorder.

Chart Paper Installation

1. Pull release lever on front left side of the recorder forward to pull out chart drawer for loading.

2. Insert metal tube inside new roll of chart paper. Metal insert provides proper weight/tension to supply roll as it is expended.

3. Drop new chart roll through (directly behind paper feed sprockets) with round sprocket holes on the left side. The supply chart paper roll sits on the base of the tray, directly behind the faceplate.

4. Remove tape and pull out about 12 inches of paper. Pull paper up, over sprockets, and down across the front of the face plate.

5. Push end of chart paper through slot in bottom of face plate from front to back.

6. Push spare core on take-up post. Pull chart paper under the spare core. Roll once around take-up core and tape end of paper to core.

7. Turn core to take up slack in chart.

some point in the GC run and the entire run may have to be rejected. A sample GC run print-out indicating all appropriate information is shown in Figure 4.

Calibration in the GC Mode

4) The OVA in the GC mode is intended for applications where there are a limited number of compounds of interest and the "universe" of compounds which may be expected is well characterized. For example, an ideal application of the OVA/GC would be to identify specific contaminants on a remediation site where considerable data regarding the known contaminants already exists. In other situations, it may be desirable to screen a sample for one or two "key" contaminants. For example, at a site contaminated with petroleum hydrocarbons it may be necessary to determine the presence (or absence) and concentration of benzene to establish an appropriate level of respiratory protection. Under these types of field conditions the OVA should be calibrated in the GC mode, by testing the retention times and peak area (using peak height analysis) characteristics for the specific compound(s) of interest. Calibration should be conducted using the same column(s) to be utilized and over the concentration of concern and similar temperature conditions as will be encountered during sample analysis.

When calibrating (i.e., running "standards" through) the GC the following information should be recorded on the chromatogram and/or on a calibration chart (Figure 3):

a) Column - description and serial number as applicable

b) Temperature - column temperature, normally room or ambient

c) Chart Speed - distance/unit time

d) Sample concentration - ppm for each compound

e) Recorder Scaling - ppm per unit of deflection

f) Range - range of OVA, i.e., x1, x10, x100

g) OVA type and serial number

h) Date and sample inject time

i) Backflush time

8. Close drawer by pushing on center of chart. DO NOT push release lever.

9. Turn on chart drive at 10 cm/min for several seconds to take up slack.

Pen Installation

1. Remove the pen cap and store it where it can be easily found. Pens can be left uncapped for long periods of time, but greater pen life results from capping when not in use.

2. Lift marker scale (on front of recorder) by pushing up on center of scale; the scale should pivot 90° and raise the pen carriage.

3. Insert pen in carriage until it stops.

4. Push lever on right side of scale plate down to drop the pen onto the chart, and return scale to normal position.

Power Requirements

The Model 142 is designed to operate from a self-contained, rechargeable, 12 VDC, "sealed" lead acid battery. The battery will be charged when operating from 115 or 230 VAC, 50/60/400 Hz, 1 0. Be sure to set the conversion slide switch located on the back panel to match the correct line voltage. Note that the unit will run from AC power or the battery pack but actually always runs off of the batteries. The AC power only serves to charge the batteries continuously during use. The batteries must be sufficiently charged to use the instrument even off of external AC power.

8.4.4.6.2 Initial Recording/Calibration

With chart paper and pen installed and recorder plugged into proper AC source (or in DC battery operation) perform the following steps for routine recording:

1. Turn power switch on and chart switch off. On Model 142 the power light is on when the battery is charging and off when the battery is charged.

2. Set input scale to desired range (normally 1 volt) and attenuator to Cal position.

3. "Zero" readout on OVA with "Adjust" knob. 1

4. Push zero toggle switch on strip chart recorder.

5. Zero pen to left side of chart using zero control knob while holding down zero toggle switch.

6. Release zero toggle switch and apply input signal by increasing the OVA read-out with the "adjust" knob.

7. Adjust the attenuator knob on strip chart recorder until strip chart readout corresponds to OVA probe readout. Select a full scale deflection value on the strip chart recorder which will best display the anticipated OVA readout levels, i.e., if you don't expect any readings greater than 5 ppm, set up the strip chart such that full chart deflection corresponds to 5 ppm on the OVA. 1

8. Document the OVA readout value (i.e., 1, 5, 10) which corresponds to full chart deflection on the strip chart recorder.

9. Turn chart speed on and set chart drive for desired speed (normally 2 cm/min).

8.4.4.7 Interpretation of Results

Interpretation of the recorded chromatogram is always based on the predetermined calibration data discussed in Section 8.4.4.5. The compound(s) detected in the sample chromatogram may be tentatively identified by comparing the retention times of the observed peak(s) with those of the calibration standards. The compounds can be quantified based on the height of the peak in relation to the peak heights of the calibration standards of known concentration. Caution should be exercised in interpreting results. GC results cannot be considered positive identification with complete certainty. However, since the OVA is normally used in the GC mode when some of the compounds of interest are known, the results can be considered fairly reliable when standards of those compounds are used for calibration. 1

8.4.5 Shut Down Procedure

1. Close H_2 SUPPLY VALVE.

2. Close H_2 TANK VALVE.

3. Move INSTR Switch to OFF.

4. Wait 5 seconds and move PUMP Switch to OFF. INSTRUMENT IS NOW IN A SHUTDOWN CONFIGURATION.

8.5 Troubleshooting

The following activities are the responsibility of the Task Leader and Field Technicians to ensure that the OVA is functioning properly:

- Calibration procedure
- Filter replacements

Tables 2 shows instrument malfunctions that may occur in the field and the corresponding corrective action or remedy. Refer also to Section 8.6 below.

8.6 Maintenance Procedures

The following components of the OVA require maintenance. Maintenance personnel should be thoroughly familiar with instrument operation before performing maintenance.

8.6.1 Fuel Refilling

1. The instrument should be completely shut down during hydrogen tank refilling operations. The refilling should be done in a ventilated area. There should be no potential ignition sources or flame in the area.

2. The filling hose should be purged with clean H_2 prior to filling the instrument tank.

3. Connect the hose to the refill connection on the Side Pack Assembly. 1

4. Open the hydrogen supply bottle valve slightly. Open the REFILL VALVE and the H_2 TANK VALVE on the instrument panel and place the FILL/BLEED Valve on the filling hose assembly in the FILL position. The pressure in the instrument tank will now be indicated on the H_2 TANK PRESSURE indicator.

5. After the instrument fuel tank is filled, shut off the REFILL VALVE on the panel, the FILL/BLEED Valve on the filling hose assembly and the hydrogen supply bottle valve.

6. The hydrogen trapped in the hose should now be bled off to atmospheric pressure. CAUTION should be used in this operation as described in Step (7) below, since the hose will contain a significant amount of hydrogen at high pressure.

7. The hose is bled by turning the FILL/BLEED Valve on the filling hose assembly to the BLEED position. After the hose is bled down to atmospheric pressure, the FILL/BLEED Valve should be turned to the FILL position to allow the hydrogen trapped in the connection fittings to go into the hose assembly. Then, again, turn the

FILL/BLEED Valve to the BLEED position and exhaust the trapped hydrogen. Then turn the FILL/BLEED Valve to OFF to keep the hydrogen at one atmosphere in the hose so that at the time of the next filling there will be no air trapped in the filling line.

8. Close the H_2 Tank Valve.

9. With the H_2 TANK VALVE and the H_2 SUPPLY VALVE closed, a small amount of H_2 at high pressure will be present in the regulators and plumbing. As a leak check, observe the H_2 TANK PRESSURE indicator while the remainder of the system is shut down and ensure that the pressure indication does not go down rapidly, indicating a significant leak. If it does decrease rapidly (greater than 350 PSIG/hr.), there is a significant leak in the H_2 supply system.

8.6.2 Battery Recharging

1. Plug charger BNC connector into mating connector on battery cover and insert AC plug into 115 VAC wall outlet. Never charge in a hazardous area or environment.

2. Move the battery charger switch to the ON position. The light above the switch button should illuminate.

3. Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the right when charging. When fully charged, the pointer will be in line with "charged" marker above the scale.

4. Approximately one hour of charging time is required for each hour of operation. However, an overnight charge is recommended. The charger can be left on indefinitely without damaging the batteries. When finished, move the battery charger switch to OFF and disconnect the Side Pack Assembly.

8.6.3 Primary Filter

This filter is located behind the sample inlet connector on the Side Pack Assembly and is removed for cleaning by using a small adjustable wrench, or a thin wall socket to unscrew the Fitting Assembly. The porous stainless filter cup can be cleaned by blowing out or washing in a solvent. If a solvent is used, care should be taken to ensure that all solvent is removed by blowing out or heating the filter.

8.6.4 Particle Filter

A particle filter is located in each pickup fixture. The filter should be changed or cleaned, depending upon the OVA model number.

8.6.5 Mixer/Burner Assembly Filter

Another porous metal particle filter is incorporated in the Mixer/Burner Assembly which screws into the Preamp Assembly. This filter should not become contaminated under normal conditions but can be cleaned or the assembly replaced if necessary.

8.6.6 Exhaust Flame Arrestor

A porous metal flame arrestor is located in the exhaust port of the detector chamber. This filter may be cleaned, if required, by removing the exhaust port from the Preamp Assembly. Clean the filter with a solvent or detergent but ensure that it is dry and any solvent is completely baked out at 120° F before installation.

8.6.7 Seal Maintenance

After some time, the teflon washers under each valve packing nut can "cold flow" and allow hydrogen to leak. This leakage can usually be stopped by tightening the compression nut (adapter). This compression nut is against soft material and only a small amount of force is necessary to sufficiently compress the teflon washers. If after tightening, leakage still occurs, it is advisable to replace the two teflon washers. The replacement procedure is outlined in the Users Manual for the OVA.

8.6.8 Refiller Valve Packing Adjustment

Adjustment for the valve on the refiller can be made by loosening the set screws with a 3/32" hex key, so that the handle turns freely on the stem. Insert two 3/32" hex keys through the holes provided in the handle and turn until they engage the holes in the packing adjuster. Then tighten the packing by turning the handle.

8.6.9 Air Sampling System Maintenance

A potential problem associated with the OVA is that leaks can develop in the air sample pumping system. The OVA is equipped with a flow gauge, which provides a method to check for air leaks. To isolate a leak, remove parts of the air-sampling system, one at a time, and recheck for leaks.

8.6.10 Contamination Control and Maintenance

If instrument readings fluctuate widely either the GC column or the hydrogen fuel is contaminated. Try a replacement column or try using a heating pad to heat the column while the instrument is running. It may take an hour or longer but many times contamination on the column can be flushed out. If it is determined that the column is not the problem, the hydrogen fuel tank in the instrument is probably contaminated. Empty the hydrogen fuel tank through the refill valve. Purge the hydrogen refill hose with (hopefully) clean hydrogen from the source and flush out (i.e., refill and drain) the H_2 tank several times. Refill the tank. If the problem still exists either your fuel source is contaminated, the instrument is contaminated internally, or both. The OVA should be returned to the equipment manager and/or the manufacturer for maintenance.

8.6.11 Pump Stops Running

The pump may periodically "hang up" and stop running. It is located in the lower right hand corner of the instrument immediately below the rotameter (air flow meter). Unscrew the lower right hand case fastening screw and gently pry up the corner of the face plate. With the electronics and pump switches "on" insert a flat dull utensil (popsicle stick is ideal) into the case and give the pump a "push." It should start immediately.

8.6.12 Does Not Return to Zero When GC Inject is Pushed

If the readout fails to go to zero or increases upon hitting the GC inject button, the carbon filter bed is probably contaminated. Remove the filter tube from the instrument and pull out (DO NOT TWIST OR USE PLIERS) the end cap. Discard the old charcoal. Rinse the tube with acetone and distilled water and allow it to dry completely. It can be dried in an oven at 120°-140°F. Fill with fresh activated carbon (fish tank filter carbon will do or in a pinch open up a fresh organic vapor respirator cartridge) and replace filter on instrument.

8.6.13 Factory Maintenance

To ensure continuous trouble-free operation, Century recommends a periodic factory maintenance overhaul and recalibration every six to nine months.

9. RECORDS

Calibration of the OVA and air monitoring results are documented using the Air Monitoring Data Sheet (Exhibit A). In addition, when used in the GC mode a calibration chart (Figure 3) should be prepared and chromatograms kept of all calibration and environmental samples as described in Section 8.4.4.5.

10.0 PROCEDURE ALTERATION CHECKLIST

Variation from established procedure requirements may be necessary due to unique circumstances encountered on individual projects. All variations from established procedures shall be documented on Procedure Alteration Checklists (Exhibit B) and reviewed by the Project Manger and the QA Manager.

The Project Manager may authorize individual Task Leaders to initiate variations as necessary. If practical, the request for variation shall be reviewed by the Project Manager and the QA Manager prior to implementation. If prior review is not possible, the variation may be implemented immediately at the direction of the Task Leader, provided that the Project Manager is notified of the variation within 24 hours of implementation, and the Procedure Alteration Checklist is forwarded to the Project Manger and QA Manager for review within 2 working days of implementation. If the variation is unacceptable to either reviewer, the activity shall be reperformed or action shall be taken as indicated in the Comments section of the Checklist.

All completed Procedure Alteration Checklists shall be maintained in project records.

FIGURE 1

GAS HANDLING SYSTEM

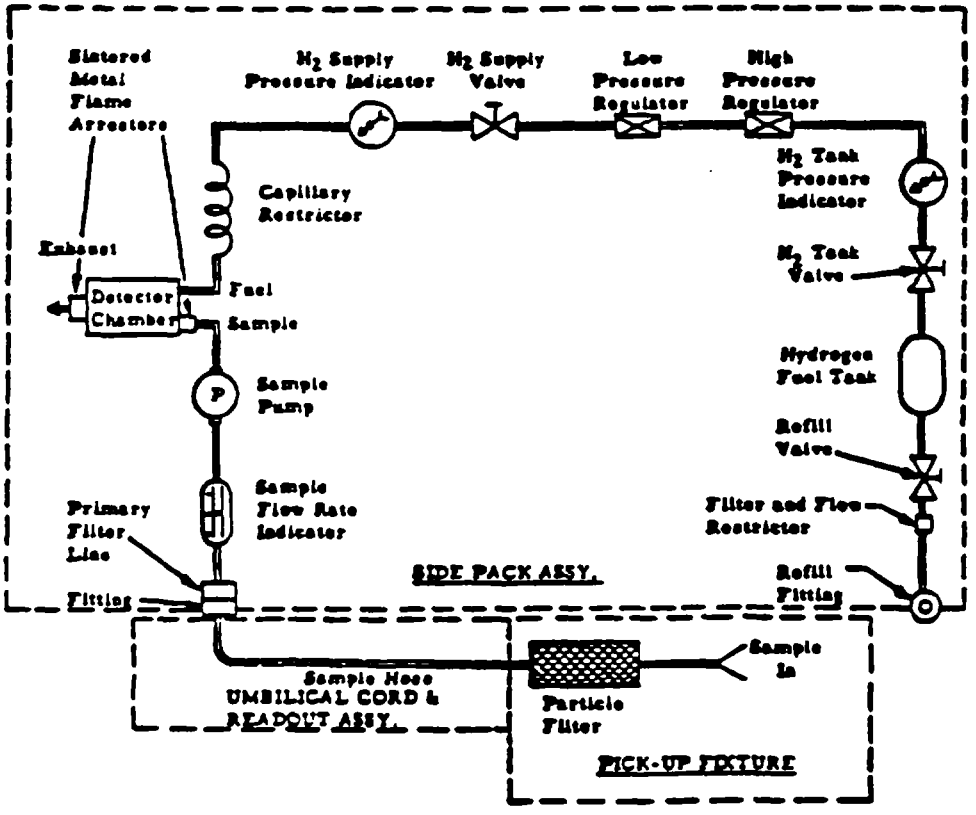


FIGURE 3 EXAMPLE CALIBRATION CHART

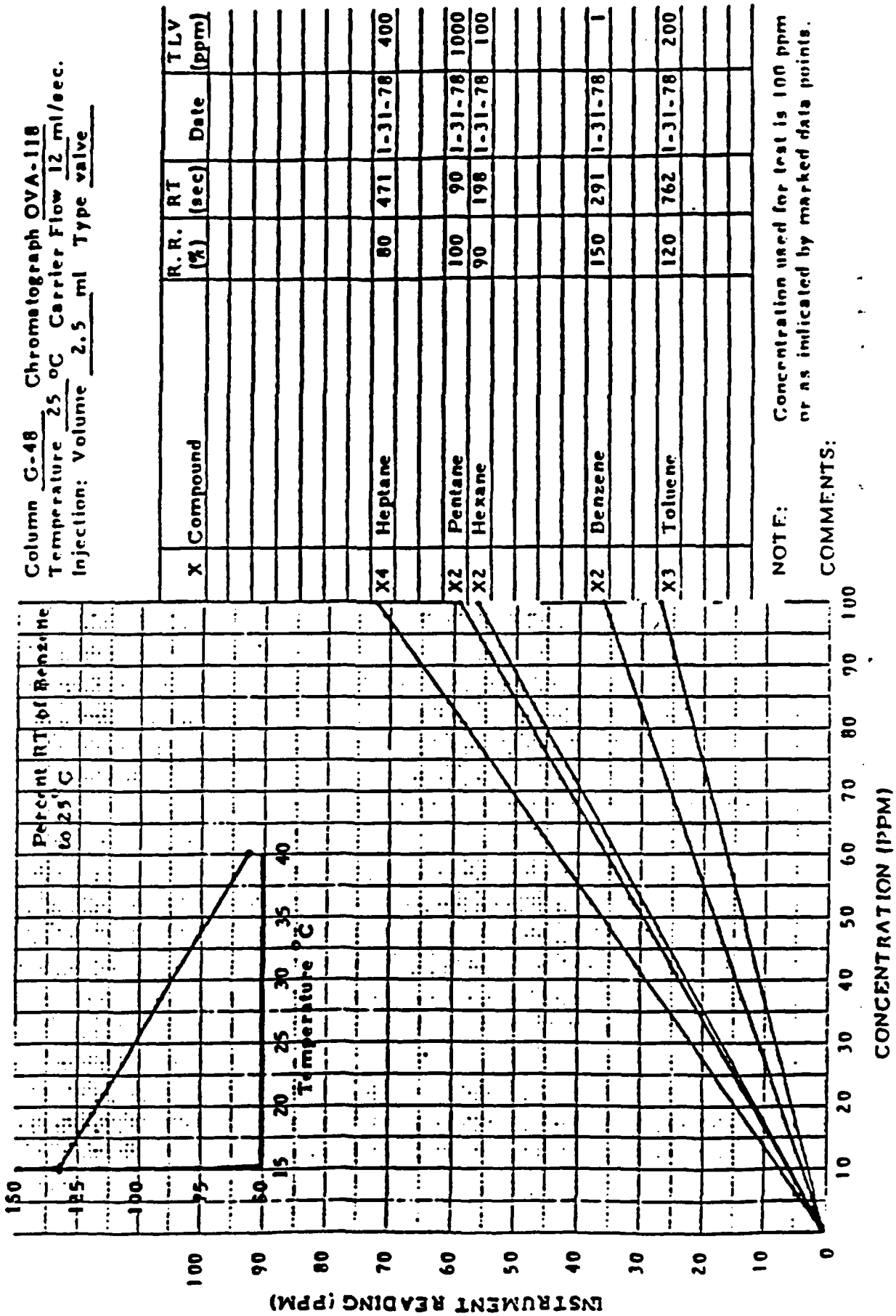


EXHIBIT A

TP-2.3-2

Golder Associates Inc.

AIR MONITORING DATA SHEET

Date _____ Sheet ____ of ____

Job Name _____ Job Number _____

Location _____

Time In _____ Time Out _____ Weather _____ Temp. _____ Wind D. _____ V _____

Instrument Type _____ Serial No. _____

Calibration Gas and Concentration _____ Instrument Reading _____ Span/Gain/RF Setting _____

** If more than one instrument is used, document calibration procedures and results for each additional instrument in recommendations section below and indicate the instrument used (eg. OVA, 361, OVM, etc.) for each observation.*

Time	Station	Instr.*	Reading	Procedure/Observations/Comments

Recommendations

_____

_____

_____

_____

_____

Printed Name _____ Signature _____

TABLE 1

CHROMATOGRAPHIC COLUMN SELECTION GUIDE

COMPOUND	Maximum Allowable Exposure (8 Hour Weighted Average) PPM	Relative Response (%)	Column Temperature (°C)	Column Packing (Retention Time in Minutes) 0.9	0.9	1.9
Acetone	1000	60	0	0 37	0 27	1 50
			40	0 20	0 15	0 30
Acetonitrile	40	70	0	0 40	0 30	5 24
			40	0.20	0 18	1 15
Acrylonitrile	2	70	0	0 35	0 30	3 45
			40	0 19	0 17	0 51
Allyl Alcohol	2	30	0	1:15	0.44	11 45
			40	0.37	0.26	1 35
Allyl Chloride	1	50	0	0 16	0.28	0 31
			40	0:08	0 16	0 15
Benzene	10	150	0	0:50	1:19	1 43
			40	0.22	0.25	0 32
2-Bromo-2-chloro-1,1,1-trifluoroethane (Halothane)	—	45	0	0:37	0.35	0 58
			40	0:17	0:14	0 19
Bromoethane	200	75	0	0:15	0 26	0 23
			40	0.05	0 15	0 14
1-Bromopropane	—	75	0	0 21	0 58	0 40
			40	0.08	0 22	0 15
2-Butane	—	60	0	0.15	0 15	0 10
			40	—	—	—
n-Butanol	100	50	0	4 10	2 20	15 07
			40	0 24	0:53	1 44
2-Butanol	150	65	0	1:45	0:55	6 07
			40	0.24	0:24	0 53
n-Butyl Acetate	150	80	0	4:15	7:30	11 40
			40	0:50	1:14	1.31
n-Butyl Acrylate	—	60	0	—	—	—
			40	2:30	2:15	2 40
2-Butyl Acrylate	—	70	0	—	—	—
			40	1:22	1:45	1:46
n-Butyl Formate	—	50	0	2:15	2:57	4:40
			40	0:28	0:34	0 49
2-Butyl Formate	—	60	0	1:22	2:00	3 31
			40	0.20	0.26	0.37
n-Butyl Methacrylate	—	60	0	—	—	—
			40	4:03	5:46	5.10
2-Butyl Methacrylate	—	80	0	—	—	—
			40	2:46	3:40	3.34
Carbon Tetrachloride	10	10	0	0:20	1:24	0 37
			40	0:10	0.25	0 17
Chlorobenzene	75	200	0	5:45	8:00	11:20
			40	1:08	1:24	1:35
Chlorodifluoromethane (Freon 22)	1000	40	0	0:11	0:11	0 15
			40	—	—	—
Chloroform	50	65	0	0:55	0:57	2.00
			40	0:20	0:20	0.31
1-Chloropropane	—	75	0	0:16	0:31	0:23
			40	0:05	0:16	0:14
2-Chloropropane	—	90	0	0.15	0:23	0 18
			40	0:05	0.05	0 05
2-Chloro-1,1,2-trifluoroethyl difluoromethyl ether (Ethrane)	—	150	0	0.36	0.26	1 22
			40	0.13	0.12	0 19
Cumene	50	100	0	11:00	20:00	12.45
			40	2.20	3:03	2.19
Cyclohexane	300	85	0	0.36	1:25	0 19
			40	0.18	0.26	0 14
Cyclohexanone	50	100	0	18 00	12:45	—
			40	3:00	2 00	—
n-Decane	—	75	0	—	—	—
			40	2.57	6 20	1 35
O-Dichlorobenzene	50	50	0	—	—	—
			40	8:06	10.00	11 20
Dichlorodifluoromethane (Freon 12)	1000	15	0	0:10	0:11	0 12
			40	—	—	—
1,1 Dichloroethane	100	80	0	0.17	0.37	0 45
			40	0:08	0.17	0 18

TABLE 1

CHROMATOGRAPHIC COLUMN SELECTION GUIDE

COMPOUND	Maximum Allowable Exposure (8 Hour Weighted Average) PPM	Relative Response (%)	Column Temperature (°C)	Column Packing (Retention Time in Minutes)		
				[illegible]	[illegible]	[illegible]
Methyl Ethyl Ketone	200	80	0	1.00	0.50	3.30
			40	0.22	0.20	0.43
Methyl Isobutyl Ketone	100	80	0	4.20	3.15	7.30
			40	0.42	0.40	1.25
Methyl Methacrylate	100	50	0	1.41	2.22	4.08
			40	0.27	0.31	0.55
Methyl Propyl Ketone	200	70	0	2.20	2.05	6.14
			40	0.33	0.32	0.52
Nitromethane	100	35	0	0.51	0.40	3.00
			40	0.25	0.19	1.31
1-Nitropropane	25	60	0	4.50	2.50	25.00
			40	0.46	0.41	4.05
2-Nitropropane	25	70	0	2.53	1.51	10.00
			40	[illegible]	[illegible]	[illegible]
Nonane	--	90	0	7.26	8.00	2.32
			40	1.08	2.40	0.42
Octane	500	80	0	2.47	7.39	1.07
			40	0.39	1.09	0.27
Pentane	1000	65	0	0.18	0.25	0.14
			40	0.12	0.12	0.12
Pentanol	—	40	0	12.00	6.00	20.00
			40	2.44	1.17	3.36
Propane	1000	80	0	0.05	0.11	0.05
			40	—	—	—
n-Propanol	200	40	0	2.50	1.00	6.30
			40	0.35	0.30	1.05
2-Propanol	400	65	0	1.13	0.30	3.43
			40	0.25	0.20	0.38
n-Propyl Acetate	200	75	0	2.04	2.52	5.52
			40	0.30	0.36	0.51
n-Propyl Ether	—	65	0	1.37	2.05	1.15
			40	0.43	0.29	0.18
n-Propyl Formate	—	50	0	0.46	1.07	2.10
			40	0.17	0.18	0.27
Pyridine	5	128	0	8.00	4.15	—
			40	—	1.26	—
Styrene	100	85	0	20.00	25.00	28.00
			40	2.06	2.26	2.39
1,1,1,2-Tetrachloroethane	—	100	0	8.25	7.48	10.00
			40	1.16	1.27	1.37
1,1,2,2-Tetrachloroethane	5	100	0	32.00	14.00	50.00
			40	4.09	2.37	7.51
Tetrachloroethylene	100	70	0	3.00	5.45	2.10
			40	0.41	1.06	0.33
Tetrahydrofuran	200	40	0	1.05	1.05	1.45
			40	0.23	0.23	0.30
Toluene	200	110	0	2.30	4.05	4.30
			40	0.38	0.47	0.53
1,1,1 Trichloroethane	350	105	0	0.31	1.10	0.47
			40	0.15	0.23	0.20
1,1,2 Trichloroethane	10	85	0	5.13	3.43	15.00
			40	0.40	0.45	2.30
Trichloroethylene	100	70	0	1.17	2.02	1.25
			40	0.23	0.28	0.28
Trichlorotrifluoroethane (Freon 113)	1000	80	0	0.15	0.30	0.16
			40	0.13	0.14	0.13
Triethylamine	25	70	0	—	2.49	—
			40	—	—	—
Vinyl Acetate	—	50	0	0.34	0.43	1.55
			40	0.15	0.17	0.30
Vinylidene Chloride	—	40	0	0.20	0.25	0.22
			40	0.13	0.14	0.14
m-Xylene	100	111	0	2.39	12.03	8.31
			40	0.39	1.42	1.17
o-Xylene	100	116	0	3.29	15.07	8.40
			40	0.48	1.58	1.45
p-Xylene	100	116	0	2.46	12.25	8.23
			40	0.39	1.42	1.19

TABLE 2

TROUBLESHOOTING

Trouble	Troubleshooting Procedures	Remedy
1. Low Sample flow rate on flow indicator. Nominally 2 units on flow gauge (See also 6 below).	a) Check the primary filter in sidepack and particle filters in the pickup assembly.	Replace or clean filter if clogged.
	b) Determine assembly containing restriction by process of elimination, i.e., remove probe, remove readout assembly, remove primary filter.	Investigate the assembly containing this restriction to determine cause of blockage. Clean or replace as required.
	c) If the restriction is in the Side Pack Assembly, further isolate by disconnecting the sample flow tubing at various points, i.e., pump output, chamber input, etc. NOTE: The inherent restrictions due to length of sample line, flame arrestors, etc., must be taken into account when trouble shooting.	If in the detector chamber, remove and clean or replace porous metal flame arrestors. If pump is found to be the problem, remove and clean or replace. Return to the equipment manager for repair.
2. H_2 flame will not light.	a) Check sample flow rate (see 1 above).	If sample flow rate is low, follow procedure 1 above.
	b) Check battery. If charge is low the instrument will often not light.	If battery is low, recharge. Once lit, the OVA will continue to run on a low battery but will not light. If the battery is not severely discharged, the charger can be hooked up to light the flame, then the instrument can be removed from the charger used until the battery is to low to run the pump.
	c) Check igniter by removing the chamber exhaust port and observing the glow when the IGNITE button is depressed.	If igniter does not light up, replace the igniter element. (The small "spark plug" that screws into the exhaust chamber.) If igniter still does not light, check the battery and wiring.
	d) Check for rated H_2 Supply Pressure. (Listed on calibration plate on pump bracket).	If low, remove battery pack and adjust to proper level by turning the allen wrench adjustment on the low pressure regulator cap.
	e) Check H_2 tank pressure gauge. OVA will run below 500 psi but becomes increasingly difficult to light.	Refill with H_2.

TABLE 2 (Cont.)

TROUBLESHOOTING

Trouble	Troubleshooting Procedures	Remedy
3) Ambient background reading in clean environment is too high.	a) Check for contamination in charcoal filter assembly. This can be detected if ambient reading increases when going in to the chromatographic mode.	Replace activated charcoal in charcoal filter assembly.
	b) Check for contamination in column.	Replace or clean column.
	c) Check for contamination in column valve assembly.	Remove valve stems and wipe with clean lint-free cloth. Heat valve assembly during operation to vaporize and remove contaminants.
	d) Check for contamination in H_2 fuel.	If H_2 fuel supply is available, drain H_2 tank. Bleed H_2 supply hose with clean H_2. Refill, purge and refill H_2 tank with ultrapure H_2. Relight OVA and check ambient readings.
4) Flame-out when operating either by-pass or GC inject valves.	a) Ensure valves are being operated with a quick, positive motion.	Operate valve with positive motion.
	b) Either H_2 or air may be leaking around one or more of the valve quad rings. Assess by tests and "O" ring inspection.	Remove stems and lightly coat with silicone grease, only on contact surface of the "O" ring. Wipe off excess (do not remove quad rings).
	c) Damaged or worn quad rings causing leak.	Return to equipment manage for repair.
5) Excessive peak tailing.	a) Change or clean GC column and see if problem disappears.	Ensure columns are clean prior to use. If one of a same type of column tails worse than others, repack the column or discard.
	b) Inspect GC valves for excessive silicone grease or contamination.	Excessively lubricant or foreign matter in the valve assembly can cause excessive tailing. Clean valve assemblies and lightly relubricate as required. Lubricant should be put only on the outside contact surface of the "O" right. Do not get grease into "O" ring grooves.

INSTRUCTION MANUAL S/N 801468

MODEL PI 101

Portable Photoionization Analyzer

©HNU Systems, Inc. 1986

TABLE 2-1

CONTROLS

Name	Position	Function
Function Switch	---	Controls the operation of the analyzer
	OFF	All operations OFF
	BATT (battery check)	Checks the condition of the battery. If the meter needle is in the green arc, the battery is charged. If not the battery should be recharged. Charging can be done in any position, best in OFF; see directions on charger.
	STANDBY	All electronics ON, ultraviolet (UV) light source OFF. This position conserves power and extends battery life. This position is used to set the analyzer zero position. (i.e. no UV light, no signal)
	0-2000	Sets range of meter at 0-2000 ppm.
	0-200	Sets range of meter at 0-200 ppm.
	0-20	Sets range of meter at 0-20 ppm.
ZERO	---	With the function switch in STANDBY position, this potentiometer is used to adjust the reading to zero.

NOTE: See Figure 2-1 for locations.

n. The analyzer is now operational.

o. Hold the probe so that the extension is at the point where the measurement is to be made. The instrument measures the concentration by drawing the gas in at the end of the extension, through the ionization chamber, and out the handle end of the probe.

WARNING

The instrument measures gases in the vicinity of the operator and a high reading when measuring toxic or explosive gases should be cause for action for operator safety.

p. Take the reading or readings as desired taking into account that air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings. Change the ranges as required.

q. Check battery condition as required. If the Low Battery Indicator comes on, turn analyzer off and recharge.

CAUTION

Use only in an emergency with a low battery when on battery charge.

This insures that the PI 101 sees the calibration gas at atmospheric pressure and ambient temperature.

d. Usage - Generally, a gas cylinder should not be used below 200-300 psi as pressure effects could cause concentration variations. The cylinder should not be used past the recommended age of the contents as indicated by the manufacturer. In case of difficulty, verify the contents and concentration of the gas cylinder.

e. Alternate means of calibration are possible. For more information, contact the HNU Service Department.

3.3 PROBE

a. Identify the probe by the lamp label. If a question exists, disassemble the probe and inspect the lamp. The energy of the lamp is etched into the glass envelope.

b. Connect the probe to the readout assembly, making sure the red interlock switch is depressed by the ring on the connector.

c. Set the SPAN pot to the proper value for the probe being calibrated. Refer to the calibration memo accompanying the probe.

d. Check the Ionization Potential (IP) of the calibration gas to be used. The IP of the calibration gas must be at or below the IP of the lamp.

e. Proceed with the calibration as described in Section 3.4. Check the calibration memo for specific data. If any questions develop, call the HNU representative.

f. NOTE: The 11.7eV lamp has a special cleaning compound. Do not use water or any other cleaning compound with the 11.7 eV lamp. Do not interchange ion chambers, amplifier boards or lamps between probes. (See Section 5.2).

3.4 PROCEDURE

a. Battery check - Turn the function switch to BATT. The needle should be in the green region. If not, recharge the battery.

3.0 STANDARD OPERATING PROCEDURE FOR MEASURING WATER TEMPERATURE

3.1 Calibration

- Calibration of thermometers will be performed before entering the field and checked upon return to the office.

- Thermometers will be calibrated against a National Bureau of Standards (NBS) - traceable thermometer.

- The thermometer must read within 1° - 1.5° C of the NBS - traceable thermometer. If the thermometer does not read within this range and the thermometer cannot be calibrated, then it will not be used for temperature measurements and will be disposed of in an appropriate manner. If the thermometer does not read within this range and the thermometer can be calibrated, then the thermometer will be calibrated to the NBS - traceable thermometer.

- The following information is documented in the calibration logbook at the time of calibration:

  a. Date
  b. Thermometer Identification
  c. Initials
  d. Calibration Data

3.2 Procedure

- The thermometer is immersed in water until the temperature equilibrates. The temperature is read in Celsius (°C).

- Temperature data are recorded in the field notebook, and initialed and dated.

4.0 STANDARD OPERATING PROCEDURE FOR MEASURING THE pH OF WATER SAMPLES

4.1 Calibration

- Calibration of the pH meter is to be performed prior to its use each day at the end of the day, and at least every four hours.

- Recalibration must occur if: (1) the pH of the samples being measured is outside the previous calibration range, or (2) the battery is replaced.

- Two buffer calibrations bracketing the expected pH range of samples are to be performed prior to the meters use each day. Three pH buffers (4.0, 7.0, and 10.0) are read after standardization at pH of 7.0 to evaluate the linearity and electrodes. The samples and buffers are to be measured at the same temperature.

- The following information is documented in the calibration logbook at the time of calibration:

  a. Date
  b. pH meter identification
  c. Initials
  d. Calibration results using pH standards

4.2 Procedure

- The pH electrode must be kept moist.

- The electrodes must be carefully rinsed with deionized water before each measurement.

- Follow manufacturer's operating instructions.

- The pH readings are documented in the field notebook, and initialed and dated.

- The electrodes are rinsed with deionized/distilled water and the unit stored properly. The electrodes are not to be stored in tap water or deionized/distilled water.

5.0 STANDARD OPERATING PROCEDURE FOR MEASURING THE CONDUCTIVITY OF WATER SAMPLES

5.1 Calibration

- Calibration is in accordance with the manufacturer's specific directions, and the following information is documented in the calibration logbook:

  a. Date
  b. Conductivity meter identification
  c. Calibration results
  d. Initials

- Calibration is performed at the beginning and end of the day, and at least every four hours.

5.2 Procedure

- The probe is immersed in a water sample until the meter equilibrates.

- In reading the conductivity meter scale, one or more of the following may have to be considered:

  a. The reading may have to be multiplied appropriately (e.g., the reading is expressed in x1, x10, x100 scales).

  b. If the conductivity meter is not capable of compensating for temperature differences, then note that the conductance measurements are not temperature compensated and document the temperatures of the standards and samples.

  c. If the conductivity meter can be compensated for temperature, then adjust the temperature control before reading the conductance measurement.

- Conductivity measurements and any other relevant information are recorded in the field notebook, and initialed and dated.

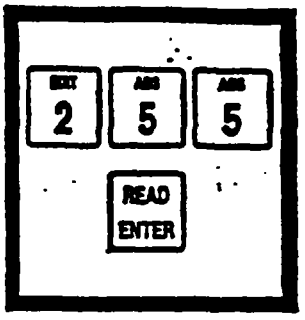
6.0 STANDARD OPERATING PROCEDURE FOR MEASURING TOTAL AND FERROUS IRON IN WATER SAMPLES

IRON, FERROUS (0 to 3.00 mg/L)

For water, wastewater and seawater

1,10 Phenanthroline Method* (Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



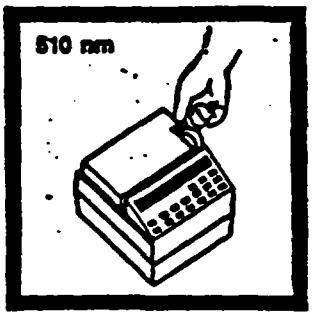
1. Enter the stored program number for ferrous iron, (Fe^{2+})– powder pillows.

Press: 2 5 5 READ/ENTER

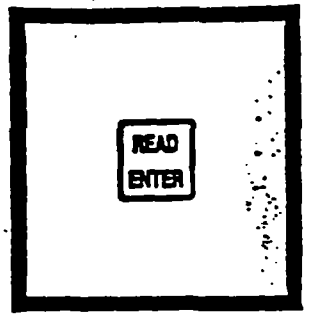
The display will show:
DIAL nm TO 510

Note: Or, use the up and down arrows to scroll the display to:
255 mg/l Fe^{2+}
and press: READ/ENTER

Note: Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.



2. Rotate the wavelength dial until the small display shows:
510 nm

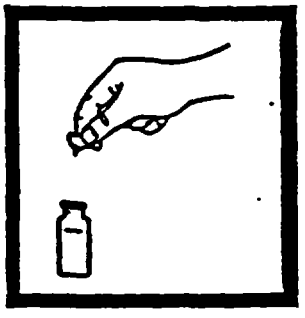


3. Press: READ/ENTER

The display will show:
mg/l Fe^{2+}

4. Fill a sample cell with 25 mL of sample.

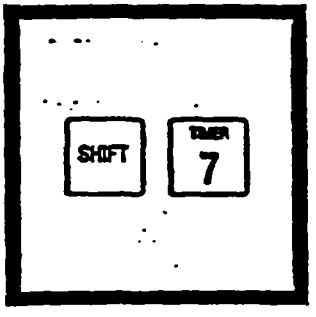
Note: For proof of accuracy, use a 1.0 mg/L ferrous iron standard solution (preparation given in the Accuracy Check) in place of the sample.



5. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

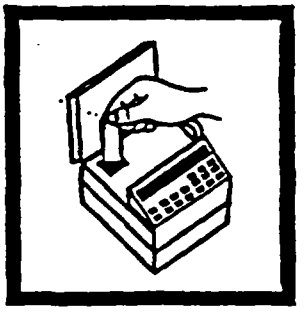
Note: An orange color will form if ferrous iron is present.

Note: Undissolved powder does not affect accuracy.



6. Press: SHIFT TIMER

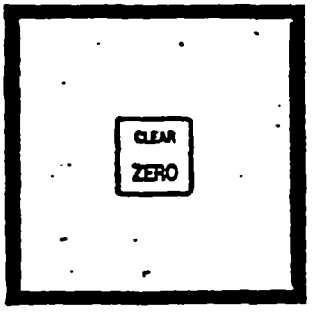
A three-minute reaction period will begin.



7. When the timer beeps, the display will show:
mg/l Fe^{2+}

Fill a second sample cell (the blank) with 25 mL of sample. Place it into the cell holder.

Note: The Pour-Thru Cell can be used with this procedure.



8. Press: ZERO

The display will show:
WAIT

then:
0.00 mg/l Fe^{2+}

ACCURACY CHECK

Prepare a ferrous iron stock solution (100 mg/L Fe) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.0 mg/L standard solution. Prepare this immediately before use.

PRECISION

In a single laboratory using an iron standard solution of 1.000 mg/L Fe^{2+} and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.006 mg/L Fe^{2+}.

In a single laboratory using a standard solution of 1.000 mg/L Fe^{2+} and two representative lots of AccuVac ampuls with the DR/2000, a single operator obtained a standard deviation of ± 0.009 mg/L Fe^{2+}.

SUMMARY OF METHOD

The 1,10 phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test. See *Chemical Procedures Explained*, Publication 7013, for more information.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
Ferrous Iron Reagent Powder Pillows	1 pillow	100/pkg	1037-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

Ferrous Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25140-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	43784-00
Beaker, 50 mL	1	each	500-41
Vial, zeroing	1	each	21228-00

OPTIONAL REAGENTS

Ferrous ammonium sulfate, hexahydrate	113 g	11256-14
Water, deionized	3.78 L	272-17

OPTIONAL APPARATUS

Flask, volumetric, 100 mL	each	547-42
Flask, volumetric, 1000 mL	each	547-53
Pipet, volumetric, 1 mL	each	515-35
Pipet filler, safety bulb	each	14651-00
Pour-Thru Cell Assembly Kit	each	45215-00

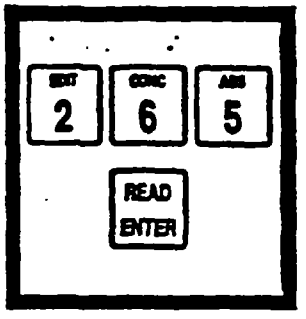
For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

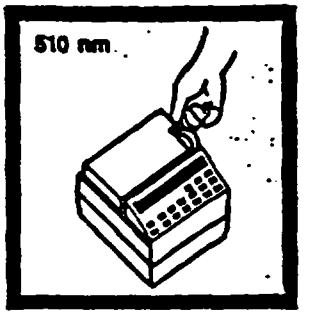
IRON, TOTAL (0 to 3.00 mg/L)

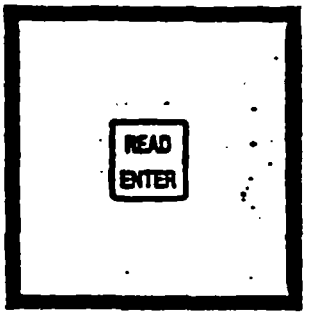
For water, wastewater and seawater

FerroVer Method* (Powder Pillows or AccuVac Ampuls), EPA Approved†—Digestion is required; see Section I.

USING POWDER PILLOWS







1. Enter the stored program number for iron (Fe), FerroVer, powder pillows.

Press: 2 6 5 READ/ENTER

The display will show:
DIAL nm TO 510

Note: Or, use the up and down arrows to scroll the display to:
265 mg/l Fe FV
and press: READ/ENTER

Note: If samples cannot be analyzed immediately, see Sampling and Storage, below. Adjust pH of stored samples before analysis.

2. Rotate the wavelength dial until the small display shows:
510 nm

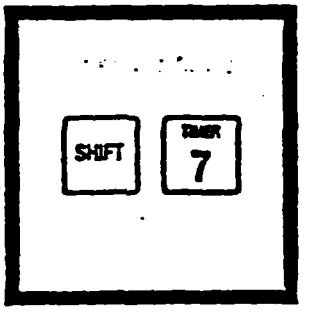
Note: Determination of total iron needs a prior digestion; use the mild, vigorous or Digesdahl digestion (Section I).

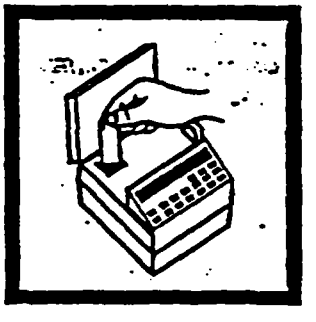
3. Press: READ/ENTER

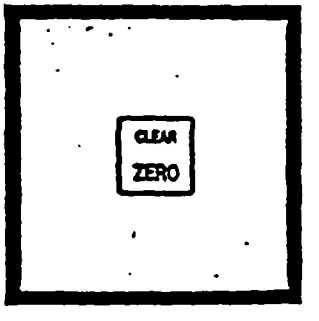
The display will show:
mg/l Fe FV

4. Fill a cell with 25 mL of sample.

Note: For proof of accuracy, use a 1.0 mg/L iron standard solution (preparation given in the Accuracy Check) in place of the sample.







5. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

Note: An orange color will form if iron is present.

Note: Accuracy is not affected by undissolved powder.

6. Press: SHIFT TIMER

A three minute reaction period will begin.

Note: Samples containing visible rust should be allowed to react at least five minutes.

7. When the timer beeps, the display will show:
mg/l Fe FV

Fill another sample cell (the blank) with 25 mL of sample. Place it into the cell holder.

Note: For turbid samples, treat the blank with one 0.2-gram scoop of Rover Rust Remover. Swirl to mix.

Note: The PourThru Cell can be used with this procedure.

8. Press: ZERO

The display will show:
WAIT

then:
0.00 mg/l Fe FV

*Adapted from Standard Methods for the Examination of Water and Wastewater
†Federal Register, 45 (126) 43459 (June 27, 1980)

SAMPLING AND STORAGE

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide before analysis. Correct the test result for volume additions; see Sampling and Storage, Volume Additions (Section I) for more information.

If only dissolved iron is to be determined, filter the sample before acid addition using the labware listed under Optional Apparatus. No acid addition is necessary if analyzing the sample immediately.

ACCURACY CHECK

a) Snap the neck off an Iron Voluette Ampule Standard Solution, 50 mg/L.

b) Use the TenSette pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL water samples and mix thoroughly. (For AccuVac Ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Prepare a 1.0 mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water. Or, use the TenSette pipet to dilute 1.0 mL of the contents of an Iron Voluette Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily.

PRECISION

In a single laboratory, using a standard solution of 1.000 mg/L Fe and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.006 mg/L.

In a single laboratory, using a standard solution of 1.000 mg/L Fe and two representative lots of AccuVac ampuls with the DR/2000, a single operator obtained a standard deviation of ± 0.009 mg/L Fe.

INTERFERENCES

The following will not interfere below the levels shown:

Chloride	185,000 mg/L
Calcium	10,000 mg/L as $CaCO_3$
Magnesium	100,000 mg/L as $CaCO_3$

A large excess of iron will inhibit color development. A diluted sample should be tested if there is any doubt about the validity of a result.

FerroVer Iron Reagent Powder Pillows and AccuVac Ampuls contain a masking agent which eliminates potential interferences from copper.

Samples containing some forms of iron oxide require the mild, vigorous or Digesdahl digestion (Section I). After digestion adjust the pH to between 2.5 and 5 with ammonium hydroxide.

Samples containing large amounts of sulfide should be treated as follows in a fume hood, or well ventilated area: Add 5 mL of Hydrochloric Acid to 100 mL of sample and boil for 20 minutes. Adjust the pH to between 2.5 and 5 with 5 N Sodium Hydroxide and readjust the volume to 100 mL with deionized water. Analyze as described above.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Interferences, pH (Section I).

REAGENT STORAGE

FerroVer Reagent Powder Pillows are stable indefinitely if stored properly. A cool, dry atmosphere is recommended. The reagent can be checked by adding the contents of a pillow to about 25 mL of water containing visual rust (such as a few drops of Rust Suspension). If the orange color does not form, the reagent should be replaced.

SUMMARY OF METHOD

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample, to produce soluble ferrous iron. This reacts with the 1,10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration. See *Chemical Procedures Explained*, Publication 7013, for more information.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
FerroVer Reagent Powder Pillows	1 pillow	50/pkg	854-66

REQUIRED REAGENTS (Using AccuVac Ampuls)

FerroVer Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25070-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac vial	1	each	43784-00
Beaker, 50 mL	1	each	500-41
Vial, zeroing	1	each	21228-00

OPTIONAL REAGENTS

Ammonium hydroxide, ACS	473 mL	106-11
Hydrochloric Acid, 6 N	500 mL	884-49
Hydrochloric acid, ACS	473 mL	134-11
Iron Standard Solution, 100 mg/L	118 mL	14175-14
Iron Volumetric Ampule Standard, 50 mg/L	16/pkg	14254-10
Nitric acid, ACS	473 mL	152-11
Nitric Acid Solution, 1:1	473 mL	2540-11
RoVer Rust Remover	28 g	300-20
Rust Suspension	15 mL DB	1279-36
Sodium Hydroxide Standard Solution, 5.0 N	118 mL MDB	2450-37
Water, deionized	3.78 L	272-17

OPTIONAL APPARATUS

Cylinder, graduated, poly, 25 mL	each	1081-40
Cylinder, graduated, poly, 100 mL	each	1081-42
Filter discs, glass, 47 mm	100/pkg	2530-00
Filter holder, membrane	each	2340-00
Filter pump	each	2131-00
Flask, erlenmeyer, 250 mL	each	505-46
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Hot plate, micro	each	12067-01
pH meter, Hach One	each	43800-00
pH indicator paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, tips for 19700-01	50/pkg	21856-96
Pipet, volumetric, 1.00 mL	each	515-35
Pour-Thru Cell Assembly Kit	each	45215-00
Spoon, measuring, 0.1 g	each	511-00
Spoon, measuring, 0.2 g	each	638-00

For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

7.0 STANDARD OPERATING PROCEDURE FOR COLLECTION OF TIME WEIGHTED AVERAGE AIR SAMPLES

7.1 Purpose

This technical procedure is to be used to establish a uniform procedure for the collection of air samples for laboratory quantification of constituents.

7.2 Applicability

This technical procedure is applicable to all personnel involved with the collection of air samples.

7.3 Definitions

- Summa Passivated Cylinder: A stainless steel cylinder with an inlet valve where the surfaces which contact the sample have been conditioned to reduce reactivity of the sample.

- Vacuum Flow Regulator: A flow regulator which attaches to the Summa Cylinder and allows the air sample to enter the cylinder at a controlled rate. A vacuum gauge is attached to the flow regulator.

- Cryogenic Focusing: A method of pre-concentrating an air sample where a precise volume of the sample is pumped through a supercooled loop where the analytes are condensed and then liberated by heating into a precise volume of gas which is smaller than that of the original sample.

7.4 Discussion

This procedure is based upon USEPA Method TO-14, which is widely considered to be one of the most reliable and sensitive test methods for ambient air toxics.

7.5 Equipment and Materials

- Summa passivated cylinders (see Figure 1) equipped with vacuum flow regulators and vacuum gauges. The cylinders will have been

cleaned and evacuated, and the flow regulator will have been calibrated before and after sampling, by the analytical laboratory.

- Air ultra zero.
- Teflon or stainless steel tubing and connections to the Summa canisters.
- Shipping containers and chain-of-custody documentation (Figure 2).

7.6 Procedure

All samples should be collected at approximately the same time. Inlet valves on the Summa cylinders will have to be opened manually and will therefore vary in the exact start and stop time. However, they can be collected at approximately the same time within a window of ±2 hours.

Upon arrival at the Site, check that all necessary sampling equipment is available. Plan to deploy the samplers in early afternoon such that the shipping time to the laboratory can be minimized.

When deploying the Summa cylinders (by opening the inlet valve) note the time and the vacuum gauge reading on the cylinder chain-of-custody form. Also note a description of the sample location, any noticeable odors, and approximate meteorological conditions such as temperature, wind speed and direction, cloud cover, and precipitation in the field book. Two cylinders should be deployed at the duplicate sample station.

Vents can be sampled using the Summa cylinders by installing a hole into the vent (sample port), inserting stainless steel or Teflon tubing into the vent sufficiently upstream of the sample port (approximately 12 inches) and sealing the sample port securing the tubing. The Summa cylinder is then connected to the tubing (see 13.6.1 below).

At the end of the sampling period (usually 8 or 24 hours) record the time and vacuum gauge reading on the chain-of-custody form, close the valve, and note any changes in the site conditions in the field book. Return the cylinder to the shipping container and record the date and time of sampling on the chain-of-custody record. Collect cylinders from all stations in the same order that the cylinders were deployed.

After all chain-of-custody documentation has been completed, seal the form in the shipping container with the samples and send them to the laboratory by overnight courier. Notify the laboratory that the samples have been shipped.

7.7 Specific Procedures for Gas Vent Sampling Using Summa Passivated Canisters

The analytical laboratory will have prepared (cleaned) the Summa canisters and calibrated the inlet flow rate prior to sampling. The vacuum pressure of the canister should be checked prior to sampling to ensure that it has not leaked during transit from the laboratory to the site. Chain-of-custody documentation should also be inspected and completed in accordance with the QAPjP.

The gas vent will have a small hole drilled in the side of the riser pipe in order to measure the gas exit velocity, temperature, and humidity and to provide access for sampling. A length of Teflon on stainless steel tubing can be inserted through this hole, or through the top of the riser pipe, and connected to the Summa canister inlet using a Swagelock (or equivalent) compression fitting. The end of the tubing should extend 12 inches upstream of the vent access port which should be sealed during sampling.

The inlet to the Summa canister should be opened to begin sampling. Sampling will proceed without the need for any operator attendance. At the end of the designated sampling period (usually 8 or 24 hours depending on the size of the

canister and the sampling flow rate) the inlet valve to the canister should be closed and the vacuum pressure in the canister recorded.

The canister should be shipped to the laboratory in accordance with the chain-of-custody procedures given in the QAPjP. Special care should be used when handling and packing the canisters to prevent damage to the valves.

Notes should be kept of the date and time that sampling was initialized and terminated at each location, weather conditions such as cloud cover, temperature, humidity, precipitation (including any recent precipitation prior to sampling), and barometric pressure. All notes and paperwork should include the project name and number, the sampler's name, and the date.

7.8 References

Bernstein, D.M, and M. Lippman. Calibration of Air Sampling Instruments.

California Air Resources Board, 1986. Testing Guidelines for Active Solid Waste Disposal Sites.

U.S. Environmental Protection Agency, 1984. Characterization of Hazardous Waste Site - A Methods Manual: Volume II. Available Sampling Methods, Second Edition.

U.S. Environmental Protection Agency, 1987. A Compendium of Superfund Field Operations Methods.

U.S. Environmental Protection Agency, 1984. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.

8.0 GROUNDWATER AND SURFACE WATER SAMPLING

To ensure the collection of representative ground and surface water samples from the study area, the following procedures will be implemented for field activities conducted during the investigation.

8.1 Monitoring Well Purging

All groundwater sampling will be accomplished after the monitoring wells have been developed and allowed to stabilize. Prior to collecting samples, each well will be purged by pumping or bailing to ensure that a representative sample is obtained. Field procedures for purging monitoring wells include:

- Inspect well for effects of tampering;
- Measure inside diameter of well casing;
- Measure depth to water and total depth of well from the top of the well casing;
- Calculate the volume of water to be purged based on the height of standing water in the well and the diameter of the well casing;
- Remove at a minimum three times the calculated volume of water from the well. Wells will be evacuated two feet above the screen, if possible. If the well can be pumped or bailed dry, the well will be evacuated once and allowed to recover enough for a sample volume to be collected as soon as possible; and,
- The water level probe and evacuation equipment will be decontaminated prior to use and after purging is completed according to the protocols described in Section 7.0 of the FSP.

8.2 Monitoring Well Sample Collection

The following procedures will be used to collect samples from monitoring wells after the well has been purged:

- Remove and inspect sample containers, sample forms, and chain-of-custody forms for consistency with sample location;

- Attached a clean sample line and slowly lower a clean teflon bailer, dedicated to the sample location, into the well screen and allow it to fill with water;

- Retrieve bailer and slowly transfer the sample into the appropriate sample containers. The first sample will be poured into a clean glass beaker to measure pH, temperature, and specific conductance. Equipment operating procedures are contained in the field equipment manual. Sample containers will be filled in the following order: volatile organics, semi-volatile organics, pesticides, specialty parameters, and inorganics. The volatile organic sample container should be inverted to ensure it contains no headspace or air bubbles. All other sample containers should be filled to the top. Containers that have preservatives added to them prior to sampling should not be overfilled;

- Label sample containers with time and date of collection;

- Place sample in the appropriate shipment containers;

- Wrap bailer in aluminum foil; and,

- Replace well cap and secure the protective lid.

8.3 Surface Water Sampling

Surface water samples will be collected according to the following procedures:

- Commence sampling at the furthest downstream point and continue sampling moving upstream. Identification flags will mark each location for future sampling;

- Remove sample containers, sample forms, and chain-of-custody forms and check for consistency with sample location;

- Slowly lower an inverted, clean laboratory glass beaker or sampling bottle to mid depth of the stream and fill. Pond samples will be collected from the middle of the water column using a Kemmerer sampler; and,

- Transfer sample into appropriate sample containers.

9.0 SOIL AND SEDIMENT SAMPLING

To ensure the collection of representative soil and sediment samples, the following procedures will be implemented during all soil and sediment sample collection activities.

9.1 Soil

Surface soil samples will be collected using several different types of equipment. Unless field conditions warrant otherwise, trowel or scoop samplers will be used at sample locations, and shelby tube (for physical analysis samples) samplers will be used at all other soil/sludge sampling locations. The choice of a specific sampling device will be based upon: depth of sample collection; quantity of sample required; type of analysis; type of material being sample; and field conditions. The following describes the sample collection procedures for each piece of equipment that may be utilized.

9.1.1 Trowels and Scoops

This method provides for a fast and relatively easy means to collect disturbed samples of specified soils to a depth of six inches.

A trowel is a commonly used gardening tool for digging. It acts as a small hand held shovel. For the purpose of this project the only difference between our trowel and the garden variety will be that the trowel will be constructed of stainless steel. Trowels are ideally useful in obtaining surface soil samples from a depth of up to six inches. Trowels and scoops may be used during off-site surficial soils sample collection, or collection of sediment samples. The following technique is used in obtaining a surface soil sample:

- Using a decontaminated trowel, obtain soil from the required depth at the proper location;

- Transfer the sample into a laboratory supplied container (for chemical analysis), or into other suitable container if the sample is being obtained for other purposes than chemical analysis or being composited;
- Close container, label in indelible ink seal;
- Complete COC label for analysis desired;
- Preserve and/or refrigerate to 4°C;
- Record appropriate data and information into project log book; and,
- Decontaminate equipment prior to next use or before storage.

9.1.2 Hand Auger

A hand auger consists of a horizontal hand bar and a vertical shaft connected to stainless steel spiral blades (open or closed) which act as the auger. This tool can be used to collect disturbed or undisturbed samples depending on the type of extension. The following technique is used when using a hand auger:

- Clear the surface of any debris that may impede the auger's penetration;
- Place stainless steel hand auger perpendicular to the ground and rotate the auger while exerting pressure on the hand bar until the desired depth;
- Pull auger out of ground using the least amount of force necessary. Transfer sample into laboratory supplied container (if sample is to be used for chemical analysis) or other appropriate container (if sample was obtained for other purposes). Use a decontaminated stainless steel trowel or spoon to dislodge sample if necessary;
- Close the sample container, label in indelible ink;
- Complete COC label and specify analysis desired;
- Preserve and/or refrigerate to 4°C;

- Record appropriate data and information in project log book; and,
- Decontaminate equipment prior to next use or before storage.

9.1.3 Trier

This device may be substituted for the hand auger if a less disturbed sample is required or a core profile is desirable. It is either stainless steel or stainless steel plated and is a cylindrical tube with a section of the tube cut away enabling inspection and removal of soil where desirable. The procedure for sampling is similar to the hand auger sampling procedure:

- Place decontaminated trier perpendicular to the sampling location;
- Press down while rotating trier;
- After reaching the desired depth, rotate the trier out in the opposite direction;
- Inspect contents of trier and transfer sample to laboratory supplied containers, using a stainless steel spatula;
- Attach labels and complete chain-of-custody protocols;
- Preserve and/or refrigerate sample at 4°C;
- Record data and appropriate information in project log book; and,
- Decontaminate trier equipment prior to next use or before storage.

9.1.4 Shelby Tube

A shelby tube is a cylindrical stainless steel tube with open ends. The thin-walled tubes come in several lengths and diameters. They are of use in a variety of situations where trowels and hand augers are impractical due to a deeper sampling interval or quantity of sample needed.

Additionally, they are used in obtaining core soil samples that are sealed into the shelby tube for shipment. The core sample represents a cross-section of the subsurface soils at the specific sampling location. The soil core is also used in the analysis of physical parameters such as moisture content and Atterburg Limits. Therefore, shelby tubes are usually only used once per sampling round or event. The following technique is used when employing a shelby tube to sample soils:

- Position the shelby tube perpendicular to the surface of the soil to be sampled;
- Push the tube into the soil without twisting or disrupting the soil;
- In the event that insertion of the shelby tube into the soil is impractical, a drive shoe and weight may be used. In this event, increments of the tube will be measured and the subsequent blows of the weight will be recorded;
- After the desired depth has been obtained, the tube will be rotated in order to shear the soil core off;
- Both ends of the shelby tube will be sealed with wax or other appropriate material to preserve the soil core. The top of or driven end of the tube will be indicated on the shelby tube itself;
- The shelby tube will be labeled and receive chain-of-custody protocols;
- The shelby tube will be placed in a container for shipment; and,
- Record all appropriate data and information per sample in the project log book.

9.2 Sediment Sampling

Sediment samples can be obtained using the same methods previously described in the Soil Sampling section. The sampling tool used depends upon the location of the sediment (e.g., under a fluid, a bank of a river or creek, lake, pond, or lagoon bottom). For dryer sediments a trowel or hand auger is the most practical.

For moist sediments or those under a water surface, a trier or hand auger may be desirable. It should be noted that the hand auger and trier use is dependent upon sample depth, although extensions can be employed for greater depth. General sampling procedures for sediment are as follows:

- Identify the collection point location;
- Using a decontaminated stainless steel sampling tool, retrieve sample from upper six inches of sediment at the location;
- Transfer sample into a clean stainless steel bowl after filling the volatile organic sample containers;
- Thoroughly mix the sample in the bowl and fill the remaining sample containers in the appropriate order;
- Seal container, attach and complete label and all chain-of-custody protocols, and specify analysis required;
- Preserve and/or refrigerate at 4°C;
- Record all data and appropriate information in the project log book; and,
- Decontaminate all sampling equipment before next use and/or before storage.

9.2.1 Pond Bottom

Samples of pond bottom sediments will be collected using a Ponar[R] dredge from a small boat or by personnel wearing waders if the pond is sufficiently shallow. The dredge is a clam shell scoop constructed of galvanized steel and steel mesh screening. The dredge is activated by a counter lever system. The following procedures will be implemented when collecting samples:

- Attach a clean sample line to the dredge (i.e., nylon);
- Measure distance to bottom sediments with a weighted tape;

- Open sampler until the jaws are latched;
- Lift dredge by sample line and lower into the water. When it is approximately three feet from the bottom slow the rate of descent until the bottom is reached;
- Allow sample line to slack several inches;
- Slowly raise sampler to surface and place in a clean stainless steel bowl;
- Open dredge and transfer sediment to the appropriate sample containers using a clean stainless steel spatula filling the containers for volatile organic analysis first;
- Seal and label container, and complete chain-of-custody procedures; and,
- Record all data and appropriate information in the project log book.

VOLUME 2

SECTION II - QUALITY ASSURANCE PROJECT PLAN

REVISION #2

April 1994

Project No.: 933-6158

QUALITY ASSURANCE PROJECT PLAN
TABLE OF CONTENTS

Table of Contents i

SECTION PAGE

1.0 INTRODUCTION 1
1.1 Background Statement 1
1.2 Project Objectives and Scope 1
1.3 Facility Information 2
1.3.1 Site Location 2
1.3.2 Site Operations History 3
1.3.3 Site Regulation History 4
1.3.4 Previous Investigations 4
1.3.5 Previous Remedial Actions 5
1.4 Sample Network Design and Rationale 6
1.5 Parameters To Be Tested and Frequency 6
1.6 Data Quality Objectives 6
1.7 Project Schedule 7

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY 8

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA . 12
3.1 Level of Quality Control Effort 12
3.2 Accuracy, Precision, and Sensitivity of Analysis 13
3.3 Completeness, Representativeness and Comparability 13

4.0 SAMPLING PROCEDURES 15

5.0 SAMPLE CUSTODY 16
5.1 Field Chain of Custody Procedures 16
5.1.1 Field Procedures 16
5.1.2 Field Logbooks/Documentation 16
5.1.3 Transfer of Custody and Shipment Procedures 18
5.2 Laboratory Chain of Custody Procedures 19
5.3 Final Evidence Files Custody Procedures 19

6.0 CALIBRATION PROCEDURES AND FREQUENCY 20
6.1 Field Instruments/Equipment 20

7.0 ANALYTICAL PROCEDURES 24
7.1 Laboratory Analysis 24
7.2 Field Screening Analytical Protocols 25

TABLE OF CONTENTS (con't)

SECTION PAGE

8.0 INTERNAL QUALITY CONTROL CHECKS 26
8.1 Field Sample Collection 26
8.2 Field Measurement 26
8.3 Laboratory Analysis 26

9.0 DATA REDUCTION, VALIDATION, AND REPORTING 27
9.1 Environmental 27
9.2 Laboratory and Quality Control Reports 28

10.0 PERFORMANCE AND SYSTEM AUDIT 29

11.0 PREVENTIVE MAINTENANCE 30

12.0 SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS 31
12.1 Field Measurements 31
12.2 Laboratory Data 31
12.2.1 Precision 31
12.2.2 Accuracy 32
12.2.3 Completeness 32
12.2.4 Sensitivity 32

13.0 CORRECTIVE ACTION PROCEDURES 33

14.0 QUALITY ASSURANCE REPORTS 34

TABLE OF CONTENTS (con't)

In Order Following Page 34

LIST OF TABLES

Table 1-1 Sample Locations and Laboratory Analyses
Table 1-2 Sample Locations and Field Analysis

Table 1-3a Sample and Data Quality Objective Summary: Treatment Plant Evaluation
Table 1-3b Sample and Data Quality Objective Summary: Field Sampling
Table 1-4 Levels of Quality Assurance and Analytical Data Methodologies

Table 3-1a Performance Evaluation Sampling and Analysis Plan Summary Target Analytes, Analytical Methods, and Quality Assurance Samples: Treatment Plant Evaluation
Table 3-1b Performance Evaluation Sampling and Analysis Plan Summary Target Analytes, Analytical Methods, and Quality Assurance Samples: Field Sampling
Table 3-2 Practical Quantitation Limits for Volatile Organics
Table 3-3 Practical Quantitation Limits for Semivolatile Organics
Table 3-4 Practical Quantitation Limits for Pesticides
Table 3-5 Inorganic Estimated Instrumental Detection Limits
Table 3-6 Analytical Methods, Sample Containers, Preservation and Analytical Hold Times for Aqueous and Gaseous Samples

LIST OF FIGURES

Figure 1 Site Location Plan
Figure 2 Treatment Plant Sampling Locations
Figure 3 Removal Actions Work Plan Schedule
Figure 4 Project Organization Chart
Figure 5 Meter Calibration Record

LIST OF ATTACHMENTS

Attachment A Laboratory Standard Operating Procedures (SOPs)
Attachment B Sampling and Field Testing Procedures

1.0 INTRODUCTION

1.1 Background Statement

On November 17, 1993 Ruetgers-Nease Corporation (RNC) entered into a Administrative Order by Consent (Removal AOC) with the U.S. EPA and the Ohio EPA (OEPA) concerning removal actions to take place at the Nease Chemical Site, Salem, Ohio.

The Removal AOC "requires the Respondent to undertake and complete emergency removal activities to abate conditions which the U.S. EPA has determined may present an imminent and substantial endangerment to the public health or welfare or the environment because of an actual or threatened release of hazardous substances at the Site."

Based upon the findings and determinations of the U.S. EPA contained therein, and pursuant to Section 106(a) of CERCLA, 42 U.S.C. Section 9606(a), the Respondent is required to undertake certain removal actions/measures (as described in Paragraph 4. of the AOC) at the Nease Chemical Site.

Pursuant to Paragraphs 4a., 4b., 4c., 4d., 4e., and 4f. of the Removal AOC, and so, part of the Work Plan, Removal Actions shall be undertaken by RNC to continue to collect contaminated groundwater, commence operation and testing of an on-site leachate treatment system, continue to inspect and monitor the site, and contain and address documented leachate releases and seeps on the Nease Chemical Site, at large. In accordance, the Work Plan must and shall contain a Quality Assurance Project Plan (QAPjP).

1.2 Project Objectives and Scope

This QAPjP was developed by Golder Associates at the request of RNC to meet the requirements of the Removal Actions mandated in the November 17, 1993 Removal AOC for the Nease Chemical Site in Salem, Ohio. An objective of the Removal Actions is to gather data of adequate technical content, quality, and quantity to 1) complete a performance evaluation of the existing groundwater treatment plantto determine the ability of the treatment plant to meet the allowable discharge criteria; and, 2) define the nature of documented leachate releases, or other hydrologic or geologic materials that would provide information necessary to perform actions contained in the Removal AOC for the Nease Chemical Site, at large. The QAPjP describes the Quality Assurance Procedures associated with sampling, analysis and testing to be performed during the Removal Actions.

The following elements will be included in the work:

- Collection of aqueous samples.
- Collection of air samples.
- Collection of soil/sediment samples.

Aqueous samples will be analyzed for VOCs, SVOCs (including 3,4-dichloronitrobenzene and diphenyl sulfone), pesticides, and filtered and unfiltered metals (SW846 Methodology). Mirex, photomirex, and kepone will be analyzed utilizing GC/MS with the Pulsed Positive Negative Ion Chemical Ionization (PPNICI) Version 4.1 detection system (as per the Approved RI Work Plan, 1990). Conventional parameters such as pH, specific conductance, etc., will also be analyzed. Acute toxicity tests will be performed for both *Ceriodaphnia dubia* and *Pimephales promelas* by the 48-Hour Static Daily Non-Renewal Definitive Bioassay.

Air samples will be analyzed for VOCs (EPA Method TO-14). Specific conductance, pH, and temperature will be analyzed in the field. Iron will be analyzed in the field by a Hach Kit (or equivalent). Vapor phase VOCs will be analyzed in the field with an Organic Vapor Analyzer (OVA) (or equivalent).

Soil and/or sediment samples will be analyzed for VOCs, SVOCs (including 3,4-dichloronitrobenzene and diphenyl sulfone), pesticides, and filtered and unfiltered metals (SW846 Methodology). Mirex, photomirex, and kepone will be analyzed utilizing GC/MS with the Pulsed Positive Negative Ion Chemical Ionization (PPNICI) Version 4.1 detection system (as per the Approved RI Work Plan, 1990).

The QAPjP is anticipated to be used in conjunction with the Field Sampling Plan (FSP).

1.3 Facility Information

This section provides an overview of the operational and administrative history of the Nease Chemical Site. It describes the site location, operation and regulatory history, past investigations, and past remedial actions at the Site.

1.3.1 Site Location

The Site is located approximately 2.5 miles northwest of the City of Salem, Ohio in northern Columbiana County, near the southern border of Mahoning County

(Figure 1). The Site, covering approximately 44 acres, is situated on the north side of State Route 14 and west of Allen Road. It is bordered by a large field and a residential area on the south, a large wooded area to the north, an industrial facility (Crane-Deming Company) to the northeast, agricultural/residential areas to the northwest, and a residential area to the east. There are a few homes immediately to the east and southeast of the Site. The Site is dissected by Conrail railroad tracks that run southeast-northwest.

The Site is located on a topographic high, the axis of which runs southeast-northwest. The majority of the site slopes to the northeast and drains to the Middle Fork of the Little Beaver Creek (MFLBC). The geology is surficial Wisconsin Age glacial deposits (till, loam, sands) of variable thicknesses and extent, overlying a thick sequence of Middle Pennsylvanian Age sandstones and shales, with limestone and coal interbeds.

1.3.2 Site Operations History

A comprehensive history of site operations can be found in the RI Report (RNC, 1993). Briefly, from 1961 through 1977, Nease Chemical Company owned and operated a chemical manufacturing plant at the site. At various times during the period of 1961 through 1973, Nease produced a variety of chemical compounds, including household cleaning compounds, fire retardants,pesticides and chemical intermediates used in agricultural, pharmaceutical, and other chemical products. Some of these chemicals included mirex, diphenylsulfone, chloramine B, benzene, sulfonic acid, methoxychlor and hexachloroethylene.

Products and chemical intermediates were produced in batch processes. Waste generated was neutralized and treated on-site. Five unlined ponds (designated 1, 2, 3, 4, and 7) were used for treatment and storage of either acidic plant waste or lime slurries from neutralization of acidic wastes. After final settling, the neutralized liquid was discharge to the Salem Waste Water Treatment Plant (WWTP) from the late 1960s to 1975. In addition, prior to 1977, some drummed wastes were buried in an area, on-site, which is referred to as Exclusion Area A (Figure 1). The migration of these contaminants via the groundwater and overland via surface water and sediments has been documented.

In 1973, Nease discontinued manufacturing operations at the site and subsequently, decided to close the facility permanently. In 1974 and 1975, under OEPA supervision, all buildings and manufacturing equipment were removed from the site, except for a warehouse and two small block buildings. The five ponds were decommissioned (under OEPA supervision) by Nease in December 1975 pursuant to the 1973 Consent Judgment.

As of December 30, 1977, Nease Chemical Company, Inc. (including the site) was acquired by and merged with Ruetgers Chemical, Inc. The company resulting from the merger is Ruetgers-Nease Chemical Company, Inc. In 1983, the site was placed on the National Priorities List (NPL). The company officially changed its name to Ruetgers-Nease Corporation (RNC) on October 1, 1993.

1.3.3 Site Regulation History

The Remedial Investigation/Feasibility Study (RI/FS) AOC of January 1988 required RNC to conduct a remedial investigation (RI) "to determine fully the fact, nature and extent of any release or threatened release of hazardous substances, pollutants or contaminants at and/or from the Facility and to perform a Feasibility Study (FS) to identify and evaluate alternatives for the appropriate extent of remedial action to achieve in offer to comply with applicable or relevant and appropriate requirements, standards, limitations, criteria or goals and/or to prevent or mitigate the migration or release or threatened release of hazardous substances, pollutants, or contaminants from the facility, in accordance with Section 121 of CERCLA."

On April 5, 1991 RNC submitted a partial RI report to the agencies addressing the Middle Fork Little Beaver Creek.

On July 6, 1993, RNC submitted to the Agencies a Remedial Investigation Report . That report presents information obtained from existing data and from the data gathered during the RI field work conducted under the 1988 AOC.

1.3.4 Previous Investigations

A detailed account of past Response Actions at the Nease Chemical Site can be found in the RI Report (RNC, 1993). Briefly, RNC undertook programs for remediating constituents of concern at the site prior to the 1988 RI/FS AOC as follows:

Fall 1982: Soil borings taken, deep and shallow monitoring wells installed, geophysical investigation, collection and analysis of surface water, soil, and sediment, progress reports to OEPA.

Fall 1983: Additional monitoring wells, depth-discrete and composite surface soil samples, and sediment and surface water samples collected from the MFLBC and analyzed.

Spring 1984: Additional monitoring wells installed.

Summer 1984: Additional monitoring wells installed, deep groundwater test wells installed, aquifer testing for hydraulic properties and hydraulic interconnectedness of aquifers.

Spring 1985: Fish, sediment, surface water, and benthic organism sampling and analysis of MFLBC.

Winter 1985: *Additional soil borings.*

Spring 1986: Additional monitoring wells installed, additional soil borings.

April 1982, December 1982, April, September, and December 1983, February and July 1984, January, May, and December 1985, April 1986, March 1987: Groundwater sampling and analysis.

The findings of these studies have been incorporated into the Remedial Investigation Report submitted by RNC on July 6, 1993.

1.3.5 Previous Remedial Actions

RNC initiated remedial activities with a remediation plan submitted to the OEPA on March 12, 1983. The plan called for Phase 1 removal of buried drums in Exclusion Area A, and removal of affected soil from Exclusion Areas A and B; and Phase 2 assessment of constituents of concern in surface water, groundwater, soil, and sediment.

In the fall of 1983, RNC performed Phase 1 work as described above, and implemented various steps to control soil erosion at the site, including seeding to establish ground cover, installation of geotextile fabric barriers across drainages and ditches, installation of rock dams, and installation of barriers around the Exclusion Areas. Additionally, a leachate collection system was installed on the south side of the railroad tracks to mitigate the migration of groundwater seepage. The leachate has been intermittently collected and disposed of at a permitted off-site waste treatment facility since 1983.

Beginning in late 1991 and concluding in September of 1993, RNC voluntarily installed equipment at the Site, that was approved by U.S. EPA and OPEA, to reduce the potential for off-site transport and mitigation of certain constituents prior to implementing any permanent remedies deemed necessary in the future. The equipment was designed for surface water management and sediment control, and seep control and treatment.

1.4 Sample Network Design and Rationale

The sample network design and rationale for sample locations is described in detail in (Section 3.0) of the FSP.

1.5 Parameters To Be Tested and Frequency

For the Treatment Plant Performance Evaluation, sample matrices, analytical parameters and frequencies of sample collection can be found in Tables 1-1 and 1-2. Sample locations are shown on Figure 2. For field sampling associated with future investigative works, sampling will be conducted in accordance with the site specific conditions encountered. Field sampling will, in general, follow the field sampling protocols of the RI/FS investigation.

1.6 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions made during Removal Actions activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. Sample and data quality objectives for both the treatment plant evaluation and future field sampling are summarized in Table 1-3a and 1-3b. The levels of quality assurance and analytical data methodologies can be found in Table 1-4. There are five analytical levels which address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to ARARs, initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives (bench-scale tests). These types of data include those generated on-site through the use of OVA, pH, conductivity, and other real-time monitoring equipment at the site.

- Field Analyses (DQO Level 2): This provides rapid results and better quality than in Level 1. This level may include mobile lab generated data depending on the level of quality control exercised.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile lab generated data and some analytical

lab methods (e.g., laboratory data with quick turnaround used for screening but without full quality control documentation). Aqueous samples for VOCs, SVOCs, pesticides, metals and conventional parameters and air samples for VOCs (EPA Method TO-14) will require DQO Level 3.

- Confirmational (DQO Level 4): This provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives and PRP determination. These analyses require full Contract Laboratory Program (CLP) analytical and data validation procedures in accordance with EPA recognized protocol.

- Non-Standard (DQO Level 5): This refers to analyses by non-standard protocols, for example, when exacting detection limits or analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level 4 data. Aqueous samples for mirex, photomirex and kepone (MPK) will require DQO Level 4.

- Other: This category includes data obtained from other methods, e.g. visual analysis. For the Removal AOC Work Plan, recording of pressures across the air stripper and visual analysis of Granular Activated Carbon (GAC) are included in this category.

1.7 Project Schedule

The schedule for the treatment plant performance evaluation is shown on Figure 3. The schedule for field activities (as needed) to address removal actions of Paragraph 4f. of the Removal AOC is contained, in general, in the Removal Action Work Plan, Section 3.4.7.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

Several organizations will have responsibilities for implementing portions of the QAPjP. RNC owns the Site. Golder Associates Inc. (Golder) has been retained to conduct the Removal Actions. Midwest Research Institute (MRI) has been retained by RNC to perform chemical analysis for mirex, photomirex and kepone on the aqueous samples. Lancaster Laboratories, Inc. has been retained by Golder Associates Inc. to perform chemical analysis of all samples except for the aquatic toxicity testing which will be performed by RMC Environmental Services. The following descriptions summarize project responsibilities for several key team members (see Figure 4):

Project Coordinator

Ralph Pearce, P. E. of RNC is the Project Coordinator. Responsibilities of the Project Coordinator include:

1. Interface with U.S. EPA/OEPA RPM/Project Coordinator on all aspects of the Interim Remedial Measures.

2. Oversight and approval of Project plans.

3. Provide an independent evaluation of the quality assurance aspects of the Project.

Project Director

P. Stephen Finn, C. Eng., of Golder is the Project Director and is responsible for the following:

1. Resource availability to the project.

2. Overall technical direction of the project.

3. Review all technical documents for accuracy and completeness with respect to the QAPjP and associated site plans.

Project QA/QC Manager

Robert M. Glazier of Golder is the Project Quality Assurance Manager. He will be responsible for assuring that field, office, and laboratory activities and analyses are

conducted in accordance with the QAPjP. Specific responsibilities include the following:

1. Review all documents with respect to adherence to QA procedures provided in the QAPjP.

2. Preparation of analytical data tables and quality assurance reviews.

3. Recommend and institute corrective actions based on reviews and audits.

Removal Actions Project Manager

Geoffrey R. Forrest, C. P. Eng. of Golder is the Removal Actions Project Manager. He has overall project responsibility and direct responsibility for the management of staff involved with this project. Specific duties are as follows:

1. Providing an interface between the Project Coordinator and the Field Operations Manager.

2. Implementing project plans.

3. Coordinating project activities.

4. Coordinating project personnel and staffing.

5. Completing project deliverable reviews.

6. Providing input on technical direction.

Field Operations Manager

Joseph E. Cavanagh of Golder is the Field Operations Manager for this project and will be responsible for on-site activities including sampling and health and safety. Specific activities include the following:

1. Ensure that field activities are conducted in accordance with the Field Sampling Plan, QAPjP, and Health and Safety Plan.

2. Review and evaluate field records.

3. Subcontractor coordination.

4. Oversight of field staff.

Analytical Laboratory QA Manager

The Analytical Laboratory QA Managers, Eric McClellen of RMC Environmental Services, Louise Hess of Lancaster Laboratories and Gil Radolovich of Midwest Research Institute, will be responsible for the inspection and verification of laboratory QA/QC records and results. Any deviations or discrepancies in the data will be reported to the Laboratory Manager for review and, if necessary, appropriate corrective action. Additional responsibilities of the Analytical Laboratory QA Manager include:

1. Implementation of all laboratory QA/QC procedures contained in the QAPjP.

2. Overseeing corrective actions as required.

3. Conducting internal system and performance audits and inspection of analytical procedures and implementing modifications as approved by the Project QA/QC Manager.

4. Developing laboratory QA programs including statistical procedures and techniques.

Laboratory Sample Custodian

The Sample Management Department for Lancaster Laboratories, RMC Environmental Services and Midwest Research Institute will ensure proper sample entry, handling procedures and documentation by laboratory personnel.

U.S. EPA Remedial Project Manager and OEPA Project Coordinator

Ms. Sheila Sullivan is the U.S. EPA Region 5 Remedial Project Manager and Mr. Joseph Trocchio is the OEPA Project Coordinator and are responsible for the following:

1. Technical review and approval of all plans and data, including raw data, submitted as part of this Work Plan.

2. Coordination of Removal Actions activities with the Project Coordinator.

3. Authority vested in an On-Scene Coordinator and a Remedial Project Manager by the National Contingency Plan, 40 CFR Part 300, as amended, including the authority as provided therein to halt conduct, or direct any work described in the Removal Actions Work Plan, or to direct any response action undertaken by the U.S. EPA when conditions at the facility may present an imminent and substantial endangerment to the public health and welfare or the environment. The U.S. EPA Project Coordinator's actions shall, at all times be controlled and limited by provisions of the National Contingency Plan, 40 CFR Part 300.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The Overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instruments calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPjP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

3.1 Level of Quality Control Effort

Trip blank, field duplicate, equipment rinsate blank and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Trip blanks are provided by the laboratory and accompany sample containers from the lab to the site, and from the site returning to the lab. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One matrix spike/matrix spike duplicate will be collected for every 20 or fewer investigative samples. MS/MSD samples are designated/collected for organic and metal analyses. One equipment rinsate blank will be collected for every 10 or fewer investigative samples. Equipment rinsate blank samples are designated/collected for filtered metals.

The general level of the QC effort will be one field duplicate for every 10 or fewer samples. One volatile organic analysis (VOA) trip blank consisting of distilled deionized ultra pure water will be included along with each shipment of aqueous VOA samples.

Aqueous MS/MSD samples will be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer samples per matrix (i.e., groundwater, soil). The number of duplicate samples to be collected are listed in Table 3-1. Sampling procedures are specified in the FSP.

The level of QC effort provided by the laboratory will be equivalent to the level of QC effort specified under U.S. EPA SW-846 Methodologies for Test Methods for Evaluating Solid Waste (U.S. EPA, November 1986). The level of QC effort for

testing of organics (VOCs, SVOCs, pesticides) and inorganics will conform to the protocols of U.S. EPA SW-846 and EPA Method TO-14 (Attachment A). Tables 3-2, 3-3, 3-4 and 3-5 contain Practical Quantitation Levels (PQLs) for organic and inorganic compounds respectively. The level of QC effort for testing of mirex, photomirex and kepone will conform to the U.S. EPA-approved protocol for this site which will be analyzed by a Pulsed Positive Negative Chemical Ionization detection system. The level of QC effort for testing of ammonia, total organic carbon, chemical oxygen demand, total phenolics, biochemical oxygen demand, total dissolved solids, total suspended solids and total oil and grease will conform to protocols EPA 350.2, 415.1, 410.4, 420.1, 405.1, 160.1, 160.2 and gravimetric, respectively. The level of QC effort for aquatic toxicity testing will conform to protocols EPA/600/4-89/001 Short-term Methods for Estimating the Chronic Toxicity of Effluent and Receiving Waters to Freshwater Organisms, and EPA/600/4-90/027 Methods for Measuring the Acute Toxicity of Effluents on Receiving Waters to Freshwater and Marine Organism.

The QC level of effort for the field measurement of pH consists of pre-measurement calibration and a post-measurement verification using two standard reference solutions each time as appropriate to the sample pH. This procedure will be performed for each sample tested. The QC effort for field conductivity measurements will include daily calibration of the instrument using standard solutions of known conductivity.

3.2 Accuracy, Precision, and Sensitivity of Analysis

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols.

SOPs for laboratory analyses are provided in Attachment A. These include the required accuracy, precision, sensitivity of the analyses. SOPs for the field equipment to measure pH, conductivity, temperature, VOCs and iron are outlined in Attachment B. Accuracy and precision requirement for field screening analyses are also included in Attachment B.

3.3 Completeness, Representativeness and Comparability

COMPLETENESS is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that Lancaster Laboratories, RMC Environmental Services and Midwest Research Institute will provide data meeting QC acceptance criteria for 80 percent or more for all samples tested using the

methods outlined in Table 3-6. Following completion of the analytical testing, the percent completeness will be calculated by the following equations:

completeness (%): =

$$\frac{\text{(number of data reported)}}{\text{(number of samples submitted for each parameter analyzed)}} \times 100$$

REPRESENTATIVENESS expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to existing analytical data. The rationale of the sampling network is discussed in detail in the FSP. Representativeness will be satisfied by ensuring that the FSP is followed, proper sampling techniques are used, proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by the analysis of field duplicated samples.

COMPARABILITY expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPjP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of difference in procedures and QA objectives.

4.0 SAMPLING PROCEDURES

Sampling procedures are described in detail in the Field Sampling Plan (FSP) provided as Section I to this Volume.

5.0 SAMPLE CUSTODY

It is U.S. EPA and Region V Policy to follow the U.S. EPA Region V sample custody, or chain-of-custody protocols as described in "NEIC Policies and Procedures", EPA-330/9-78DDI-R, Revised June 1985. This custody is in three parts: Sample collection, Laboratory analysis, and Final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if they

- are in your possession;
- are in your view, after being in your possession;
- are in your possession and you place them in a secured location; or
- are in a designated secure area.

5.1 Field Chain-of-Custody Procedures

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact. The protocol for specific sample numbering using case numbers and traffic report numbers if applicable and other sample designations are included in the FSP.

5.1.1 Field Procedures

(a) The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

(b) The U.S. EPA's on-site representative will review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required.

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that

persons going to the site could re-construct a particular situation without reliance on memory.

Field logbooks, field survey books or notebooks will be bound. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned.
- Logbook number.
- Project name.
- Project start date, and
- End date.

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

Measurements made and samples collected will be recorded. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. The number of the photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the FSP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

5.1.3 Transfer of Custody and Shipment Procedures

(a) Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

(b) Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

(c) Whenever samples are co-located with a source or government agency, a separate Sample Receipt is prepared for those samples and marked to indicate with whom the samples are being co-located. The person relinquishing the samples to the facility or agency should request the representatives signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

(d) All shipments will be accompanied by the Chain-of-Custody Record identifying the contents. The original record will accompany the shipment.

(e) If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

5.2 Laboratory Chain-of-Custody Procedures

Laboratory custody procedures for sample receiving and log-in; sample storage; tracking during sample preparation and analysis; and storage of data are described in the SOP for each laboratory in Attachment A.

5.3 Final Evidence Files Custody Procedures

Golder will maintain all original documentation including but not limited to: field chain-of-custody forms, laboratory analytical reports, field notes, sample logs, telecons, photographs, memoranda, logbooks and letters in a designated secure, limited access area at the Golder Mt. Laurel, New Jersey office. All items being placed into this file will be assigned a document control identification number and a document description. All items will then be logged onto the file "Table of Contents." All items added or removed from the file will be signed and dated by the person making the entry. All records will be maintained in this fashion for a period of ten years.

The laboratories' final project report, which includes the raw data, will be retained in the laboratories' archives. These are permanent, locked archives which are not purged. All associated original logbooks will also be retained in these archives.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment which are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use on a scheduled, periodic basis.

6.1 Field Instruments/Equipment

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Equipment to be used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturing's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that the notations on any prior equipment problem are not overlooked, and all necessary repairs to equipment have been carried out.

Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. Field instruments will include a pH meter, specific conductivity meter, and Organic Vapor Analyzer (OVA) or Organic Vapor Photoionization Detector (PID). In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service.

The pH meter will be calibrated with standard buffer solutions prior to a field trip. In the field, the meter will be calibrated daily with two buffers before use. Thereafter, the meter will be checked against two buffer solutions which will be used for each field trip. Calibration procedures and frequency will be recorded in a field log book along with the lot numbers of the buffer. A general procedures for pH meter, specific conductivity meter and thermometer are described below:

pH Calibration

- Temperature of sample and buffer should be the same.
- Connect pH electrode into pH meter and turn on pH meter.

- Set temperature setting based on the temperature of buffer; place electrode in first buffer solution.
- After reading has stabilized, adjust "CALIB" knob to display correct value.
- Repeat procedure for second buffer solution.
- Place pH electrode in the sample and record the pH as displayed.
- Remove pH electrode from sample and rinse off with distilled water.
- The pH meter must be recalibrated every time it is turned off and turned back on, or if it starts giving erratic results.

The calibrations performed, standard used, and sample pH values are to be recorded in the field notebook. Appropriate new batteries will be purchased and kept with the meters to facilitate immediate replacement in the field as necessary.

Temperature Calibration

Temperature measurements are carried out utilizing a thermometer. The thermometers must be inspected before use to ensure there is no mercury separation. The thermometers should be rechecked in the field before and after use to see if the readings are logical and the mercury is still intact. The thermometers should be checked biannually for calibration, by immersing them in a bath of known temperature until equilibrium is reached. They should be discarded if found to have more than 10% error. The reference thermometer used for the bath calibration should be NBS traceable.

Conductivity Meter Calibration

The conductivity cells of the specific conductivity meter will be cleaned and checked against known conductivity standards before each field trip. In the field, the instrument will be checked daily with NBS traceable standards. The calibration procedure is described below.

- Place the probe in conductivity calibration standard solution.
- Set temperature knob for temperature of standard solution.

- Turn to appropriate scale and set the instrument for the value of calibration standard.

- Rinse off the electrode with distilled water.

- Measure the conductivity for distilled water to be used for a field blank, making sure temperature is set correctly for temperature of solution to be tested.

- If the conductivity of blank (distilled) water is high, it must be discarded and a new blank sample procured.

All readings and calibrations should be recorded in the field notebook.

The OVA will be checked daily by use of the internal calibration mechanism. The PID will be calibrated daily with a gas of known concentration.

Calibration of laboratory equipment will be based on approved written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing quality control activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will maintain a factory-trained repair staff with in-house spare parts or will maintain service contracts with vendors.

The records of calibration will be kept as follows:

- If possible each instrument will have record of calibration permanently affixed with an assigned record number.

- A label will be affixed to each instrument showing description, manufacturer, model numbers, date of last calibration, by whom calibrated (signature), and due date of next calibration reports and compensation or correction figures will be maintained with instrument.

- A written stepwise calibration procedure will be available for each piece of test and measurement equipment.

- Any instrument that is not calibrated to/with the manufacturer's original specification will display a warning tag to alert that analyst that the device carrier only a "Limited Calibration".

In all cases where analyses are conducted according to the U.S. Environmental Protection Agency (U.S. EPA) Contract Laboratory Program (CLP)/SW 846 protocols, the calibration procedures and frequencies specified in the applicable CLP Routine Analytical Services (RAS) Statement of Work (SOW) or SW 846 methods will be followed exactly. For analyses governed by SOPs, see the appropriate SOP for the required calibration procedures and frequencies, which are provided in Attachment A.

7.0 ANALYTICAL PROCEDURES

All leachate and air samples, collected during field sampling activities for Removal Actions at the Nease Chemical Site, Salem Ohio will be analyzed by either Lancaster Laboratories, RMC Environmental Services or Midwest Research Institute.

7.1 Laboratory Analysis

The analytical procedures for samples obtained under the Removal Actions AOC include SW846 Methodologies for VOCs, SVOCs (including 3,4-dichloronitrobenzene and diphenyl sulfone), pesticides, and filtered and unfiltered metals. Mirex, photomirex, and kepone will be analyzed utilizing GC/MS with the Pulsed Positive Negative Ion Chemical Ionization (PPNICI) detection system Version 4.1 (as per the Approved RI Work Plan, 1990). Conventional parameters such a pH, specific conductance, etc., will be analyzed as per the methods outlined in Section 7.2 below.

The acute toxicity tests will be performed for both *Ceriodaphnia dubia* and *Pimephales promelas* by the 48-Hour Static Daily Non-Renewal Definitive Bioassay. The chronic toxicity tests will be performed by a 7-day *Ceriodaphnia dubia* survival and reproduction test and a 7-Day fathead minnow, *Pimephales promelas*, survival and growth test. Air samples will be analyzed for VOCs (EPA Method TO-14).

Non-CLP Methodologies for chemical testing will be from the following documents:

> Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, SW-846, 3rd edition, U.S. EPA Office of Solid Waste, Washington, D.C., November 1986.
>
> Short-term Methods for Estimating the Chronic Toxicity of Effluent and Receiving Waters to Freshwater Organisms, EPA/600/4-89/001.
>
> Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, EPA/600/4-90/027.

Method references for the analyses to be performed for this project are summarized in Table 3-6.

7.2 Field Screening Analytical Protocols

The procedures for field measurement of pH, specific conductivity, iron and VOCs are described in Attachment B of this document.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Field Sample Collection

Please see the FSP for initial QC check. The assessment of field sampling precision and accuracy will be made through collection of field duplicates, trip blanks, and equipment rinsate blanks in accordance with the applicable procedures described in Section 3.1 at the frequency indicated in Table 3-1.

8.2 Field Measurement

QC procedures for pH, conductivity, and temperature measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings on a single sample or standard and by calibrating the instruments (where appropriate).

8.3 Laboratory Analysis

Internal quality control check procedures for analytical testing of all environmental samples will follow the specified methodologies, as discussed previously. These procedures specify the number of laboratory blanks to be used, the number of calibration standards, the frequency that the calibration standards must be run, and the frequency at which spiked and referenced samples must be run.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

Raw data from field measurements and sample collection activities will be appropriately recorded in the field log book. If the data are to be used in the project reports, they will be reduced for summaries and the method of reduction will be documented in the report.

9.1 Environmental Samples for Chemical Analysis

At a minimum, the laboratory report will show traceability of the samples and the reports will contain the following information:

1. Project identification;
2. Field sample identification numbers;
3. Laboratory sample identification numbers;
4. Sample matrix description;
5. Date and time of sample collection;
6. Analytical method description and reference citation;
7. Individual parameter results;
8. Date of analysis (extraction, first run, and subsequent runs);
9. Detection limits achieved;
10. Dilution or concentration factors; and,
11. Corresponding QC summary report.

Completed copies of the original chain-of-custody records for the appropriate samples will be included in the analytical results reports. Reporting units shall be consistent with the actual methodologies.

When testing for non-CLP parameters in environmental samples, the analytical reports from the laboratory may include the following information, where applicable, for data review and evaluation:

1. Sample results;

2. Laboratory and field duplicate results;

3. Matrix spike and/or matrix spike duplicate recovery results;

4. Laboratory control sample recovery and sample duplicate results;

5. Surrogate recovery limits;

6. Gas chromatograms and quantitation reports for environmental and quality control samples;

7. Procedural blank results;

8. Mass spectra for detected target analytes in environmental samples and blanks;

9. Instrument calibration results;

10. Detection limits and appropriate reporting units; and,

11. Laboratory chronicles.

9.2 Laboratory and Quality Control Reports

All laboratory results will be verified prior to release of the laboratory reports. The overall responsibility for reporting laboratory data lies with the laboratory manager.

For non-CLP methodologies, all of the above items may not be included in the laboratory report depending upon the parameters to be analyzed and methodologies used.

Quality control reports will be prepared which summarize the results of samples analyzed by the laboratory for quality control purposes. These reports will summarize all the quality control data results of the samples, including results for method blanks, duplicates, and matrix spikes. Spike concentrations, percent recoveries and relative percent differences will be reported. These reports will be used to prepare a project quality assurance report.

10.0 PERFORMANCE AND SYSTEM AUDIT

The Field Operations Manager will monitor the performance of the QA/QC procedures to ensure that the project is executed in accordance with the QAPjP and FSP.

Performance and systems audits of field activities may be conducted by the Project QA/QC Manager or Remedial Action Project Manager if deemed necessary. Alternatively, the U.S. EPA oversight contractor may undertake an independent, third-party field audit while at the Site.

11.0 PREVENTIVE MAINTENANCE

Preventive Maintenance Procedures will be carried out on all field and laboratory equipment in accordance with the procedures recommended by each manufacturer.

Field equipment must be subject to a routine maintenance program prior to and after each use. Equipment will be cleaned and its integrity checked after each use. In addition, parts with limited life (such as batteries, membranes, and some electronic components) shall periodically be checked and shall be replaced as necessary. Calibration checks will be performed twice daily and will be documented on the Field Meter/Calibration Log Sheets (see Figure 5).

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

12.1 Field Measurements

Field data will be assessed by the site Quality Control (QC) Officer. The site QC Officer will review the field results for compliance with the established QC criteria that are specified in the QAPjP and FSP. Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed on the basis of reproducibility by multiple reading of a single sample. An 80 percent goal for completeness has been established for this project. Data completeness will be calculated using Equation 12-1.

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100 \qquad \text{Eqt. 12-1}$$

12.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, completeness and sensitivity as follows:

12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between matrix spike/matrix spike duplicate (MS/MSD) for organic analysis, and laboratory duplicate analyses for inorganic analysis. The relative percent difference (%RPD) will be calculated for each pair of duplicate analysis using the Equation 12-2.

$$\%RPD = \frac{S - D}{(S + D)/2} \times 100 \qquad \text{Eqt. 12-2}$$

Where: S = First sample value (original or MS value)

D = Second sample value (duplicate or MSD value)

12.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section 3 of the QAPjP using the analytical results of method blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples, field blank, and bottle blanks. The percent recovery (%R) of matrix spike samples will be calculated using Equation 12-3.

$$\%R = \frac{A - B}{C} \times 100 \qquad \text{Eqt. 12-3}$$

Where:

A = The analyte concentration determined experimentally from the spiked sample;

B = The background level determined by a separate analysis of the unspiked sample, and;

C = The amount of the spike added.

12.2.3 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. The completeness is calculated using Equation 12-1.

12.2.4 Sensitivity

The achievement of method detection limits depend on instrumental sensitivity and matrix effects. Therefore it is important to monitor the instrumental sensitivity to ensure data quality through constant instrument performance. The instrumental sensitivity will be monitored through the analysis of method blank, calibration check sample, and laboratory control samples, etc.

13.0 CORRECTIVE ACTION PROCEDURES

The following procedures have been established to assure that conditions that adversely impact quality, such as malfunctions, deficiencies, deviations, and errors, are promptly investigated, documented, evaluated, and corrected.

When a significant adverse condition is noted at the site, laboratory, or subcontractor locations, the cause of the condition will be determined and corrective action taken to preclude repetition. Condition identification, cause, reference documents, and corrective action planned to be taken will be documented and reported to the Site Project Coordinator, Removal Actions Project Manager, involved subcontractor management, and the RPM, at a minimum. Implementation of corrective action is verified by documented follow-up on the part of the Project Manager. All project personnel have the responsibility, as part of the normal work duties, to promptly identify, solicit approved correction, and report such adverse conditions.

14.0 QUALITY ASSURANCE REPORTS

Effective management of a field sampling and analytical effort requires timely assessment and review of field activities. This will require effective interaction and feedback between the field team members, the Site Project Coordinator, and the Removal Actions Project Manager.

The Field Operations Manager will keep the Project Manager up-to-date regarding potential quality control problems so that a quick and effective solution can be implemented. Topics they may address include:

1. Summary of activities and general program status;
2. Summary of calibration data;
3. Summary of unscheduled maintenance activities;
4. Summary of corrective action activities;
5. Status of any unresolved problems;
6. Assessment and summary of data completeness; and,
7. Summary of any significant QA/QC problems and recommended and/or implemented solutions not included above.

Problems requiring swift resolution will be brought to the immediate attention of the Project Manager.

Matters pertaining to project quality assurance will be included in interim reports or correspondence submitted to U.S. EPA, as necessary.

TABLE 1-1
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE LOCATIONS AND LABORATORY ANALYSES

SAMPLE LOC.	DESCRIPTION	ESTIMATED COLLECTION TIME (MIN.X10)	VOCS(1)			SVOCS(2)			PEST.(3)			MPK(4)			METALS(5)			CONV.(6)			NPDES(8)		
			Day			Day			Day			Day			Day			Day			Day		
			1	3	5	1	3	5	1	3	5	1	3	5	1	3	5	1	3	5	1	3	5
T-1	Surge Tank (Influent)	0	X	X (9)	X	X	X (9)	X	X	X (9)	X	X	X (9)	X	X	X (9)	X	X	X	X			
SP-1	Influent to Air Stripper	2	X		X										X	X	X	X(7)	X(7)	X(7)			
SP-2	Influent to Bag Filter 2	24	X		X										X	X	X						
SP-3	Influent to Liquid GAC 1	26													X	X	X						
SP-4	Influent to Liquid GAC 2	43				X		X	X		X	X		X	X	X	X						
SP-5	Effluent (Liquid)	60	X	X*	X	X	X*	X	X	X*	X	X	X*	X	X	X*	X	X	X	X			X
A-1	Influent to Vapor GAC 1		X		X (9)																		
A-2	Effluent from Vapor GAC 2		X		X																		

Notes:
(1) Volatile organics will be analyzed by SW846 Method 8240 (liquid-phase) and Method TO14 (gaseous-phase).
(2) Semi-volatile organics will be analyzed by SW846 Method 8270 and will include 3,4-Dichloronitrobenzene and Diphenyl Sulfone.
(3) Pesticides will be analyzed by SW846 Method 8080.
(4) Mirex, photomirex, and kepone will be analyzed by Ruetgers-Nease Method as described in the Approved RI Work Plan.
(5) TAL Metals (unfiltered and filtered) will be analyzed by appropriate SW846 Methodolgies.
(6) Conventional analytes are pH, specific conductance, TDS, TSS, TOC, COD, BOD, and ammonia.
(7) This sample will be analyzed for TSS only.
(8) NPDES parameters include: oil and grease (total); Phenols (total), TUa ceriodaphnia (acute toxicity); TUc ceriodaphnia (chronic toxicity); TUa pimephales promelas (acute toxicity); and TUc pimephales promelas (chronic toxicity).
(9) A field duplicate will be collected from the influent on Day 3 for all analyses.
(10) Samples will be collected following the estimated hydraulic retention times from the influent (T-1) to the point of sample locations.
(*) Sample location where sufficient samples will be collected for MS/MSD analyses.

TABLE 1-2
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE LOCATIONS AND FIELD ANALYSIS (1)

SAMPLE LOC.	DESCRIPTION	IRON(2)	pH(3)	SPEC. COND.(3)	VOCs(4)
T-1	Surge Tank (Influent)	X	X	X	
SP-1	Influent to Air Stripper	X	X	X	
SP-2	Influent to Bag Filter 2	X	X	X	
SP-3	Influent to Liquid GAC 1	X	X	X	
SP-4	Influent to Liquid GAC 2	X	X	X	
SP-5	Effluent (Liquid)	X	X	X	
A-1	Influent to Vapor GAC 1				X
A-2	Effluent from Vapor GAC 2				X

NOTES:
(1) Samples will be collected from each location at least three times on each of the days during the performance evaluation.
(2) Iron will be analyzed by a Hach Kit (or equivalent).
(3) Specific conductance, temperature, and pH will be analyzed by an appropriate field instrument.
(4) Air-phase VOCs will be monitored by an OVA.

TABLE 1-3a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE AND DATA QUALITY OBJECTIVE SUMMARY
TREATMENT PLANT EVALUATION

Media	Sample Point Designation	Number of Samples	Data Quality Level	Parameters of Analysis	Objective
Aqueous	T-1	3	III	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters	Establish influent constituent concentrations
		3	V	MPK	
	SP-1	2	III	VOCs	Assess removal efficiency of metals and solids by Bag Filter 1; also, establish VOC concentration entering the air stripper
		3	III	TSS and Metals	
	SP-2	2	III	VOCs	Assess removal efficiency of VOCs by the air stripper; assess metals precipitation potential
		3	III	Metals	
	SP-3	3	III	Metals	Assess removal efficiency of metals by Bag Filter 2
	SP-4	2	III	SVOCs and Pesticides	Assess the removal efficiency of organics and metals by Aqueous GAC 1
		3	III	Metals	
		2	V	MPK	
	SP-5	3	III	VOCs, SVOCs, Pesticides, Metals, and Conventional Parameters	Assess the overall effluent quality in terms of discharge limits
		3	V	MPK	
		1	III	NPDES Parameters	
Air	A-1	2	III	VOCs	Establish vapor phase VOC concentrations
	A-2	2	III	VOCs	Assess removal efficiency of the vapor phase carbon

TABLE 1-3b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

SAMPLE AND DATA QUALITY OBJECTIVE SUMMARY
FIELD SAMPLING

Media	Sample Point Designation	Number of Samples	Data Quality Level	Parameters of Analysis	Objective
Aqueous	On- and off-site surface water bodies	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	On- and off-site groundwater	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
Soils/Sediments	On- and off-site surface soils	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	On- and off-site subsurface soils	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	Pond materials	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
	On- and off-site sediments	TBD	III V	VOCs, SVOCs, Pesticides Metals, and Conventional Parameters MPK	Establish contaminant concentrations, distributions, and migration pathways.
Air	On- and off-site air monitoring	TBD	I	VOCs	Establish contaminant concentrations for health and safety during sampling

TBD - To Be Determined

TABLE 1-4
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

LEVELS OF QUALITY ASSURANCE AND ANALYTICAL DATA METHODOLOGIES

Level	Description (1)	Associated Activity
I	Level I is the lowest quality data but provides the fastest results. Field screening or analysis provides Level I data. It can be used for health and safety monitoring. The generated data can indicate the presence or absence of certain constituents and generally qualitative rather than quantitative. It is the least costly of the analytical options.	- Health and safety monitoring - Field analyses - pH, specific conductivity, & temperature - Air screening using organic vapor analyzer
II	Level II data are generated by field laboratory analysis using more sophisticated portable analytical instruments or a mobile laboratory onsite. This provides fast results and better-quality data than in Level I. The analyses can be used to direct a removal action in an area, re-evaluate sampling locations, or direct installation of a monitoring well network.	- Not Applicable
III	Level III data may be obtained by a commercial laboratory with or without CLP procedures. (The laboratory may or may not participate in the CLP.) The analyses do not usually use the validation or documentation procedures required of CLP Level IV analysis. The analyzed parameters are relevant to the NPDES discharge criteria.	- Conventional parameters - NPDES Parameters - VOCs, SVOCs, Pesticides, & Metals - EPA Method TO14
IV	Level IV data are used for risk assessment, engineering design, and cost-recovery documentation. All analyses are performed in a CLP analytical laboratory and follow CLP procedures. Level IV is characterized by rigorous QC protocols, documentation, and validation.	- Not Applicable
V	Level V data are those obtained by nonstandard analytical procedures.	- MPK
OTHER	Other. This category includes data obtained from visual analyses of the carbon and recording of pressures across the air stripper and GAC beds.	- Carbon Core Samples

(1) EPA DQO Guidance Documents

TABLE 3-1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
Aqueous				
T-1	Volatile Organics	SW846 Method 8240	3	Primary
			1	Field Duplicates
			1	Trip Blank (1)
	Semi-Volatile Organics (2)	SW846 Method 8270	3	Primary
			1	Field Duplicate
	Pesticides	SW846 Method 8080	3	Primary
			1	Field Duplicate
	MPK	R-N Method (3)	3	Primary
			1	Field Duplicate
	Filtered Metals	SW846 Methodologies	3	Primary
			1	Field Duplicate
	Unfiltered Metals	SW846 Methodologies	3	Primary
			1	Field Duplicate
	Conventional Parameters	(4)	3	Primary
			1	Field Duplicate
SP-1	Volatile Organics	SW846 Method 8240	2	Primary
	Filtered Metals	SW846 Methodologies	3	Primary

TABLE 3-1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
	Unfiltered Metals	SW846 Methodologies	3	Primary
	Conventional Parameters (TSS Only)	EPA 160.2	3	Primary
SP-2	Volatile Organics	SW846 Method 8240	2	Primary
	Filtered Metals	SW846 Methodologies	3	Primary
	Unfiltered Metals	SW846 Methodologies	3	Primary
SP-3	Filtered Metals	SW846 Methodologies	3	Primary
	Unfiltered Metals	SW846 Methodologies	3	Primary
SP-4	Semi-Volatile Organics	SW846 Method 8270	2	Primary
	Pesticides	SW846 Method 8080	2	Primary
	MPK	R-N Method (3)	2	Primary

TABLE 3-1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
	Filtered Metals	SW846 Methodologies	2	Primary
	Unfiltered Metals	SW846 Methodologies	2	Primary
SP-5	Volatile Organics	SW846 Method 8240	3 1 1	Primary MS/MSD Trip Blank (1)
	Semi-Volatile Organics (2)	SW846 Method 8270	3 1	Primary MS/MSD
	Pesticides	SW846 Method 8080	3 1	Primary MS/MSD
	MPK	R-N Method (3)	3 1	Primary MS/MSD
	Filtered Metals	SW846 Methodologies	3 1	Primary MS/MSD
	Unfiltered Metals	SW846 Methodologies	3 1	Primary MS/MSD
	Conventional Parameters	(4)	3 1	Primary MS/MSD
	NPDES Parameters	(5)	1 1	Primary MS/MSD

TABLE 3-1a
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

PERFORMANCE EVALUATION SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
A-1	Volatile Organics	EPA Method TO14	2 1	Primary Field Duplicate
A-2	Volatile Organics	EPA Method TO14	2	Primary

NOTES:

1) One trip blank to be analyzed per day of sample shipment (3 Total).
2) Semi-volatile organic analysis will include 3,4-dichloronitrobenzene and diphenyl sulfone.
3) Mirex, photomirex, and kepone will be analyzed according to the Ruetgers-Nease Method as described in the approved RI Work Plan.
4) Conventional parameters include pH, specific conductance, TDS, TSS, TOC, COD, BOD, and ammonia. The respective methodologies are presented in Table 9.
5) NPDES parameters include total phenolics, total oil and grease, TUa ceriodaphnia, TUc ceriodaphnia, TUa pimephales promelas, and TUc pimephales promelas. The respective methods are presented in Table 9.

TABLE 3-1b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

FIELD SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
Aqueous Field Samples	Volatile Organics	SW846 Method 8240	TBD	Primary
			TBD	Field Duplicates
			TBD	Rinsate Blank
			TBD	Trip Blank (1)
			TBD	MS/MSD
	Semi-Volatile Organics (2)	SW846 Method 8270	TBD	Primary
			TBD	Rinsate Blank
			TBD	Field Duplicate
			TBD	MS/MSD
	Pesticides	SW846 Method 8080	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
	MPK	R-N Method (3)	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
	Filtered Metals(6)	SW846 Methodologies	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank
	Unfiltered Metals	SW846 Methodologies	TBD	Primary
			TBD	Field Duplicate
			TBD	Rinsate Blank

TABLE 3-1b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

FIELD SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples
	Conventional Parameters	(4)	TBD TBD TBD	Primary Field Duplicate Rinsate Blank
Soils/Sediments Field Samples	Volatile Organics	SW846 Method 8240	TBD TBD TBD TBD	Primary Field Duplicates Rinsate Blank Trip Blank (1)
	Semi-Volatile Organics (2)	SW846 Method 8270	TBD TBD TBD	Primary Field Duplicate Rinsate Blank
	Pesticides	SW846 Method 8080	TBD TBD TBD	Primary Field Duplicate Rinsate Blank
	MPK	R-N Method (3)	TBD	Primary
	Unfiltered Metals	SW846 Methodologies	TBD TBD TBD	Primary Field Duplicate Rinsate Blank
	Conventional Parameters	(4)	TBD	Primary Field Duplicate
Air Ambient Air Monitoring	Volatile Organics	Field Measurement	TBD	Primary

TABLE 3-1b
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

FIELD SAMPLING AND ANALYSIS PLAN SUMMARY
TARGET ANALYTES, ANALYTICAL METHODS, AND QUALITY ASSURANCE SAMPLES

Matrix	Parameter	Methodology	Number of Samples	Types of Samples

NOTES:

1) One trip blank to be analyzed per day of sample shipment (3 Total).
2) Semi-volatile organic analysis will include 3,4-dichloronitrobenzene and diphenyl sulfone.
3) Mirex, photomirex, and kepone will be analyzed according to the Ruetgers-Nease Method as described in the approved RI Work Plan.
4) Conventional parameters include pH, specific conductance, TDS, TSS, TOC, COD, BOD, and ammonia. The respective methodologies are presented in Table 9.
5) NPDES parameters include total phenolics, total oil and grease, TUa ceriodaphnia, TUc ceriodaphnia, TUa pimephales promelas, and TUc pimephales promelas. The respective methods are presented in Table 9.
   TBD - To be determined.
6) Filtered metals will be collected for groundwater samples only.

TABLE 3-2

PRACTICAL QUANTITATION LIMITS (PQL) FOR
VOLATILE ORGANICS

VOLATILES	CAS NUMBER	WATER (ug/L)	SOIL/SEDIMENT (ug/kg)
Acetone	67-64-1	100	100
Benzene	71-43-2	5	5
Bromodichloromethane	75-27-4	5	5
Bromoform	75-25-2	5	5
Bromomethane	74-83-9	10	10
2-Butanone (MEK)	78-93-3	100	100
Carbon Disulfide	75-15-0	100(1)	100(1)
Carbon Tetrachloride	56-23-5	5	5
Chlorobenzene	108-90-7	5	5
Chloroethane	75-00-3	10	10
Chloroform	67-66-3	5	5
Chloromethane	74-87-3	10	10
Dibromochloromethane	124-48-1	5	5
1,1-Dichloroethane	75-34-3	5	5
1,2-Dichloroethane	107-06-2	5	5
1,1-Dichloroethene	75-35-4	5	5
1,2-Dichloroethene (total)	540-59-0	5	5
1,2-Dichloropropane	78-87-5	5	5
cis-1,3-Dichloropropene	10061-02-6	5	5
Ethylbenzene	100-41-4	5	5
2-Hexanone	591-78-6	50	50
Methylene Chloride	75-09-2	5	5
4-Methyl-2-pentanone (MIEK)	108-10-1	50	50
Styrene	100-42-5	5	5
1,1,2,2-Tetrachloroethane	79-34-5	5	5
Tetrachloroethene	127-18-4	5	5
Toluene	108-88-3	5	5
1,1,1-Trichloroethane	71-55-6	5	5
1,1,2-Trichloroethane	79-00-5	5	5
Trichloroethene	79-01-6	5	5
Vinyl Chloride	75-01-4	10	10
Xylenes	1330-20-7	5	5
Vinyl Acetate	108-05-4	50	50
2-Chloroethylvinyl ether	110-75-8	10	10

NOTES:

(1) Carbon Disulfide will be reported with a "J" flag down to MDLs of 5 ug/L and 5 ug/kg, respectively.

TABLE 3-3

PRACTICAL QUANTITATION LIMITS (PQL) FOR
SEMI-VOLATILE ORGANICS

SEMI-VOLATILES	CAS NUMBER	WATER (ug/L)	SOIL/SEDIMENT (ug/kg)
Acenaphthene	83-32-9	10	330
Acenaphthylene	208-96-8	10	330
Anthracene	120-12-7	10	330
Benzo(a)anthracene	56-55-3	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(ghi)perylene	191-24-2	10	330
Benzo(a)pyrene	50-32-8	10	330
bis(2-Chloroethoxy)methane	111-91-1	10	330
bis(2-Chloroethyl)ether	111-44-4	10	330
bis(2-Chloroisopropyl)ether	39638-32-9	10	330
bis(2-Ethylhexyl)phthalate	117-81-7	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Butyl benzyl phthalate	85-68-7	10	330
4-Chloroaniline	106-47-8	10	330
p-Chloro-m-cresol	59-50-7	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Chlorophenol	95-57-8	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Chrysene	218-01-9	10	330
Dibenzofuran	132-64-9	10	330
Di-n-butyl phthalate	86-74-2	10	330
Dibenzo(a,h)anthracene	53-70-3	10	330
o-Dichlorobenzene	95-50-1	10	330
m-Dichlorobenzene	541-73-1	10	330
p-Dichlorobenzene	106-46-7	10	330
3,3'-Dichlorobenzidene	91-94-1	20	670
2,4-Dichlorophenol	120-83-2	10	330
Diethyl phthalate	84-66-2	10	330
2,4-Dimethylphenol	105-67-9	10	330
Dimethyl phthalate	131-11-3	10	330
4,6-Dinitro-o-cresol	534-52-1	25	830
2,4-Dinitrophenol	51-28-5	25	830
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Di-n-octyl phthalate	117-84-0	10	330

TABLE 3-3

PRACTICAL QUANTITATION LIMITS (PQL) FOR
SEMI-VOLATILE ORGANICS

SEMI-VOLATILES	CAS NUMBER	WATER (ug/L)	SOIL/SEDIMENT (ug/kg)
Fluoranthene	206-44-0	10	330
Fluorene	86-73-7	10	330
Hexachlorobenzene	118-74-1	10	330
Hexachlorocyclopentadiene	77-47-4	10	330
Hexachloroethane	67-72-1	10	330
Hexachlorobutadiene	87-68-3	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Isophorone	78-59-1	10	330
2-Methyl naphthalene	91-57-6	10	330
2-Methyl phenol	95-48-7	10	330
4-Methylphenol	106-44-5	10	330
Naphthalene	91-20-3	10	330
2-Nitroaniline	88-74-4	10	330
3-Nitroaniline	99-09-2	10	330
4-Nitroaniline	100-01-6	10	330
Nitrobenzene	98-95-3	10	330
o-Nitrophenol	88-75-5	10	330
p-Nitrophenol	100-02-7	25	830
n-Nitroso-di-n-propylamine	621-64-7	10	330
Pentachlorophenol	87-86-5	25	830
Phenanthrene	85-01-8	10	330
Phenol	108-95-2	10	330
Pyrene	129-00-0	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2,4,6-Trichlorophenol	88-06-2	10	330
Benzyl Alcohol	100-51-6	20	660
Benzoic Acid	65-85-0	50	1700
Diphenylsulfone	127-63-9	10	330
3,4-Dichloronitrobenzene	99-54-7	10	330

TABLE 3-4

PRACTICAL QUANTITATION LIMITS (PQL) FOR
PESTICIDES/PCBs

PESTICIDES/PCBs	CAS NUMBER	WATER (ug/L)	SOIL/SEDIMENT (mg/kg)
SW846 METHOD 8080			
Aldrin	309-00-2	0.01	0.01
alpha-BHC	319-84-6	0.01	0.01
beta-BHC	319-85-7	0.01	0.01
delta-BHC	319-86-8	0.01	0.01
gamma-BHC (Lindane)	58-89-9	0.01	0.01
Chlordane	57-74-9	0.1	0.05
4,4'-DDD	72-54-8	0.01	0.01
4,4'-DDE	72-55-9	0.01	0.01
4,4'-DDT	50-29-3	0.01	0.01
Dieldrin	60-57-1	0.01	0.01
Endosulfan I	959-98-8	0.01	0.01
Endosulfan II	33213-65-9	0.01	0.01
Endosulfan sulfate	1031-07-8	0.03	0.03
Endrin	72-20-8	0.01	0.01
Endrin aldehyde	7421-36-3	0.1	0.1
Endrin Ketone	53494-70-5	0.1	0.1
Heptachlor	76-44-8	0.01	0.01
Heptachlor epoxide	1024-57-3	0.01	0.01
Methoxychlor	72-43-5	0.05	0.05
Toxaphene	8001-35-2	2	2
Aroclor 1016	12674-11-2	1	0.2
Aroclor 1221	11104-28-2	1	0.2
Aroclor 1232	11141-16-5	1	0.2
Aroclor 1242	53469-21-9	1	0.2
Aroclor 1248	12672-29-6	1	0.2
Aroclor 1254	11097-69-1	1	0.2
Aroclor 1260	11096-82-5	1	0.2
R-N METHOD PPNCIMS VERSION 4.1			
Mirex	2385-85-5	0.00544	0.0185
Photomirex	---	0.474	0.0204
Kepone	143-50-0	0.132	0.068

TABLE 3-5

PRACTICAL QUANTITATION LIMITS (PQL) FOR INORGANICS

ANALYTE	IDL(1) (ug/L)	WATER (mg/L)	SOIL/SEDIMENT (mg/kg)
Aluminum	22	0.2	30
Antimony	21	0.2	20
Arsenic (GFAA)	1	0.005	2
Barium	2	0.1	10
Beryllium	0.8	0.01	0.5
Boron	5	0.04	10
Cadmium	3	0.01	2
Calcium	48	0.2	30
Chromium	6	0.03	4
Cobalt	4	0.05	5
Copper	5	0.025	5
Iron	7	0.1	10
Lead (GFAA)	1	0.003	1
Magnesium	45	0.1	15
Manganese	2	0.01	2
Mercury	0.008	0.0002	0.3
Molybdenum	8	0.05	5
Nickel	8	0.05	5
Potassium	174	0.5	50
Selenium (GFAA)	1	0.003	2
Silicon	57	0.5	25
Silver	4	0.02	2
Sodium	62	0.4	50
Thallium (GFAA)	3	0.01	2
Vanadium	5	0.015	2
Zinc	4	0.025	10
Cyanide(2)	--	0.005	0.1

(1) Instrument Detection Limits as indicated by Lancaster Laboratories, Inc. which are highly dependent on operating conditions and plasma position.

(2) Cyanide does not have an IDL.

TABLE 3-6
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION AND ANALYTICAL HOLD TIMES FOR AQUEOUS AND GASEOUS SAMPLES

PARAMETER	METHODOLOGY	CONTAINER		MINIMUM SAMPLE	PRESERVATION (a)	HOLD TIME (b)
Volatile Organics (Aqueous)	SW846 Method 8240	(3)-40 ml	G	(3) - 40 ml (d)	Cool 4 deg C;HCl,pH<2	14 days (c)
Volatile Organics (Gaseous)	EPA Method TO14	(1) 5 l	Summa Can.	5 l	None	14 days (c)
Semi-Volatile Organics	SW846 Method 8270	(2)-1000 ml	Amber G	1000 ml (d)	Cool 4 deg C	7 days (e)
Pesticides	SW846 Method 8080	(2)-1000 ml	Amber G	1000 ml (d)	Cool 4 deg C	7 days (e)
MPK	R-N Method(g)	(2)-1000 ml	Amber G	1000 ml (d)	Cool 4 deg C	7 days (e)
Total Cyanide-TAL	SW846 Methodologies	(2)-1000 ml	P or G	1000 ml (d)	Cool 4 deg C;NaOH,pH>12	14 days
Total Metals-TAL	SW846 Methodologies	(1)-1000 ml	P or G	1000 ml (d)	Cool 4 deg C;HNO3,pH<2	180 days (f)
Ammonia	EPA 350.2	(1)-1000 ml	P or G	400 ml	Cool 4 deg C;H2SO4,pH<2	28 days
Total Organic Carbon	EPA 415.1	(1)-500 ml	P or G	250 ml	Cool 4 deg C;HCl,pH<2	28 days
Chemical Oxygen Demand	EPA 410.4	(1)-125 ml	P or G	50 ml	Cool 4 deg C;H2SO4,pH<2	28 days
Total Phenolics	EPA 420.1	(1)-1000 ml	G	1000 ml	Cool 4 deg C;H2SO4,pH<2	28 days
Biochemical Oxygen Demand	EPA 405.1	(2)-1000 ml	P or G	1000 ml	Cool 4 deg C	48 hours
Total Dissolved Solids	EPA 160.1	(1)-250 ml	P or G	100 ml	Cool 4 deg C	7 days
Total Suspended Solids	EPA 160.2	(1)-250 ml	P or G	100 ml	Cool 4 deg C	7 days
Total Oil and Grease	Gravimetric	(1)-1000 ml	G	1000 ml	Cool 4 deg C	7 days
TUa ceriodaphnia	Note h	5 gal.	P	5 gal.	Cool 4 deg C	7 days
TUc ceriodaphnia	Note i	5 gal.	P	5 gal.	Cool 4 deg C	7 days
TUa pimephales promelas	Note h	5 gal.	P	5 gal.	Cool 4 deg C	7 days
TUc pimephales promelas	Note j	5 gal.	P	5 gal.	Cool 4 deg C	7 days
Specific Conductance	Electrode		P or G	NA	None	Field Measurement
pH	Electrode		P or G	NA	None	Field Measurement
Temperature	Thermometer		G	NA	None	Field Measurement

TABLE 3-6
NEASE CHEMICAL SUPERFUND SITE, SALEM, OHIO

ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION AND ANALYTICAL HOLD TIMES FOR AQUEOUS AND GASEOUS SAMPLES

NOTES:

(a) Sample Preservation is performed by sampler immediately upon sample collection. Dissolved metals samples will be filtered first and then preserved. Separate trial bottles will be used to assess the amount of preservative required to achieve pH<2 or pH>12.

(b) Hold time based upon day of sample collection not verified time of sample receipt.

(c) If preservation is not possible due to foaming, an unpreserved sample will be collected. The hold time will be 7 days for unpreserved sample. Preservation of VOCs in Water: Adjust the pH of the sample to less than 2 by carefully adding 1:1 hydrochloric acid (HCl) drop by drop to the required (3) 40 ml VOA sample vials. The number of drops of 1:1 HCl required should be determined on a fourth portion of sample of equal volume. Cool to 4°C. If acidification of the sample causes effervescence the samples must be submitted without preservation except for cooling to 4°C. This sample property should be appropriately noted when present, and the reduced holding times of seven days alerted to the laboratory.

(d) Triple sample volume is required for MS/MSD analysis of organics and metals at a rate of one per twenty field samples (excludes blank samples).

(e) 7 days for extraction, 40 days for analysis after commencement date of extraction.

(f) Hold time for Mercury is 28 days.

(g) Mirex, Photomirex, and Kepone will be analyzed by a Pulsed Positive Negative Chemical Ionization detection system.

(h) Acute Toxicity Test - 48-hour Static Non-Renewal Definitive Bioassay.

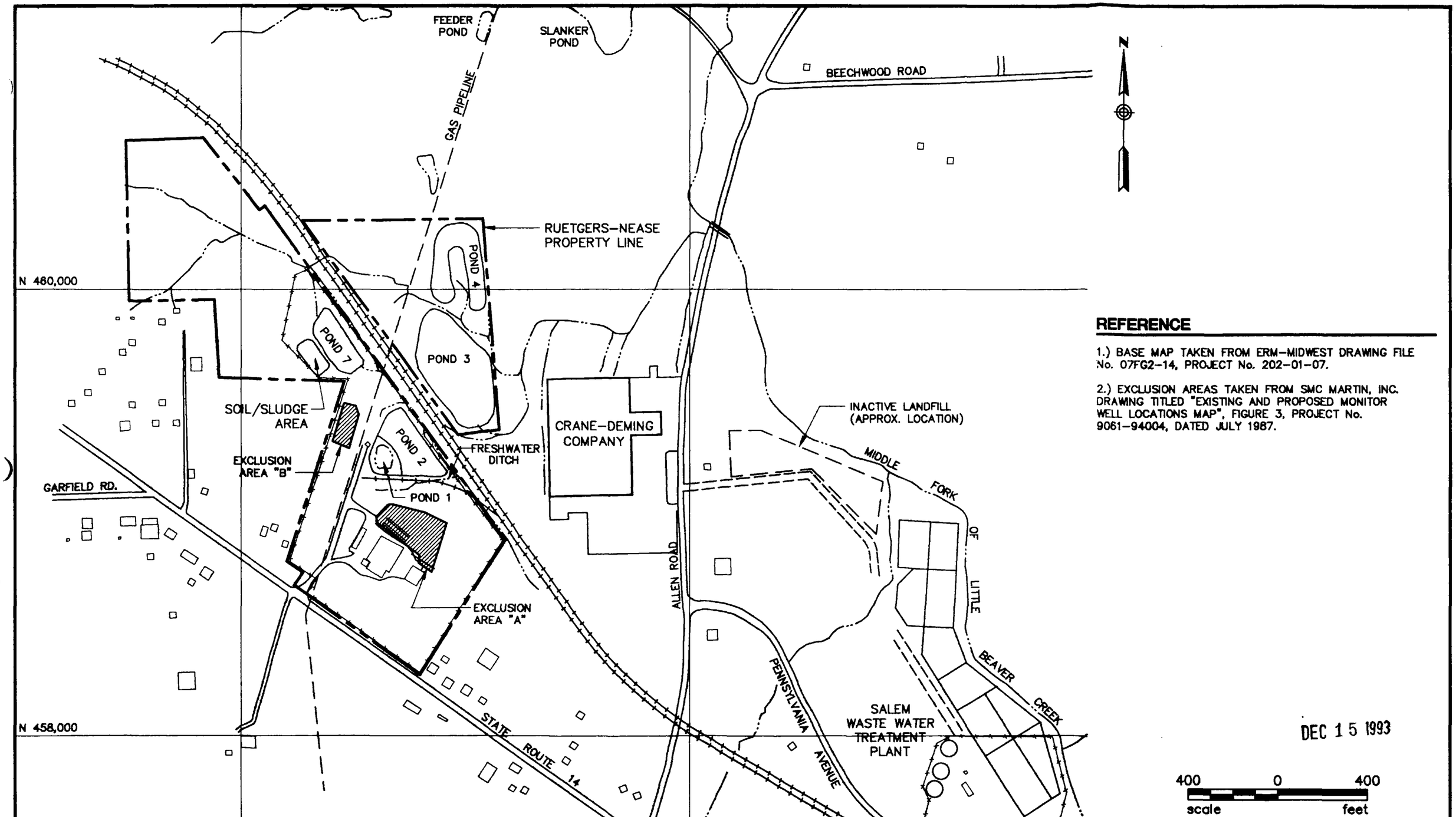
(i) Chronic Toxicity Test - 7-day Ceriodaphnia dubia survival and reproduction test.

(j) Chronic Toxicity Test - 7-day fathead minnow, Pimephales promelas, survival and growth test.

1. EPA "Methods for Chemical Analysis of Water and Waste", 1979, revised 3/83.
2. SM "Standard Methods for Water and Wastewater," 16th edition.
3. SW846 "Test Methods for Evaluating Solid Waste", November 1986.

ABBREVIATIONS

P = Polyethylene
G = Glass



REFERENCE

1.) BASE MAP TAKEN FROM ERM-MIDWEST DRAWING FILE No. 07FG2-14, PROJECT No. 202-01-07.

2.) EXCLUSION AREAS TAKEN FROM SMC MARTIN, INC. DRAWING TITLED "EXISTING AND PROPOSED MONITOR WELL LOCATIONS MAP", FIGURE 3, PROJECT No. 9061-94004, DATED JULY 1987.

DEC 15 1993

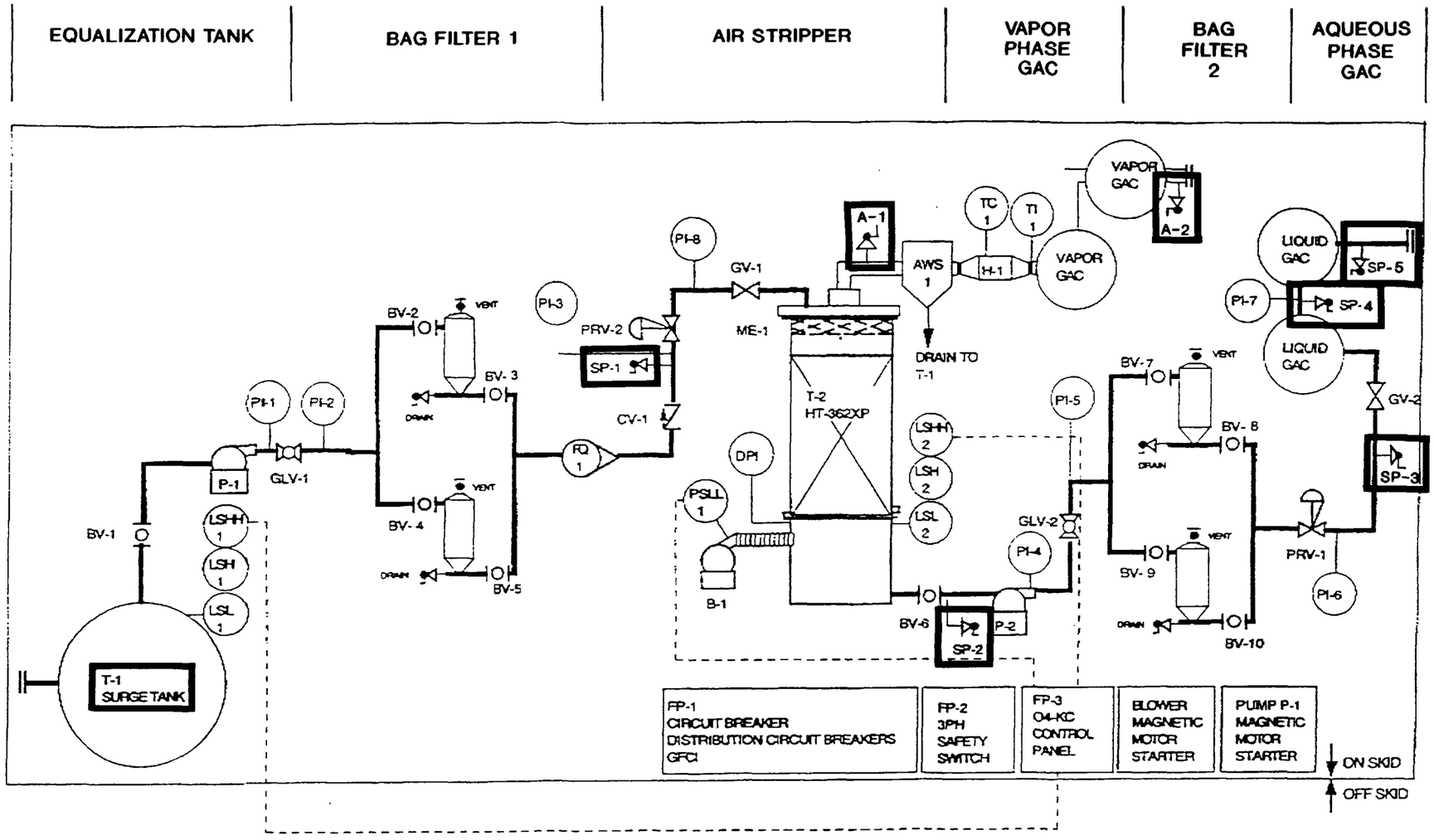
JOB No.:	933-6158	SCALE:	AS SHOWN
DR BY:	MRM	DATE:	10/01/93
CHK BY:	TH	FILE No.:	OH01-230
REV BY:	GRF	DR SUBTITLE:	01

Golder Associates

SITE LOCATION PLAN
SALEM OHIO SITE

RUETGERS-NEASE CORP.

FIGURE 1



LEGEND

SAMPLING LOCATION / PORT

SAMPLE	DESCRIPTION
T-1	SURGE TANK (INFLUENT)
SP-1	INFLUENT TO AIR STRIPPER
SP-2	INFLUENT TO BAG FILTER 2
SP-3	INFLUENT TO LIQUID GAC 1
SP-4	INFLUENT TO LIQUID GAC 2
SP-5	EFFLUENT
A-1	INFLUENT TO VAPOR GAC 1
A-2	EFFLUENT FROM VAPOR GAC 2

DEC 15 1993

JOB No.:	933-6158	SCALE:	N/A
DR BY:	EAM	DATE:	11/12/93
CHK BY:	THR	FILE No.:	OH01-220
REV BY:	GRF	DR SUBTITLE:	01

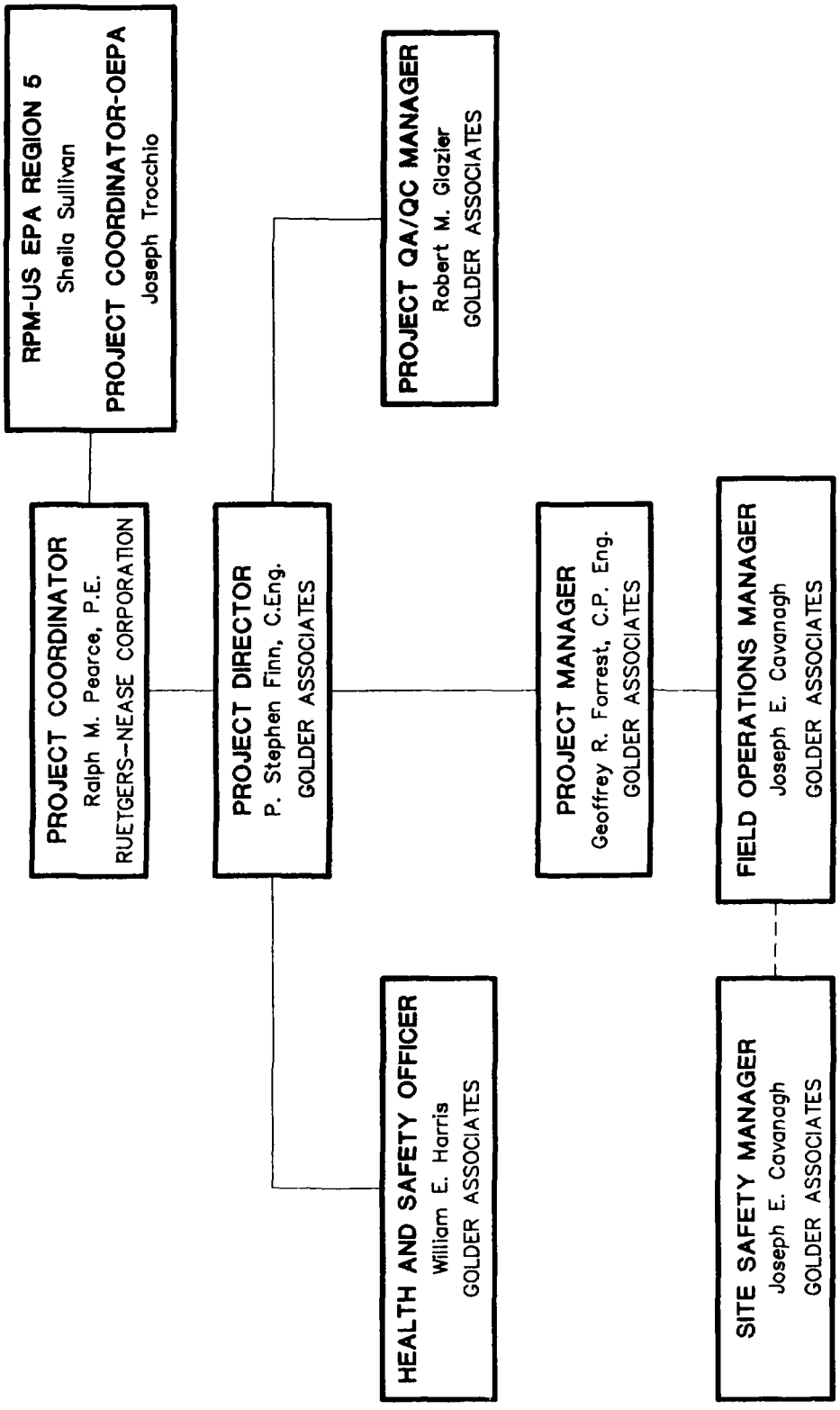
Golder Associates

SALEM IRM

TREATMENT PLANT SAMPLING LOCATIONS

RUETGERS-NEASE CORP.

FIGURE 2



DEC 15 1993

JOB No.:	933-6158	SCALE:	N/A
DR BY:	WME	DATE:	12/15/93
CHK BY:	THR 12/15/93	FILE No.:	OH01-242
REV BY:	GRF 12/15/93	DR SUBTITLE:	01

REMOVAL ACTION WORK PLAN
SALEM, OHIO SITE
PROJECT ORGANIZATION CHART

Golder Associates

RUETGERS-NEASE CORPORATION

FIGURE 4

Golder Associates

METER CALIBRATION RECORD

				TEMP. RESULTS		pH RESULTS						SPEC. COND. RESULTS			
Date	Time	By	Meter #	NBS	Meter	Buffer pH 7	Meter Reading	Buffer pH 4	Meter Reading	Buffer pH 10	Meter Reading	Conc. Std. 1 umho/cm	Meter Reading of Std. 1 umho/cm	Conc. Std. 2 umho/cm	Meter Reading of Std. 2 umho/cm

DEC 15 1993

FIGURE 5

ATTACHMENT A

STANDARD OPERATING PROCEDURES (SOP) FOR LABORATORIES

LANCASTER LABORATORY

STANDARD OPERATING PROCEDURES

Analysis #820/1848
Initiated Date: 3/6/91
Effective Date: AUG 12 1992

CONFIDENTIAL

Sample Preparation of Wastewater for Inductively Coupled Plasma (1848) and Atomic Absorption (820) Analysis of Metals

Reference:

USEPA CLP SOW No. ILM01.0, Exhibit D, Section III, A.2, D-5.

Scope:

This acid digestion procedure is used to prepare wastewater samples for measurement of metals by inductively coupled plasma or atomic absorption spectroscopy. For potable water and NPDES wastewater samples, refer to analysis #820, Sample Preparation of Potable Water and NPDES Wastewater for Atomic Absorption Analysis of Metals.

Basic Principle:

The samples are digested with nitric and hydrochloric acid to dissolve metals.

Apparatus and Reagents:

For reagent preparation, shelf life, and storage conditions, see SOP-10-007, Preparation of Standards and Solutions.

1. 250 ml beakers, or other appropriate beakers

2. Watch glasses

3. Nitric Acid, HNO_3, 70.0-71.0%, "Baker Instra-Analyzed" Reagent, 1.428 g/ml, or equivalent

4. Hydrochloric Acid, HCl, 36.5-38.0%, "Baker Instra-Analyzed" Reagent, 1.194 g/ml, or equivalent

5. Nitric Acid (1:1) - Add 500 ml of HNO_3 to 500 ml of deionized water

6. Hydrochloric Acid (1:1) - Add 500 ml of HCl to 500 ml of deionized water

7. 100 ml graduated cylinders, or other appropriate graduated cylinders

8. 100 ml volumetric flasks, or other appropriate class A volumetric flasks

9. 125 ml Nalgene bottles, or other appropriate Nalgene bottles

10. Hot plates

Notes: As long as the correct ratios are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Safety Precaution:

Refer to SOP-IO-011, Inorganic Analysis Safety Procedures.

Procedure:

For sample preservation, storage conditions and holding times, see SOP-IO-001, Preservation and Holding Times for Inorganic Analyses.

CONFIDENTIAL

Shake sample well. Using a 100 ml graduated cylinder, transfer 100 ml of sample into a 250 ml beaker. Add 2 ml of (1:1) HNO_3 and 10 ml of (1:1) HCl. Cover the beaker with a watch glass. Heat the solution on a hot plate at about 95°C for approximately two hours or until the sample volume is reduced to between 25 and 50 ml, making certain the sample does not boil.

Cool. Transfer the solution to a 100 ml volumetric flask. Adjust sample volume to 100 ml with deionized water and mix. Transfer the solution to a Nalgene bottle.

Notes: For soluble metals analysis, filter unpreserved sample through 0.45 micron filter paper. Adjust the filtered sample to pH 2 or less with nitric acid preserving solution. Measure 100 ml of sample and digest as normal.

If silicon (Si) or boron (B) is requested, digest using Teflon vessels.

If insufficient sample is submitted to allow using the method stated volume or the sample contains high solids, one may use a smaller aliquot of the sample. If lower than normal limits are requested, samples may be concentrated. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

Quality Assurance:

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every digestion batch (20 samples or less).

CONFIDENTIAL

Prepared by: Eugene K. Abel Date: 8-11-92

Approved by: [illegible] Date: 8-11-92

Approved by: Kathleen M. Lowen Date: 8-11-92

CONFIDENTIAL

Analysis #1056
Initiated Date: 4/21/88
Effective Date: AUG 12 1992

CONFIDENTIAL

Sample Preparation of Potable Water and Wastewater for Graphite Furnace Analysis of Metals

Reference:

USEPA CLP SOW No. ILM01.0, Exhibit D, Section III, A.1., D5

Scope:

This acid digestion procedure is used to prepare potable water and wastewater samples for measurement of metals by graphite furnace.

Basic Principles:

The samples are digested using nitric acid and 30% hydrogen peroxide to dissolve metals.

Apparatus and Reagents:

For reagent preparation, shelf life and storage conditions, see SOP-IO-007, Preparations of Standards and Solutions.

1. 250 ml beakers or other appropriate beakers

2. Watch glasses

3. Nitric Acid, HNO_3, 70.0-71.0%, "Baker Instra-Analyzed" Reagent, 1.428 g/ml, or equivalent

4. Nitric acid, (1:1) - Add 500 ml of HNO_3 to 500 ml of deionized water

5. Hydrogen peroxide, 30% H_2O_2

6. 100 ml graduated cylinders or other appropriate graduated cylinders

7. 100 ml volumetric flasks or other appropriate class A volumetric flasks

8. 125 ml Nalgene bottles or other appropriate Nalgene bottles

9. Hot plates

Note: As long as the correct ratios are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Safety Precautions:

Refer to SOP-IO-011, Inorganic Analysis Safety Procedures.

Procedure:

Shake sample well. Using a 100 ml graduated cylinder, transfer 100 ml of sample into a 250 ml beaker. Add 1 ml of (1:1) HNO_3 and 2 ml of 30% H_2O_2. Cover the beaker with a watch glass. Heat the solution on a hot plate at about 95°C for approximately two hours or until the sample volume is reduced to between 25 and 50 ml, making certain that the sample does not boil.

Cool. Transfer the solution to a 100 ml volumetric flask. Adjust sample volume to 100 ml with deionized water and mix. Transfer the solution to a Nalgene bottle.

CONFIDENTIAL

NOTES: For soluble metals analysis, filter unpreserved sample through 0.45 micron filter paper. Adjust the filtered sample to pH 2 or less with nitric acid preserving solution. Measure 100 ml of sample and digest as normal.

If insufficient sample is submitted to allow using the method stated volume or the sample contains high solids, one may use a smaller aliquot of the sample. If lower than normal limits are requested, samples may be concentrated. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

Quality Assurance:

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every digestion batch (20 samples or less).

NO1056.W51
IO METHODS #1
081092

CONFIDENTIAL

Prepared by: Eugene K. Abel Date: 8-11-92

Approved by: [illegible] Date: 8-11-92

Approved by: [illegible] Date: 8/12/92

Analysis #491
Initiated Date: 11/8/85
Effective Date: APR 2 0 1992

CONFIDENTIAL

Phenol Distillation
(as Preparation for Analysis on the Technicon Automated Analyzer II)

Reference:

1. EPA Methods for Chemical Analysis of Water and Wastes EPA 600/4-79-020, Method 420.1

2. Standard Methods for the Examination of Water and Wastewater, 16th ed, 1985, p. 556, Method #510A.

3. Test Methods for Evaluating Solid Waste/Physical/Chemical Methods SW-846, 9065.

Scope and Application:

This method is a preparation for colorimetric determination of phenol by the Technicon Automated Analyzer II. The method is applicable to solid wastes, soils, NPDES waters, and any water or waste too turbid to be analyzed directly on the Technicon AA II.

Method Summary:

This method takes solid wastes, soils, NPDES waters, and waters or wastes that are to turbid for direct analysis and subjects them to specific conditions in order to extract ortho- and meta-substituted phenols. Some para-substituted phenols, such as carboxyl, halogen, methoxyl, or sulfonic acid groups may also be extracted under the proper conditions.

CONFIDENTIAL

Interferences:

Interferences from sulfur containing compounds are eliminated by acidifying the sample to a pH less than four with concentrated H_2SO_4, aerating briefly by stirring, and adding $CuSO_4$ (1 g/l). The acidification and copper sulfate also help to inhibit biological degradation.

Oxidizing agents such as chlorine may partially oxidize the phenolic compounds and cause low results. For water samples, test by placing a drop of the sample on potassium iodide starch paper. A bluish color indicates the presence of oxidizing agents. Add an excess of ferrous ammonium sulfate.

Apparatus and Reagents:

1. Distillation apparatus, all glass, consisting of a one liter glass distilling apparatus with a Friedrich's condensor. See Figure 1.

2. pH meter

3. Sulfuric acid (concentrated)

4. Ferrous ammonium sulfate:
   Dissolve 1.1 g in 500 ml deionized water containing 1 ml concentrated H_2SO_4 and dilute to one liter with deionized water.

5. Copper sulfate:
   Dissolve 150 g $CuSO_4 \cdot 5H_2O$ in deionized water and dilute to one liter.

CONFIDENTIAL

Safety Precautions:

Normal laboratory safety practices must be followed. Use caution when handling any chemicals, solvents, or reagents. Avoid inhalation and skin contact of any chemicals, standards, or samples. The stock standard solution is considered toxic. Inspect all glassware before use. Discard or send for repair any glassware that is chipped, flawed, or broken.

Procedure:

1. Place 500 ml of sample or an aliquot diluted to 500 ml with deionized water in a 600 ml beaker. If the sample is a solid, weigh 20 g (if the sample is a solvent or oil, weigh 5 g or less) and place in a 600 ml beaker with 500 ml of deionized water.

2. Adjust the pH of the sample to between 3.9 and 4.1 with NaOH and/or H_2SO_4. Add 5 ml of $CuSO_4$ solution while stirring.

3. Quantitatively transfer the solution to the boiling flask, adding some boiling beads.

4. Distill 450 ml of the sample at 100°C into a clean 600 ml beaker. Stop the distillation and allow the distillation apparatus to cool slightly. Transfer the distillate to a 500 ml volumetric flask. Add 50 ml deionized water to the distillation flask and resume distillation until 500 ml has been collected.

5. Subsample the final solution into two 40 ml vials. Label each vial with the sample number, weight or volume used in the distillation, "PHENOL", employee initials and number, and date of distillation. Store the vials in the Instrumental Water Quality subsample refrigerator at 2 to 6°C. If the samples require locked storage, create a distillate chain of custody and return all subsamples to locked storage with the chain.

Quality Control:

NOTE: The acceptance criteria for the following quality control samples are listed in the colorimetric determination SOP (Analysis #434).

1. One sample will be prepared in duplicate for every batch (not to exceed 20 samples) following the same procedure as the rest of the samples.

2. A spike will be prepared for every batch (not to exceed 20 samples) following the same procedure. Spike the sample with 0.1 ml of the 1000 mg/l phenol quality control stock standard prepared in analysis #434.

3. A batch blank will be prepared for every batch or each day samples are prepared (not to exceed 20 samples). The blank is 500 ml of deionized water, taken through the distillation procedures just as the samples are.

4. A laboratory control standard shall be prepared for every batch or each day samples are prepared (not to exceed 20 samples). The standard is prepared by

CONFIDENTIAL

spiking 500 ml deionized water with 0.1 ml of the 1000 mg/l phenol quality control stock standard prepared in analysis #434, using the same procedure. The value of this standard is 0.200 mg/l.

CONFIDENTIAL

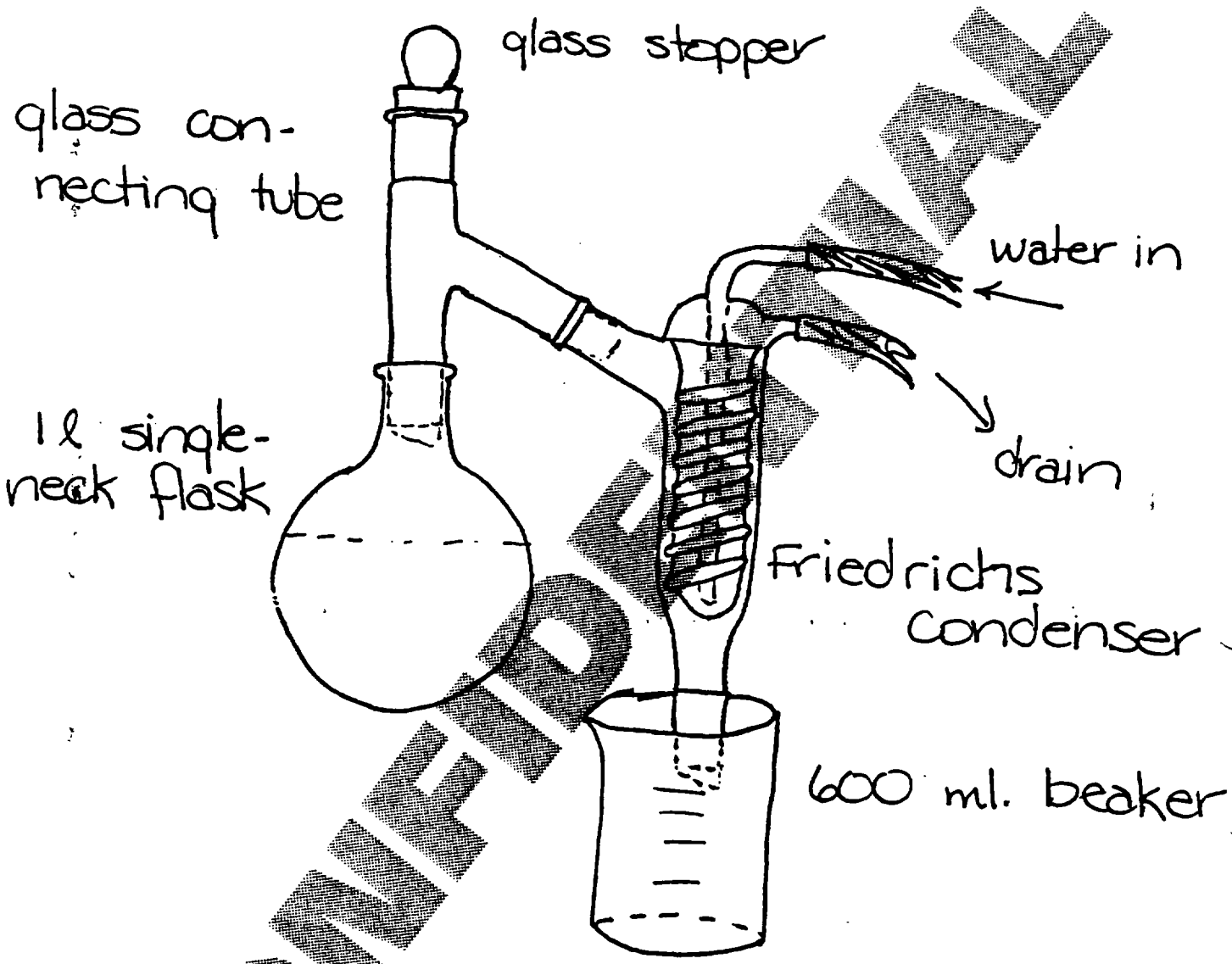
NO491
IC METHODS #1
040292

Prepared by: David Evans Date: 4/15/92

Approved by: Ramona V Layman Date: 4/15/92

Approved by: [illegible] Date: 4/16/92

Figure I



Phenol Distillation Apparatus

CONFIDENTIAL

Lancaster Laboratories INCORPORATED
Where quality is a science

RAW DATA LOGBOOK
PHENOL DISTILLATIONS
ANALYSIS #491

BATCH NO. ☐☐☐☐☐ ☐☐☐ ☐☐☐ ☐☐☐☐☐

Date: _____ Analyst: _____

QA Number	Sample Number	Distilled Weight(g)	Distilled Volume(ml)	Comments

CONFIDENTIAL

Please Note: Final extract volume is 500 ml.

1090 Rev. 6/7/90

2425 New Holland Pike, Lancaster, PA 17601-5994 / 717-656-2301

Analysis #434
Initiated Date: 12/07/84
Effective Date: MAY 13 1993

CONFIDENTIAL

Automated Determination of Phenols in Water, Wastewater, and Soils Alpkem Flow Analyzer

References:

1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846, Method #9066A.

2. Methods for Chemical Analysis of Water and Wastes, 420.2, EPA 600/4-79-020.

3. "Phenol", The Flow Solution Methodology, Document No. 000586, Alpkem Publication 3/92 Rev C.

Scope:

This method is applicable to potable water, groundwater, and wastewater. Soils, NPDES wastewaters, and water samples high in suspended solids or solid samples cannot be done using this method unless they are predistilled (Analysis #491). This automated method is capable of analyzing 30 to 40 samples per hour. The limit of quantitation is 0.005 mg/l for water at 500 ml and 0.1 mg/kg for soils at 20 grams. The range is 0.005 to 0.310 mg/l phenol. This range may be extended by dilution.

Method Summary:

The method is based on the automated distillation of phenol and the subsequent reaction with 4-aminoantipyrine (4AAP) and ferricyanide in a basic buffer to produce a red colored complex.

Absorbance of this complex is measured spectrophotometrically at 505 nm and the concentration determined by the application of Beers Law. The reagent 4AAP reacts with ortho and meta-substituted phenols. However, certain para-substituted phenols (such as alkyl, aryl, nitro, benzyl, nitroso, or aldehyde substituted groups) do not react with 4AAP and are not detected by this method.

Sample Preservation and Storage:

Samples should be acidified at the time of collection to a pH <2.0 using concentrated sulfuric acid. Store samples in glass containers at 2° to 6°C. The sample is stable for 28 days.

Interferences:

Low readings may be obtained from chlorinated water or wastewater samples due to the oxidation of phenolic compounds. This is eliminated in potable waters by adding sodium sulfite. Other oxidizing agents are stabilized by treating with excess ferrous ammonium sulfate.

Sulfur-containing compounds may distill over and produce erroneously high results. These interferences are eliminated by acidifying the sample to a pH <4.0 with concentrated H_2SO_4, and aerating briefly by stirring and adding $CuSO_4$ (1 g/l). The acidification and copper sulfate also help inhibit microbial degradation.

Background contamination from plastic containers and plastic tubing is eliminated by using glass containers and Kel-F tubing.

CONFIDENTIAL

CONFIDENTIAL

Apparatus:

An Alpkem automated flow analyzer (or equivalent) is required for this method. The Alpkem flow analyzer consists of the following parts:

1. Alpkem Automatic Sampler (Model 301) or equivalent
2. Alpkem Proportioning Pump (Model 502) or equivalent
3. Pump tubes and pump tube harness
4. Power distribution Unit (Model 509) or equivalent
5. Distillation Module (Model 515) and flash distillation head, or equivalent
6. Cartridge Heater/Holder (Model 503) or equivalent
7. Monochromator and Photodiode Detector with 3 mm flow cell (Model 510) or equivalent
8. Alpkem Phenol Super Cartridge or equivalent
9. 386 PC with analog to digital (A/D) convertor and Alpkem SoftPac™ Software

Reagents:

NOTE: All chemicals used must be ACS reagent grade unless otherwise noted.

See SOP-QA-110 for the appropriate labeling and documentation of reagent and standard preparation.

CONFIDENTIAL

1. Deionized water (no surfactant)

2. Dowfax start-up solution:

Deionized water	1000 ml
Dowfax 2A1	2 ml

To a 1 liter glass bottle containing approximately 800 ml of deionized water, add 2 ml of Dowfax 2A1. Fill the bottle with deionized water and cap. Shake well before using. Prepare daily.

3. Distillation reagent:

85% Phosphoric acid (H_3PO_4)	100 ml
Deionized water, q.s.	1000 ml

Add 100 ml of 85% H_3PO_4 to approximately 800 ml of deionized water in a one liter volumetric flask. Dilute to one liter. Store at room temperature. Prepare monthly. Indicate the date of preparation on the bottle label.

4. Buffered Potassium Ferricyanide:

Potassium Ferricyanide [$K_3Fe(CN)_6$]	2.0 g
Boric acid (H_3BO_3)	3.2 g
Potassium Chloride (KCl)	3.8 g
Sodium Hydroxide (NaOH)	
Deionized water, q.s.	1000 ml
Dowfax 2A1	2.0 ml

Dissolve 3.2 g of boric acid, 2.0 g Potassium Ferricyanide, and 3.8 g Potassium Chloride in 800 ml of deionized water in a one liter volumetric flask. Adjust the pH to 10.3 using sodium hydroxide. Dilute

to one liter and add 2.0 ml of Dowfax 2A1. Store at room temperature. Prepare weekly. Indicate the date of preparation on the bottle label.

5. 4-Aminoantipyrine:

4-Aminoantipyrine ($C_{11}H_{13}N_3O$)	.1625 g
Deionized water, q.s.	250. ml

Dissolve 0.1625 ± .0005 g of 4-aminoantipyrine in 200 ml of deionized water in a 250 ml volumetric flask. Dilute to the mark with deionized water. Prepare daily.

6. Calibration Stock Standard, 1000 mg/l phenol:

Phenol	1.000 g
85% Phosphoric acid (H_3PO_4)	0.5 ml
Copper sulfate ($CuSO_4$)	1.0 g
Deionized water, q.s.	1000. ml

Dissolve 1.000 g copper sulfate in 500 ml of deionized water in a 1000 ml volumetric flask. Add 0.5 ml of phosphoric acid to the flask. Dissolve 1.00 g of phenol in the flask and dilute to 1000 ml with deionized water. Store the standard in an amber glass bottle at 2° to 6°C. This standard should be prepared fresh every six months. Indicate the date of preparation on the bottle label.

CAUTION: Wear gloves during the procedure and avoid skin contact. Also avoid inhaling fumes.

7. Quality Control Stock Standard, 1000 mg/l phenol: Use the same procedure as for the Calibration Stock Standard using phenol from a different source.

CONFIDENTIAL

CONFIDENTIAL

Calibration Standards:

1. Working Standard A, 10 mg/l phenol - Pipette 1 ml of the calibration stock standard into a 100 ml volumetric flask containing 90 ml of deionized water and 15 to 20 drops concentrated sulfuric acid. Dilute to volume. Prepare daily.

2. Using working standard A, prepare the following calibration standards in 100 ml volumetric flasks with 15 to 20 drops concentrated sulfuric acid. Prepare daily.

Standard Name	ml working standard A	mg/l Phenol
S6	3.5	0.350
S5	2.0	0.200
S4	1.0	0.100
S3	0.5	0.050
S2	0.2	0.020
S1	0.05	0.005

Quality Control Standards:

1. Working Standard B, 10 mg/l phenol - Pipette 1 ml of the quality control stock standard into a 100 ml volumetric flask containing 90 ml of deionized water and 15 to 20 drops concentrated sulfuric acid. Dilute to volume. Prepare daily.

2. Using working standard B, prepare the following quality control standards preserved with 15 to 20 drops concentrated sulfuric acid. Prepare daily.

Standard	ml Working Standard B	Final Volume (ml)	mg/l Phenol
ICV,CCV	3.0	200	0.150
CRDL	0.05	100	0.005

CONFIDENTIAL

Safety Precautions:

Normal laboratory safety practices must be followed. Special care should be taken in working around the heating bath and distillation head because the danger of being burned by escaping steam or hot surfaces. The phenol standard solutions, especially the stock standards, are toxic and should be handled accordingly. Take care to avoid skin contact. Thoroughly rinse the area with water if skin contact occurs. Because of the hazardous nature of some samples, care should also be taken to avoid breathing the vapors of such samples. Discard or send for repair any glassware that is chipped, flawed, or broken.

Procedure:

1. Set up the Alpkem flow analyzer for phenol according to Figure 1 and turn on the power to all units.

2. Set up the pump, the monochromator detector, the sampler, and computer, using Table I as a guide.

3. Turn on the condensation head cooling water. Adjust the distillation heater in the range 160° to 175°C. When the temperature reaches 150°C, begin pump flow (see step 4.).

4. Place the 4 AAP reagent line and Potassium Ferricyanide buffer reagent line in the start-up solution (2 ml of Dowfax 2A1 per liter of deionized water). Place all other reagent lines in deionized water.

5. When the distillation heater reaches temperature (160° to 175°C), insure that:

   a. No liquid is accumulating in the reflux bowl of the distillation head.

b. The condensate appears as a smooth sheen on the inner wall of the condensation column.

c. The condensation side of the head feels cool to the touch.

NOTE: If a. and b. above are not occurring, adjust the temperature accordingly on the distillation unit until a. and b. occur (not to exceed 180°C).

6. After a stable baseline (505 nm) has been obtained, place the reagent lines in the appropriate reagents and pump the system for 9 to 10 minutes to allow the baseline to stabilize. Autozero the detector after the baseline has stabilized.

7. Monitor the analog signal from the detector using the Softpac™ Plus Software to insure that the baseline has stabilized.

8. Load the Phenol sample template which is shown in Table II. This template lists the order in which the calibration standards, blanks, and Quality Control standards must be run to calibrate the system and to verify that the analytical system is in control. Sample names can be typed in after position 16 and analyzed, if calibration and QC criteria have been met.

   When the phenol sample template is loaded, the software will request that the channel setup field and standard table be completed. Use the values in Table II to fill out these screens. Save the phenol template using the following numbering system:

CONFIDENTIAL

e.g. 93 290 PH
year Julian day

9. Load the sample tray according to the phenol template list (see step 8); turn on the group number for the detector output using the Alpkem software.

10. Press the start button on the Alpkem sampler and begin acquiring data. The data system recognizes the "Sync" peak and activates data collection. The highest level standard is normally placed in the "Sync position."

11. Following the detection limit standard (.005 mg/l phenol CRDL) in position 15, load samples with the appropriate laboratory control standard and preparation blank. A check standard (CRV) of 0.150 mg/l phenol and continuing calibration blank (CCB)(B) must be run after every 10 injections.

    Label all cups as they are loaded on the autosampler to avoid confusion with the databook and/or sample table.

12. After the calibration standards have been analyzed, check the linearity of the calibration curve. The correlation coefficient must be greater than 0.995 for the curve to be valid and for any sample data to be reported.

13. If any sample peak is greater than full scale, dilute the sample with deionized water and analyze the dilution to obtain a peak which is within the calibration range.

CONFIDENTIAL

14. Shut-down procedure. Run a check standard and blank. Shut off the autosampler. Place all reagent lines and sample line in deionized water and pump for 20 minutes. Turn off the distillation head and allow the system to pump with deionized water for 20 minutes. Remove all sample lines from the water and pump the system dry. Turn off the proportioning pump and the water coolant to the distillation head.

Calculation:

The data system automatically prepares a standard curve by plotting peak heights of standards against their concentration values and computes the concentrations of the samples (the raw result) by comparing sample peak heights with the standard curve.

If the sample was not distilled, simply apply any dilution factors that may have been used against the raw result to determine the final result (this can be done automatically if the dilution factor is placed in the sample table):

$$\textit{Final result} = \textit{raw result} \times \textit{dilution factor}$$

<u>Waters:</u>

$$\textit{Final result} = \frac{\textit{raw result} \times \textit{dil. factor} \times 500\ \textit{ml}}{\textit{ml of sample distilled}}$$

<u>Solids:</u>

$$\textit{Final res.}(\textit{mg/kg}) = \frac{\textit{raw result} \times \textit{dil. factor} \times 0.5\ \textit{l} \times 1000}{\textit{g of sample distilled}}$$

CONFIDENTIAL

Quality Control:

1. A calibration curve of six standards ranging from 0.005 to 0.350 mg/l (ppm) and a blank will be run at the beginning of every run. The correlation coefficient of the curve must be greater than 0.995. If this is not met, the curve is invalid and must be rerun.

2. An initial calibration verification standard (ICV) shall be run immediately after every calibration. The acceptable range is ± 10 % of the true value (0.150 mg/l). If the ICV does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

3. An initial calibration blank (ICB)(B) shall be run after the ICV. The acceptable result is < the limit of quantitation. If the ICB does not meet this requirement, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

4. A detection limit standard (CRDL) (0.005 mg/l phenol) should be run after every calibration. The acceptable range is ±50% of the true value. If this criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

5. A batch shall contain no more than 20 samples.

6. A laboratory control standard (LCS) shall be prepared and analyzed with every batch or each day samples are prepared (not to exceed 20 samples). The acceptable

CONFIDENTIAL

range is ± 20% of the true value (0.200 mg/l). If the LCS does not meet this acceptance criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

7. A batch blank (PB) shall be prepared and analyzed every batch or each day samples are prepared or analyzed (not to exceed 20 samples). An acceptable result is < the limit of quantitation. If the batch blank does not meet this criterion, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

8. A duplicate shall be prepared and analyzed for every batch (not to exceed 20 samples). An acceptable result is ≤20% relative percent difference (RPD). If the acceptance criterion is not met, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

9. A spike shall be prepared and analyzed for every batch (not to exceed 20 samples). An acceptable result is 75% to 125% recovery. See SOP-IC-004 for handling outliers and the corrective action which must be taken.

10. A check standard (CCV) and a blank (CCB) must be run every 10 injections. The acceptable range for the CCV is ± 10% of the true value (0.150 mg/l). An acceptable CCB result is less than the limit of quantitation or less than 10% of the sample concentration. If a CCV

CONFIDENTIAL

and/or a CCB does not meet the requirements, see SOP-IC-004 for handling outliers and the corrective action which must be taken.

CONFIDENTIAL

NO434.W51
IC Methods #1
043093

Prepared by: John L Snyder Date: 5/3/93

Approved by: Samuel G. Kelser Date: 5-10-93

Approved by: Linda L Saad Date: 5-12-93

Table I

Alpkem Autoanalyzer
Phenol Setup Parameters

Pump			
Speed	Percent	40	
Tubes	Sample	yel/blu	
	Air	orn/yel	
	Dist. reagent	orn/wht	
	4-AAP	orn/wht	
	Ferricyanide buffer	orn/wht	
	Resample	yel/yel	
	Sampler wash - DI water	pur/wht	
	Buffler pull-off	orn/wht	
	Nonvolatile waste	red/red	
Detector			
510	Wavelength	505 nm	
	Rise time	10 sec.	
	Range	0.02 AUFS	
Sampler			
	Rate	33/hr.	
	Sample time	45 sec.	
	Wash time	65 sec.	
Computer			
	Input voltage	0 to +1 V	
Valve			
	Injection loop	N/A	

CONFIDENTIAL

CONFIDENTIAL

Table II

Table Name: Phenol

Cup#	Sample ID	Dil.	Wgt.	Cup#	Sample ID	Dil.	Wgt.
1	SYNC	1	1	2	rinse	1	1
3	rinse	1	1	4	B	1	1
5	S1	1	1	6	S2	1	1
7	S3	1	1	8	S4	1	1
9	S5	1	1	10	S6	1	1
11	rinse	1	1	12	B	1	1
13	ICV	1	1	14	B	1	1
15	CRDL	1	1	16		1	1
17		1	1	18		1	1
19		1	1	20		1	1

Channel Setup

Channel #	=	[2]	
Channel Name	=	Channel 2]	
Start Ignore Time	=	[0]	
Initial Baseline Lag Time	=	[110]	
Final Baseline Lag Time	=	[110]	
Corrections Code	Y/N	[Y]	
Cycle Time	=	[110]	
Collection Rate	=	[2]	Points/Sec.
Channel Off-Scale Warning	=	[On]	
Off-Scale Warning Limit	=	[100]	
Channel Zero Scale Warning	=	[On]	
Invert Raw Data?	Y/N	[No]	

Standards Table

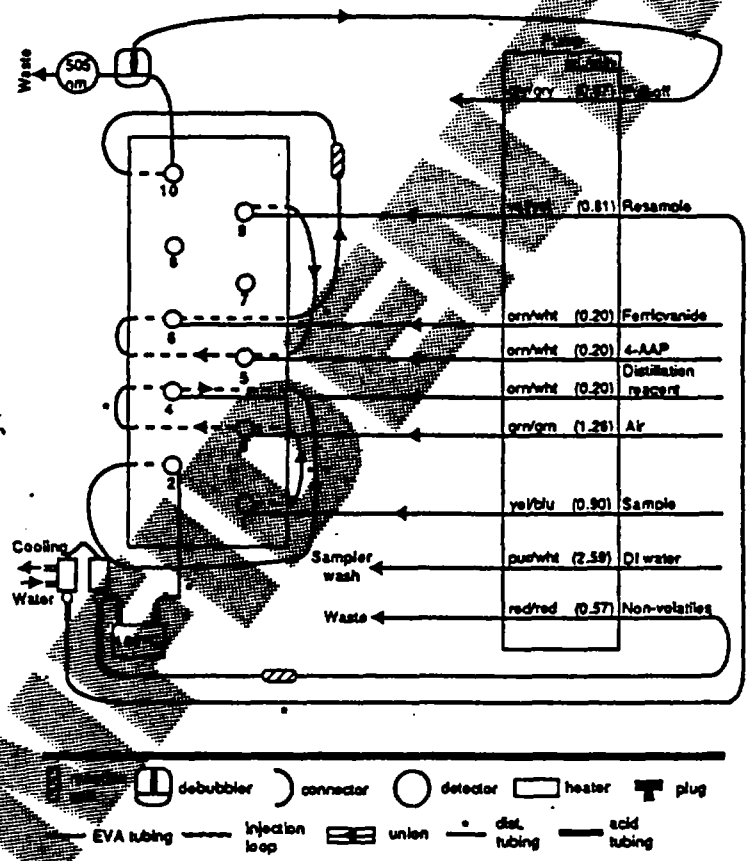
Calibration Code: 1 Channel #: 2
Units: mg/l
Calibration Mode: CF

S1	.005	S11	0
S2	.020	S12	0
S3	.050	S13	0
S4	.100	S14	0
S5	.200	S15	0
S6	.350	S16	0
S7	0	S17	0
S8	0	S18	0
S9	0	S19	0
S10	0	S20	0

Figure 1

FLOW SOLUTION FLOW DIAGRAM

Method:	*Phenol*
Technique:	*SFA, with On-line Distillation*
Range:	*2 - 500 µg/L*
EPA Number:	*420.2*
Application:	*Water and Wastewater*
Date:	*January 1992*
Pump Speed:	*40*



CONFIDENTIAL

ALPKEM
A Perstorp Analytical Company

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

RAW DATA LOGBOOK
PHENOLS
ANALYSIS #434

BATCH NO. ☐☐☐☐☐ ☐☐☐ ☐☐☐ ☐☐☐☐☐

Date: _____

Name: _____

Sample	Dilution Factor	Distilled wt/vol	Chart Reading	Peak Conc (mg/l)	Result (mg/kg - mg/l)	Comments

CONFIDENTIAL

VERIFIED BY: _____ DATE: _____ PAGE: _____

Analytical Procedure Validation
for Analysis #434
Initiated Date: 04/21/93
Effective Date: MAY 13 1993

CONFIDENTIAL

Analytical Procedure Validation
Alpkem Flow Analyzer

An Alpkem automated flow analyzer was purchased to replace the Technicon Autoanalyzer II, which is currently used in the colorimetric method to determine phenol in water and soil samples (Lancaster Laboratories, Inc. Method 434). The Alpkem system was purchased to increase sample throughput and because of its automated data collection system. The phenol method on the Alpkem and the Technicon method are both segmented flow analysis with flash distillation and colorimetric detection (505 nm). The Alpkem equipment operates under the same principles as the Technicon Autoanalyzer II, however, improvements have been made in pump design, cartridge configuration, the cartridge heating system, the UV/visible detector and the data collection system. The chemistries used in the colorimetric determination of phenol are identical on both the Alpkem and Technicon systems. All samples are still distilled prior to the automated analysis.

Listed below are changes which have been made to the phenol method to accommodate the Alpkem flow analyzer.

1. The same reagents are used for both methods, although the boric acid buffer and the ferricyanide reagent have been incorporated into a single reagent. Changes have been made in reagent concentrations and the size of the pump tubing used to introduce the reagents.

2. The dynamic range of the calibration has been extended from 0.005 - 0.200 mg/l to 0.005 - 0.350 mg/l. An additional sixth standard has been added to the calibration.

3. Data collection on the Alpkem flow analyzer occurs through an analog to digital convertor. The digital data is processed by the SoftPac™ Plus Software which automatically sets the baseline and identifies peaks, measures peak height, calculates the calibration curve, and calculates the phenol concentration in the samples from the curve. Previously, the analog signal from the Technicon AAII was recorded by a strip chart recorder and data processing was manual.

Experimental:

The following steps were taken to validate the Alpkem colorimetric determination of total phosphorus.

1. Calibration curves were generated and evaluated for linearity.

2. Detection limit studies were conducted to determine the MDL of phenol on the Alpkem system.

3. Replicate determinations were made of a certified Laboratory Control Sample (LCS) to determine precision and accuracy. Method blanks were analyzed with these replicates to verify system cleanliness.

4. Real-world samples analyzed on the Technicon AA II were analyzed on the Alpkem system and the results were compared.

5. Calculations of the calibration curves and sample results on the SoftPac™ Plus software were verified.

CONFIDENTIAL

Results and Discussion:

1. A calibration curve generated from six standards (0.005, 0.020, 0.050, 0.150, 0.200, and 0.350 mg/liter) is shown in Figure 1. The slope intercept form of this curve is y = 9363.85x - 16.33. Linearity, as demonstrated by the correlation coefficient 0.9999, exceeds the QC requirement (0.995) of the Technicon method (434).

2. The results of the phenol MDL study are shown in Table I. The calculated MDL based on seven replicates at the 99% confidence level was 0.0012 mg/liter. A limit of quantitation of 0.005 mg/liter can be maintained.

3. An average phenol recovery of 96% was obtained for 6 replicates of the LCS standard as shown in Table II. Recovery was within the ± 20% QC window and the RSD was less than 1%.

4. Seven water samples carried through the phenol distillation were analyzed for phenol using both the Technicon and Alpkem analyzers. A plot made of the Technicon result versus the Alpkem result is shown in Figure 2. If results were exactly equal, the slope of this line would equal 1.0, the intercept would equal 0, and the correlation coefficient would equal 1.0. The ideal line is drawn on Figure 2. The equation of the "best-fit" line obtained by plotting the Technicon and Alpkem data was Y = 1.0055x + 0.00348 and the correlation coefficient was 1.000. The point for sample 1952886 is included in this calculation, but is not shown on the graph in Figure 2.

CONFIDENTIAL

5. The calibration curve shown in Figure 1 was checked against the statistics program in the Lancaster Laboratories, Inc. (LLI) VS7310 computer. The linear calibration curve, as shown in Table III, was calculated when the concentration and peak height of the six standards were entered as the x and y values, respectively, under "general case plot" in the "Statistics" program. The equations of the calibration curves generated by the Alpkem software and the LLI Statistics program are the same.

The Alpkem calculation of a sample result was hand checked. This calculation is shown below.

Sample	1952886
Peak No.	65
Dilution factor	1000
Weight	1.0
Height	1585

Calibration curve:

$$y = 9363.847x - 16.33024$$

y = height
x = concentration

Substituting in the height of the peak for y,

$$1585 = 9363.847 - 16.33024$$

Solving for x,

$$1601.3302 = 9363.847$$

x = 0.171012

CONFIDENTIAL

Multiplying by 1000 (dilution factor) = 171.012

The data system returned the value 171.012

CONFIDENTIAL

NO434.W51
IC Methods #1
042193

Prepared by: John L Snyder Date: 4/29/93

Approved by: Samuel G. Hilce Date: 4.27.93

Approved by: M Laurse Hess Date: 4/28/93

 MAY 13 1993

Table I

CONFIDENTIAL

Phenol Minimum Detection Limit Study 4/6/93

Sample Volume(mL)	500
Distillation Vol.(mL)	500
Spike Conc.(mg/L)	0.005

Trial	Peak Ht.	Sample Con.(mg/L)
Blank	0	0.00174
MDL 1A	52	0.00730
MDL 2A	50	0.00708
MDL 3A	49	0.00698
MDL 4A	45	0.00655
MDL 5A	49	0.00698
MDL 6A	45	0.00655
MDL 7A	42	0.00623
	Aver. Rec.	136%
Standard Deviation	3.61	0.00037
Students t (99%)	3.143	3.143
Calculated MDL	11.02	0.00118

Table II

Phenol - Replicate Analysis of Laboratory Control Sample

Sample Volume(mL)	500
Distillation Volume (mL)	500
True Value (mg/Liter)	0.200

Trial	Peak Ht.	Concentration mg/L P	Recovery %
Blank	0	0.0017	
LCS 1	1765	0.1902	95%
LCS 2	1777	0.191[illegible]	96%
LCS 3	1796	0.19[illegible]	97%
LCS 4	1780	0.191[illegible]	96%
LCS 5	1784	[illegible]	96%
LCS 6	1751	[illegible]	94%
Average	1776	[illegible]	96%
Standard Deviation	15.7	[illegible]7	
Relative Std Dev. %	0.89%	[illegible]87%	

CONFIDENTIAL

Table III

General Case

4/21/93 10:00 021

Slope: 9363.84685

Y Intercept: -16.33022

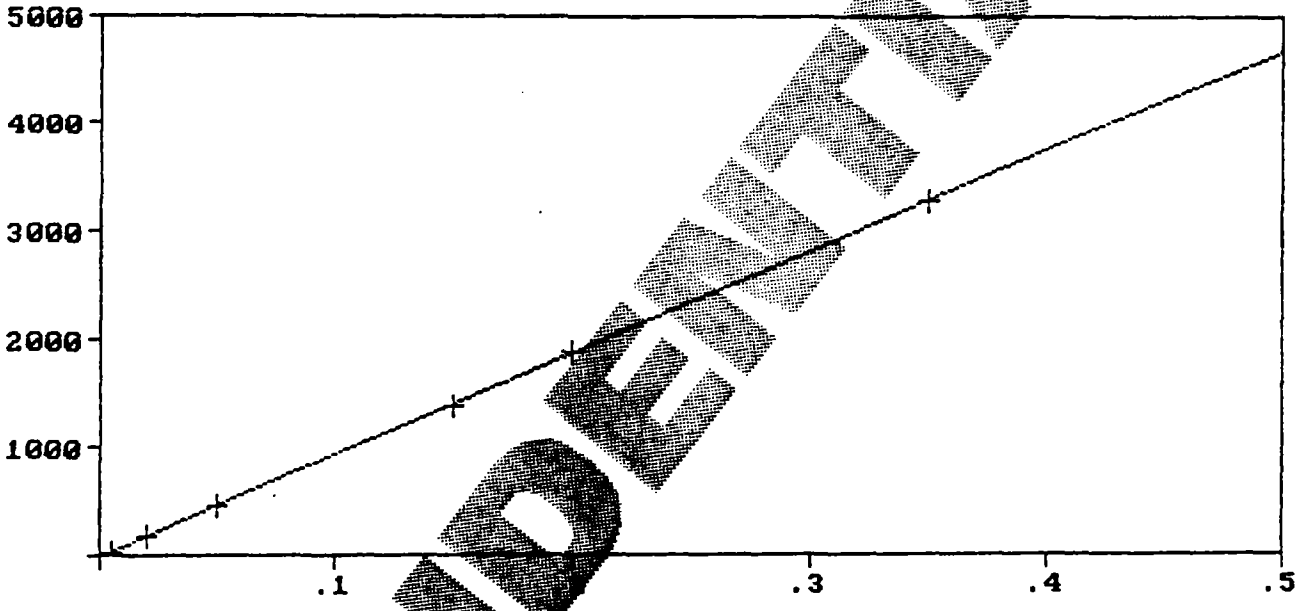
Correlation Coefficient: .999954

Phenol mg/l	Peak height	x Calculated	x Difference	% Difference
.00500	37.00000	.00570	.0007	14.000
.02000	173.00000	.02022	.0002	.999
.05000	443.00000	.04905	-.0010	-2.000
.15000	1373.00000	.14837	-.0016	-1.066
.20000	1875.00000	.20198	.0020	.999
.35000	3258.00000	.34968	-.0003	-.085

CONFIDENTIAL

Figure 1

CHANNEL NAME: 93104PH
RUN NAME: 93104ph
RUN DATE: 04-06-1993
RECALIBRATION #: 1



PEAK NAME: CF
INTERCEPT = -16.33020

CALIBRATION CODE: 1
SLOPE = 9363.847

CORRELATION: .9999538

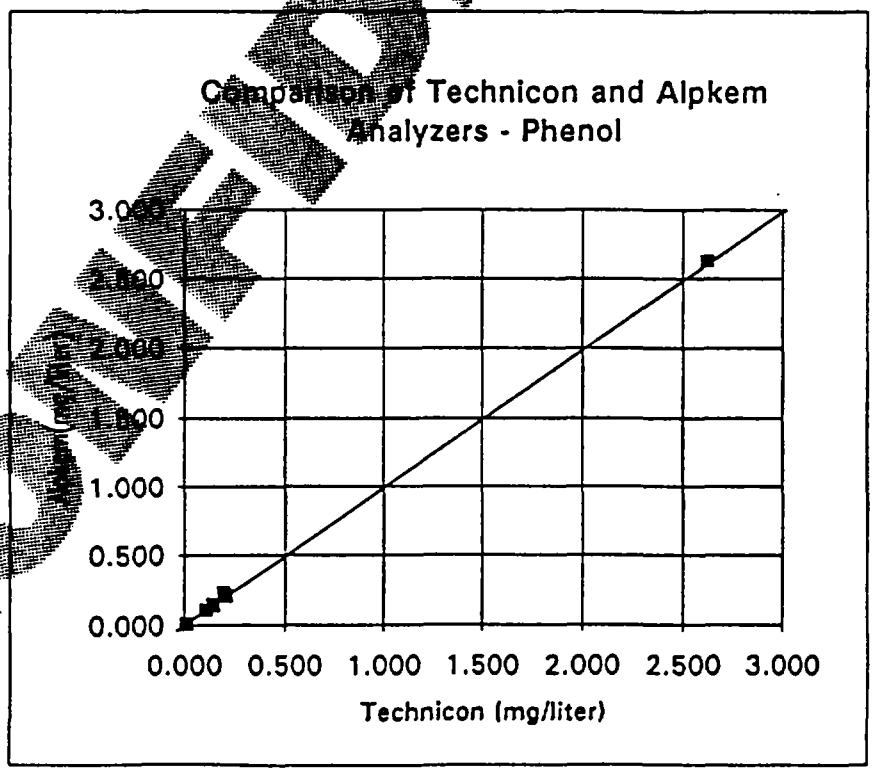
CONFIDENTIAL

Figure 2

CONFIDENTIAL

Comparison of Alpkem and Technicon Results

Sample No.	Technicon x(mg/L)	Alpkem y(mg/L)
1948715	0.010	0.008
1948884	0.144	0.145
1951012	0.110	0.104
1952116	0.195	0.223
1952891	0.204	0.203
1952888	2.620	2.633
1952886*	169.00	189.94



y = 1.0055x + 0.00348 r = 1.000

Ideal ———

"Best Fit" — —

* point not shown on graph, but included in linear regression

Analysis # 273, 354, 1550
Initiated Date: 10/8/85
Effective Date: DEC 1 1992

CONFIDENTIAL

Determination of Total Organic Carbon In Water and Wastewater

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA 600/ Method 415.1, 4-79-020.

2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods #SW-846, Method #9060.

3. Model 700 Total Organic Carbon Analyzer Operating Procedures and Service Manual, O.I. Corporation, 1984.

4. Standard Methods for the Examination of Water and Wastewater, 17th Edition, pp. 5-17 to 5-26, Method 5310 (1989).

Scope:

This method is applicable for the determination of Total Carbon (TC), Total Organic Carbon (TOC), Total Inorganic Carbon (TIC), and Dissolved Organic Carbon (DOC) in water and wastewater samples. The O.I. 700 TOC Analyzer is capable of determining these components individually or simultaneously. The procedures outlined in this method pertain to the O.I. Model 700 Analyzer when it is operated under automated or semiautomated conditions.

The limit of quantitation for samples containing less than 10 mg/l TOC is 0.5 mg/l. The limit of quantitation for samples containing greater than 10 mg/l TOC is 1 mg/l. The normal limit of quantitation for TIC is 1 mg/l. The normal limit of quantitation for TC is 2 mg/l.

High levels of inorganic carbon and high levels of chloride may interfere with the TOC determination. Heterogeneous samples or samples containing large particles (>0.2 mm) may cause poor reproducibility and/or erroneously low results.

Basic Principles:

A sample aliquot of water or wastewater is injected into a teflon digestion vessel at 100°C where metered amounts of phosphoric acid are added. The sample is purged with ultra-pure nitrogen gas to remove inorganic carbon as carbon dioxide and any purgeable organic carbon (POC). The carbon dioxide is carried by the nitrogen stream to a molecular sieve where it is trapped. After the purging cycle is complete, the molecular sieve is rapidly heated to release the CO_2, which is then carried by the nitrogen stream to an IR detector specific for carbon dioxide. The detector produces an analog signal which is proportional to the amount of carbon in the sample. Following the purging of the inorganic fraction, a metered amount of sodium persulfate is added to the digestion vessel. The persulfate oxidizes any organic carbon to carbon dioxide which is carried to and trapped on the molecular sieve. Following the completion of the persulfate digestion, the molecular sieve is heated and the desorbed carbon dioxide is carried to the IR detector by the nitrogen stream. The instrument has the capabilities of performing these determinations simultaneously or individually.

Apparatus:

1. O.I. Corporation Model 700 TOC Analyzer

2. Autosampler (optional)

3. Printer

CONFIDENTIAL

4. 2 stage gas regulator

5. 25 ml sample vials

6. 10 ml test tubes

Reagents and Standards:

NOTE: All chemicals used must be ACS reagent grade unless otherwise noted.

See SOP-QA-110 for the appropriate labeling and documentation of reagent and standard preparation.

1. Ultra-pure nitrogen gas (20 psi)

2. Oxidizing Reagent:

Sodium Persulfate ($Na_2S_2O_8$) 100 g

Dissolve 100 g ± 1 g of sodium persulfate in approximately 500 ml of deionized water in a 1000 ml volumetric flask. Dilute to volume with deionized water. This solution is stable for six months and may be stored at room temperature.

3. Phosphoric Acid (5% w/w):

Phosphoric Acid, 85% (H_3PO_4) 59 ml

Add 59 ml ± 1 ml of 85% H_3PO_4 to about 500 ml of deionized water in a 1000 ml volumetric flask. Mix thoroughly and dilute to volume with deionized water. This solution is stable for six months and may be stored at room temperature.

CONFIDENTIAL

CONFIDENTIAL

4. TOC Stock Standard "B" (500 mg C/l):

Potassium hydrogen phthalate (KHP)	1.063 g
Sulfuric acid, concentrated (H_2SO_4)	20 drops

In a 1000 ml volumetric flask containing about 500 ml of deionized water, add approximately 20 drops of concentrated H_2SO_4. Dissolve 1.063 g (to ± 0.001 g) of KHP in the flask and dilute to volume with deionized water. Store in an amber glass bottle at 2° to 6°C. Prepare monthly.

5. TIC Stock Standard "B" (500 mg C/l):

Sodium bicarbonate ($NaHCO_3$)	0.1750 g
Sodium carbonate (Na_2CO_3)	0.2209 g

Weigh the reagents listed above (to ± 0.0003 g) and transfer to a 100 ml volumetric flask. Dilute to volume with deionized water. Store in the flask at 2° to 6°C. Prepare weekly.

6. Working Standards for Calibration (TIC and TOC):

Pipet a volume of the Stock Standard "B" into the appropriate volumetric flask that contains deionized water that has been acidified with 1 to 2 drops of concentrated H_2SO_4. Prepare daily according to the table below.

NOTE: Do not add H_2SO_4 if preparing a TIC standard.

Standard (mg/l)	Volume Stock Std "B" (ml)	Final Volume (ml)
5.0	2.0	200
25	5.0	100

7. TOC Stock Standard "A" (500 mg C/l):

   Same procedure as preparation of the TOC Stock Standard "B" using KHP from a different source.

8. TIC Stock Standard "A" (500 mg C/l):

   Same procedure as preparation of the TIC Stock Standard "B" using reagents from a different source.

9. Working Standards for linearity check and calibration verification (TIC and TOC):

   Pipet a volume of the Stock Standard "A" into the appropriate volumetric flask that contains deionized water which has been acidified with 1 to 2 drops of concentrated H_2SO_4. Prepare daily according to the table below.

   NOTE: Do not add H_2SO_4 if preparing a TIC standard.

CONFIDENTIAL

Standard (mg/l)	Volume Stock Std "A" (ml)	Final Volume (ml)
75	15	100
50	10	100
25*	10	200
10	2.0	100
7.5	3.0	200
5.0**	5.0	500
1.0	0.2	100
0.5	0.1	100

*Denotes high level CCV (Continuing Calibration Verification)

**Denotes low level CCV (Continuing Calibration Verification)

10. Standards for Total Carbon (TC).

a. Working Calibration Standards

Pipet volumes of TOC Stock Standard "B" and TIC Stock Standard "B" into the appropriate volumetric flask and dilute to volume with deionized water. Prepare daily according to the table below.

Standard (mg/l)	Vol. TOC Stock "B" (ml)	Vol. TIC Stock "B" (ml)	Final Vol. (ml)
5.0	2.5	2.5	500
25	2.5	2.5	100

b. Working Standards for linearity check and calibration verification

Pipet volumes of TOC Stock Standard "A" and TIC Stock Standard "A" into the appropriate volumetric flask and dilute to volume with deionized water. Prepare daily according to the table below.

CONFIDENTIAL

Standard (mg/l)	Vol. TOC Stock "A" (ml)	Vol. TIC Stock "A" (ml)	Final Vol. (ml)
75	7.5	7.5	100
50	5.0	5.0	100
25*	5.0	5.0	200
10	1.0	1.0	100
7.5	0.75	0.75	100
5.0**	2.5	2.5	500
2.0	0.2	0.2	100
0.5	0.1	0.1	200

*Denotes high level CCV (Continuing Calibration Verification)

**Denotes low level CCV (Continuing Calibration Verification)

Safety Precautions:

Normal laboratory safety practices should be followed when performing this method. Extra care should be taken when handling the persulfate reagent (which is a strong oxidant) and unknown samples which could contain toxic or carcinogenic chemicals. Inspect all glassware before use. Discard or send for repair any glassware that is chipped, flawed, or broken.

Sample Collection, Preservation, and Storage:

All samples should be collected in glass containers and stored at 2° to 6°C. Samples for TOC should be acidified with concentrated sulfuric acid to a pH <2. Samples for TC and TIC should not be acidified, but should be collected in glass containers without headspace, stored at 2° to 6°C, and analyzed as soon as possible after they are collected. The holding time for all samples is 28 days. Groundwater samples should be filtered when they are collected.

CONFIDENTIAL

Interferences:

This method is generally free of interferences when normal wastewater and water samples are analyzed. However, high concentrations of chloride can interfere and several other problems can arise from certain sample matrices. These are listed below.

1. Chloride can react with the persulfate which can cause erroneously low TOC results. Samples high in chloride may require that extra persulfate be added and the reaction time be extended. The chlorine gas which is produced is also damaging to the molecular sieve. For these reasons samples should not be preserved with hydrochloric acid.

2. Samples with high levels of inorganic carbon may require extra acid and longer purge time to ensure all the carbonates and bicarbonates are removed from the sample. A smaller sample volume may also be used.

3. Samples with high levels of organic carbon may require that extra sodium persulfate be added and that the reaction time be extended to ensure that all the organic carbon is oxidized by the persulfate. A smaller sample volume may also be used. Spiking the sample with a known amount of carbon and calculating recovery can be used to verify the oxidation efficiency.

4. Heterogeneous samples or samples containing large particles (>0.2 mm) may cause poor reproducibility and/or erroneously low results. Such samples should be thoroughly mixed and homogenized using the polytron blender. If this is not possible, they should be analyzed using the ampule module of the instrument.

CONFIDENTIAL

5. When sequentially analyzing samples, carryover may occur when a sample high in carbon is followed by a sample low in carbon. This will produce erroneously high results for low level TOC samples. This is caused by adsorption of organic carbon on the teflon tubes and overloading of the molecular sieve. If an excessively high level sample is accidentally introduced into the system, it may be necessary to clean the digestion vessel with deionized water before analyzing any more samples. This is accomplished by cycling deionized water and the reagents through the system until TOC values drop to less than the normal detection limit.

Procedure:

A. General

1. Start up the instrument in the following manner:

   a. Turn on the nitrogen gas and adjust the regulator to 30 psi.

   b. Turn on the power to the instrument and printer.

      CAUTION: Make sure that nitrogen gas is flowing or the instrument can be damaged.

   c. Set the detector flow to 5 and the purge flow to 6 on the rotometers on the instrument.

   d. Fill the reagent bottles with oxidizing reagent and 5% phosphoric acid solution.

   e. Prime the acid and oxidant pumps by pressing each button on the control panel five times.

CONFIDENTIAL

2. Install the appropriate sample loop. Use the 1 ml loop for high level analysis (>10 ppm) and the 5 ml loop for low level analysis (<10 ppm).

   a. Ensure the sample loop has been calibrated for the instrument it is being installed on.

   b. Enter the sample volume into the microprocessor.

3. Run system cleanup blanks in the following manner:

   a. Set the system configuration to run blanks in the IC/TOC analysis mode. (Refer to Table III)

   b. Set the IC and OC blank values to "0".

   c. Set the oxidant volume to 5000 ul.

   d. Set the extended reaction time to 5 minutes.

   e. Set the SAMPLE ID NUMBER to "1" and the SAMPLE STOP NUMBER TO "5" AND PRESS "RUN".

B. Operation in the IC/TOC Mode - TOC Analysis

NOTE: The IC/TOC mode is used for all samples that are to be analyzed for TOC. Both TIC and TOC are determined in this mode, but the TIC quantitation is usually performed with the instrument configured for "TIC ONLY" analysis.

1. After system cleanup blanks are run, reset the oxidant volume to 2500 ul and the extended reaction time to "0".

CONFIDENTIAL

2. Run reagent blanks by resetting the SAMPLE STOP NUMBER and pressing "RUN".

   a. Run blanks (minimum of three) until a reproducible "TOC" millivolt response (generally <2 mV variation) is observed.

   b. Average the millivolt readings and enter this value as the "OC" blank.

      NOTE: For an oxidant volume of 2500 ul, a blank of 10 to 20 mV can be expected.

3. Prepare TOC calibration and working standards. Refer to the Reagents and Standards section of this method. Refer to Table I for the required standards.

Table I

Standards for each Sample Loop

1 ml Loop (mg/l)	5 ml Loop (mg/l)
75	10.0
50	7.5
25*	5.0*
10	1.0
7.5	0.5
1	

*Denotes Calibration Concentration

4. Run a single point calibration. Analyze a 5 mg/l standard for low level analysis and a 25 mg/l standard for high level analysis.

   a. Enable the SAMPLE PUMP and SAMPLE LOOP VALVE.

   b. Place the sample inlet line into the standard.

CONFIDENTIAL

c. Set the SAMPLE STOP NUMBER and press "RUN".

d. Run the calibration standard (minimum of three) until a reproducible millivolt response (generally <10 mV variation) is observed.

e. Average the TOC mV readings and enter this value as the standard average in the microprocessor.

f. Calculate and enter the standard mass (ug Carbon). This is calculated by multiplying the sample volume by the calibration standard concentration in ppm.

EXAMPLE: For a 1 ml sample loop and a 25 ppm standard, the standard mass is 25 ug.

g. Press "PRINT". The instrument will automatically calculate the response factor and list the calibration data.

5. Enable the autosampler and turn on the deionized water flow to the wash receptacle.

NOTE: This step not required if injecting samples individually.

6. Inject the following RUN QC:

Low Level (5 ml loop)	High Level (10 ml loop)
Method Blank	Method Blank
0.5 mg/l	1 mg/l
1.0 mg/l	7.5 mg/l
5.0 mg/l	10. mg/l
7.5 mg/l	25. mg/l
10.0 mg/l	50. mg/l
ICB (init cal blk)	75. mg/l
	ICB (init cal blk)

CONFIDENTIAL

7. Samples may now be injected with a check standard (CCV-N) and blank (CCB) before and after every ten samples. Be sure to set the SAMPLE STOP NUMBER.

C. Operation in the IC/TOC Mode - TIC Analysis

NOTE: This is an abbreviated IC/TOC cycle and is activated via the system configuration key. The cycle requires only three minutes to complete. Samples that are to be analyzed for TIC should not be left open to the atmosphere because of the changes that can occur in carbon dioxide equilibria. Inorganic carbon standards should be used for calibration and QC when determining inorganic carbon.

1. After the cleanup blanks are run, reset the oxidant volume to 2500 ul and the extended reaction time to "0".

2. Set the system configuration to "ANALYZE TIC ONLY".

3. Run reagent blanks (minimum of three) until a reproducible TIC millivolt response (generally <2 mV variation) is observed. Enter the average as the "IC" blank.

4. Prepare TIC calibration and working standards. Refer to the Reagents and Standards section of this method. Refer to Table II for required standards.

CONFIDENTIAL

Table II

Standards for each Sample Loop

1 ml Loop (mg/l)	5 ml Loop (mg/l)
75	10.0
50	7.5
25*	5.0*
10	1.0
5	0.5
1	

*Denotes Calibration Concentration

5. Run a single point calibration. Refer to step 4 in the previous section (TOC Analysis). Use TIC standards instead of TOC standards.

6. Enable the autosampler and turn on the deionized water flow to the wash receptacle.

   NOTE: This step not required if injecting samples individually.

7. Inject the following Run QC:

Low Level (5 ml loop)	High Level (1 ml loop)
Method Blank	Method Blank
0.5 mg/l	1 mg/l
1.0 mg/l	5 mg/l
5.0 mg/l	10 mg/l
7.5 mg/l	25 mg/l
10.0 mg/l	50 mg/l
ICB (init cal blk)	75 mg/l
	ICB (init cal blk)

8. Samples may now be injected with a check standard (CCV-N) and blank (CCB) before and after every ten samples. Be sure to set the SAMPLE STOP NUMBER.

CONFIDENTIAL

D. Operation in the IC/TOC Mode - TC Analysis

NOTE: This is an abbreviated IC/TOC cycle and is enabled via the system configuration mode. In this cycle, the persulfate and acid are added at the same time and the system is purged to capture the resulting carbon dioxide. This cycle requires 6.5 minutes to complete.

1. After the cleanup blanks are run, reset the oxidant volume to 2500 ul and the extended reaction time to "0".

2. Run reagent blanks by resetting the SAMPLE STOP NUMBER and pressing "RUN".

   a. Run blanks (minimum of three) until reproducible "TOC" and "TIC" millivolt responses (generally <2 mV variation) are observed.

   b. Average the individual millivolt responses for TIC and TOC and enter these values as the "IC" Blank and "OC" Blank.

3. Set the system configuration to "ANALYZE TC ONLY".

4. Prepare TC calibration and working standards. Refer to the Reagents and Standards section of this method. Refer to Table II for required standards.

5. Run a single point calibration. Refer to step 4 in the TOC Analysis section of this method. Use TC standards instead of TOC standards.

6. Enable the autosampler and turn on the deionized water flow to the wash receptacle.

CONFIDENTIAL

NOTE: This step not required if injecting samples individually.

7. Inject the following Run QC:

Low Level (5 ml loop)	High Level (1 ml loop)
Method Blank	Method Blank
0.5 mg/l	2 mg/l
2.0 mg/l	5 mg/l
5.0 mg/l	10 mg/l
7.5 mg/l	25 mg/l
10.0 mg/l	50 mg/l
ICB (init cal blk)	75 mg/l
	ICB (init cal blk)

8. Samples may now be injected with a check standard (CCV-N) and blank (CCB) before and after every ten samples. Be sure to set the SAMPLE STOP NUMBER.

E. Analysis for Dissolved Organic Carbon (DOC)

Samples for DOC should be filtered through a 0.45 um pore diameter filter and then analyzed as TOC. A deionized water method blank should be filtered to check for the filter's contribution to DOC. Filtration can result in the loss or gain of DOC depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it.

CONFIDENTIAL

Quality Assurance:

1. A linearity check of five (for low level) or six (for high level) standards, as listed in Tables I and II, and a deionized water method blank shall be run after every instrument calibration. The acceptance criterion for the standards is ± 10% of the true value. The acceptance criterion for the detection limit standard is ± 50% of the true value. The method blank must be less than the detection limit.

2. An initial calibration blank (ICB) shall be run after every calibration. The acceptable result is < the limit of quantitation.

3. A check standard (CCV-N) and blank (CCB) must be run before and after every ten injections. The acceptable range for the CCV-N is ± 10% of the true value. An acceptable CCB result is < the limit of quantitation or <10% of the concentration of the sample(s). If a CCV-N does not meet the acceptance criterion, all samples since the last compliant CCV-N and all samples before the next compliant CCV-N must be repeated. If a CCB does not meet the acceptance criterion, all samples since the last compliant CCB and all samples before the next compliant CCB must be repeated.

   A 5 mg/l standard should be used as the low level CCV and a 25 mg/l standard should be used as the high level CCV.

CONFIDENTIAL

4. A batch blank (PBW) shall be analyzed every batch. An acceptable result is < the limit of quantitation. If the batch blank does not meet this criterion, it should be rerun twice. If the blank result is still > the limit of quantitation, only sample results >10x the blank value may be reported. Those sample results <10x the blank value must be reprepared and reanalyzed.

   NOTE: The batch blank analyzed is a reagent blank.

5. A batch shall contain no more than 20 samples.

6. A Laboratory Control Standard (LCSW) shall be analyzed every batch. A 5 mg/l standard should be used as the LCSW for low-level analysis and a 25 mg/l standard should be used as the LCSW for high-level analysis. The acceptable range is ± 10% of the true value.

7. A duplicate shall be analyzed every ten samples. The acceptance criterion is <20% relative percent difference. If one or both of the values is below 5x the limit of quantitation, the acceptance criterion is ± the limit of quantitation. If the acceptance criterion is not met, the sample must be reanalyzed.

8. A spike shall be analyzed every batch. An acceptable result is 75 to 125% recovery. If the spike recovery does not meet the acceptance criterion and the sample exceeds the spike concentration by a factor of four or more, no further action needs to be taken. If the

CONFIDENTIAL

acceptance criterion is not met and the sample concentration does not exceed the spike concentration by a factor of four or more, the spike should be repeated.

NO273.W51
IC METHODS #3
112592

CONFIDENTIAL

Prepared by: [signature] Date: 11/25/92

Approved by: [signature] Date: 11/25/92

Approved by: Linda L Saad Date: 11/30/92

Table III

Instrument Settings:

	IC/TOC Mode	TC	TIC
Configuration:			
Acid pump	on	on	on
Oxidant pump	on	on	on
*Sample pump	on	on	on
Auto run	on	on	on
Auto print	on	on	on
**Autosampler	on	on	on
Autostart override	off	off	off
Analyze TIC	off	off	on
Analyze TC	off	on	off
POC only	off	off	off
Temperatures:			
Primary trap	200	200	200
Digestion vessel	100	100	100
Volume:			
Acid	500 ul	500 ul	500 ul
Oxidant	2500 ul	2500 ul	2500 ul

*Off for Blank

**Off for Blank and Calibration

CONFIDENTIAL

Table III
(Continued)

Instrument Settings:

	IC/TOC Mode	TC	TIC
Cycle Times:			
Sample pump	6 sec + loop size	6 sec + loop size	6 sec + loop size
TIC purge	2 minutes	NA	2 minutes
Desorb TIC	30 seconds	NA	30 seconds
POC purge	NA	NA	NA
Desorb POC	NA	NA	NA
TOC digestion	3 minutes	3 minutes	NA
TOC purge	2 minutes	2 minutes	NA
Desorb TOC	30 seconds	30 seconds	NA
Total Time	8 minutes	6 minutes 30 seconds	3 minutes

Purge flow: 6 on rotometer

Carrier flow: 5 on rotometer

CONFIDENTIAL

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

RUN SHEET
LOW LEVEL TOTAL ORGANIC CARBON ANALYZER, O.I.
ANALYSIS #273
SAMPLE LOOP _____ ml

Date: _____ Analyst: _____

Calibration Constant: _____ Run No. [ ][ ][ ][ ][ ][ ]

Sample	MV Reading	Raw Result mg/l	Trial No.	Batch Number	Comments
BLK					
STD/5					
Rinse					
MB					
ICV/0.5 mg/l					
ICV/1.0					
ICV/5.0					
ICV/7.5					
ICV/10.0					
ICB/RB					

CONFIDENTIAL

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

RUN SHEET
LOW LEVEL TOTAL ORGANIC CARBON ANALYZER, O.I.
ANALYSIS #273
SAMPLE LOOP _____ ml

Date: _____ Analyst: _____ Page: _____

Calibration Constant: _____ Run No. [ ][ ][ ][ ][ ][ ]

Sample	Raw Result mg/l	Calculated Result mg/l	Trial No.	Batch Number	Comments

CONFIDENTIAL

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

RUN SHEET
HIGH LEVEL TOTAL ORGANIC CARBON ANALYZER, O.I.
ANALYSIS #273
SAMPLE LOOP _____ ml

Date: _____ Analyst: _____

Calibration Constant: _____ Run No. E

Sample	Raw Result	Dilution Factor	Final Result	Trial No.	Batch Number	Comments
BLK						
STD/25						
Rinse						
MB						
ICV/1 mg/l						
ICV/7.5						
ICV/10						
ICV/25						
ICV/50						
ICV/75						
Rinse						
ICB/RB						

CONFIDENTIAL

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

RUN SHEET
HIGH LEVEL TOTAL ORGANIC CARBON ANALYZER, O.I.
ANALYSIS #273
SAMPLE LOOP _____ ml

Date: _____ Analyst: _____ Page: _____

Calibration Constant: _____ Run No. [ ][ ][ ][ ][ ][ ]

Sample	Raw Result mg/l	Dilution Factor	Calculated Result mg/l	Trial No.	Batch Number	Comments

CONFIDENTIAL

Analysis #821
Initiated Date: 03/86
Effective Date: SEP 08 1993

CONFIDENTIAL

Sample Preparation of Potable Water and Wastewater for Total Mercury Analysis by Cold Vapor Technique

References:

1. USEPA SW-846 Test Methods for Evaluating Solid Waste, (September 1986), Method 7470.

2. USEPA CLP SOW No. ILM02.1, Exhibit D, Method 245.5 CLP-M, D51-57, modified.

3. USEPA 600/4-79-020 Methods for Chemical Analysis of Water and Wastes, Office of R&D, USEPA-EMSL, Cincinnati, Ohio, March 1979, Method 245.1.

Scope:

This digestion procedure is used to prepare potable water and wastewater samples for measurement of mercury by cold vapor technique.

Basic Principles:

The samples are digested with nitric acid, sulfuric acid, potassium permanganate, and potassium persulfate to oxidize mercury compounds to mercuric ions. Mercuric ions are reduced to mercury metal using stannous chloride. Mercury measurement is performed using the mercury cold vapor technique.

Apparatus and Reagents:

For reagent preparation, shelf life, and storage conditions, see SOP-IO-007, Preparation of Standards and Solutions.

CONFIDENTIAL

1. 100 ml graduated cylinders, or other appropriate graduated cylinders

2. 250 ml Erlenmeyer flasks, or other appropriate Erlenmeyer flasks

3. Nitric Acid, HNO_3, 70.0-71.0%, "Baker Instra-Analyzed" Reagent, 1.428 g/ml, or equivalent

4. Sulfuric Acid, 95.0-98.0%, H_2SO_4, 36N, Fisher, Reagent A.C.S., 1.84 g/ml, or equivalent

5. Potassium Permanganate, $KMnO_4$, "Baker Analyzed" Reagent, A.C.S., or equivalent

6. Potassium Persulfate, $K_2S_2O_8$, "Baker Instra-Analyzed" Reagent, A.C.S., or equivalent

7. Sodium Chloride, NaCl, Fisher, Certified A.C.S., or equivalent

8. Hydroxylamine Hydrochloride, $NH_2OH{\cdot}HCl$, Fisher, Certified A.C.S., or equivalent

9. Potassium Permanganate Solution (5%) - Weigh 50 ± 1 g of potassium permanganate ($KMnO_4$) into a 600 ml beaker. Transfer the $KMnO_4$ into a 1000 ml volumetric flask using deionized water. Dilute to approximately 950 ml with deionized water. Using a stir plate, stir until the $KMnO_4$ is dissolved. Remove spin bar and dilute to volume with deionized water.

10. Potassium Persulfate Solution (5%) - Weigh 25 ± 1 g of potassium persulfate ($K_2S_2O_8$) into a small beaker. Transfer the $K_2S_2O_8$ into a 500 ml volumetric flask using deionized water. Add approximately 250 ml deionized water. Swirl to dissolve the $K_2S_2O_8$. (Gentle heat may be necessary.) Dilute to volume with deionized water.

11. Sodium Chloride/Hydroxylamine Hydrochloride Solution - Weigh 120 ± 1 g of sodium chloride (NaCl) into a 400 ml beaker. Transfer using deionized water, to a 1000 ml volumetric flask. Weigh 120 ± 1 g of hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) into a 400 ml beaker. Transfer, using deionized water, to the 1000 ml volumetric flask containing the NaCl. Add deionized water, swirl to dissolve solids, then dilute to volume with deionized water.

12. Water bath (95° ± 1°C)

13. 100 ml volumetric flasks, or other appropriate class A volumetric flasks

Note: As long as the correct ratios are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Safety Precautions:

Refer to SOP-IO-011, Inorganic Analysis Safety Procedures.

Procedure:

For sample preservation, storage conditions, and holding times, see SOP-IO-001, Preservation and Holding Times for Inorganic Analyses.

CONFIDENTIAL

Shake sample well. Using a 100 ml graduated cylinder, transfer 100 ml of sample (or an aliquot diluted to 100 ml) into a 250 ml Erlenmeyer flask. Add 5 ml of H_2SO_4 and mix.

Add 2.5 ml of HNO_3 and mix. Add 5 ml of 5% $KMnO_4$ solution and mix. Add additional portions of 5% $KMnO_4$ solution (in 5 ml increments), if necessary, until the purple color persists for at least 15 minutes. (Add the same amount of $KMnO_4$ solution to entire digestion batch.) Add 2.5 ml of 5% $K_2S_2O_8$ solution and mix.

Heat for about two hours in a water bath at 95 ± 1°C, or until sample solution volume is 90 ml or less. Remove from heat and cool.

Prior to analysis, add 2 ml of sodium chloride/hydroxylamine hydrochloride solution to reduce excess permanganate (color changed is from purple to colorless). Add reductant in 2 ml increments until $KMnO_4$ is completely reduced. Transfer the solution to a 100 ml volumetric flask, adjust the volume to 100 ml with deionized water, and mix. Reserve for analysis.

Notes: Calibration standards should be digested using the same procedure. For calibration standard preparation instructions, refer to SOP-IO-007 Sections E.1-E.3.

If insufficient sample is submitted to allow using the method stated volume or the sample contains high solids, one may use a smaller aliquot of the sample. If lower than normal limits are requested, samples may be concentrated. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

CONFIDENTIAL

Potassium permanganate, potassium persulfate, and sodium chloride/hydroxylamine hydrochloride solution volumes reflect modifications of the referenced methods. These modifications were approved by USEPA Region III in July 1993. Documentation is on file.

Quality Assurance:

Perform a method blank, sample duplicate, sample matrix spike, and laboratory control sample with every digestion batch (20 samples or less).

CONFIDENTIAL

NO821.W51
IO Methods #1
082793

Prepared by: _____ Date: 8-27-93

Approved by: Eugene K. Abel Date: 9-2-93

Approved by: Kathleen M. Lower Date: 9-7-93

Analysis #1424, 1425, 1426
Initiated Date: 6/9/89
Effective Date: JAN 6 1993

CONFIDENTIAL

Determination of Priority Pollutants in Water

References:

1. USEPA SW-846, 3rd Edition, Method 8270, 12/87.
2. USEPA SW-846, 3rd Edition, Method 3510, 12/87.
3. USEPA SW-846, 3rd Edition, Method 3520, 12/87.
4. Hewlett-Packard Operations Manuals.

Scope:

This method is suitable for the determination of the concentration of semivolatile organic compounds in water. The minimum detection limit for compounds in a solution of methylene chloride is 10.0 ug/ml. The presence of high levels of organic compounds may interfere with the normal detection limit. Interferences may also be caused by solvent contamination, reagent contamination or glassware contamination. These sources of interference may lead to false-positive identification of target compounds in the sample. A method blank must be analyzed to insure that all materials are free of contamination.

Basic Principles:

The sample is extracted by use of either a separatory funnel or a liquid-liquid extractor (see LLI Method #813). The extract is concentrated to a final volume of 1.0 ml and analyzed by GC/MS.

A mixture of organic compounds in methylene chloride is injected onto a 0.25 mm (internal diameter) fused silica capillary column, which is enclosed in a temperature controlled oven. A carrier gas, helium, passes continuously through the column. The GC oven is temperature programmed and the organic mixture separates into its individual components as it moves along the length of the column. This separation is a function of the polarity and boiling point of the individual compounds. The column empties into a mass selective detector. When a compound reaches the detector, it is bombarded by high energy electrons (70eV). This causes the compounds to fragment forming ions. By applying various voltages to plates in the area where the ions are formed, the positive ions are thrust along a quadruple mass analyzer which selects for a given mass fragment at a given time. These selected fragments reach an electron multiplier which amplifies the signals and sends them to a computer which makes storage and manipulation of the data possible. Target compounds are identified on the basis of relative retention times and spectral match to standards which are injected every twelve hours on the same system. Quantitation is achieved by the internal standard method and the response factor of the most recent 50 ppm check standard.

Apparatus:

1. Hewlett-Packard Model 5890 Gas Chromatograph or equivalent

CONFIDENTIAL

2. Hewlett-Packard Model 5970 Mass Selective Detector or equivalent

3. Hewlett-Packard HP-1000 RTE-6-VM or RTE-A Data System or equivalent

Safety Precautions:

The toxicity of each reagent used has not been precisely determined. However, each reagent should be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves.

Procedure:

A. Standard Preparation - These solutions are used to standardize the GC/MS system every twelve hours and are prepared weekly, or more frequently if needed. See SOP-EX-001 for preparation. Calibration standard solutions shall be used for one week or until component degradation is observed.

B. Tuning:

1. Run an autotune by using program MSCAL, or enter appropriate tune file MT0x using program MTUNE, and the appropriate soft keys.

2. Enter profile and spectrum scan. Significant loss of the 502 peak is an indication of a dirty source.

3. Use the ramp parameters (ion focus, entrance lens) to adjust the abundances to closely match the following:

CONFIDENTIAL

a. 69 100%

b. 131 Slightly less than the 219

c. 219 30%

d. 502 0.8%

4. Adjust the mass axis and widths. Adjust until an accurate and repeatable axis and width are obtained.

5. Save the tune as MTOx, where x is defined as the instrument number.

6. Exit the program.

7. Close the calibration valve on the MSD.

8. Using the BEDIT program, create an autosampler run with DFTPP as the first sample; or, using the BAMON program, create a single sample DFTPP run.

9. Set up the run, injecting DFTPP.

10. After the run is complete, check the DFTPP.

a. TR,TUNCHK, >name of data file.

b. If the DFTPP passes specifications (see Table 3), the spectrum and specifications will print. If it does not pass the spectrum will not print, If the DFTPP passes the criteria, standardization may begin. If the DFTPP fails, the tune must be modified and the DFTPP reinjected until passing criteria are obtained.

CONFIDENTIAL

C. Standardization - Standardization is performed by analyzing five levels of standards (20, 50, 80, 120, 160 ppm). The relative standard deviation (%RSD) for each compound should be less than 30%. However, the %RSD for each individual Calibration Check Compound (CCC) must be less than 30% (see Table 4). A system performance check must be performed to insure that minimum average response factors are met before the calibration curve can be used. The system performance compounds are: N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The average RF for these compounds must be at least 0.050. If any of the above criteria are not met, the source of the problem must be found and the system recalibrated.

1. Standardization is accomplished as follows:

   a. Using the BEDIT program, create a BLIST with the standards described above.

   b. Set up the run, injecting the standards.

2. Review the quantitation reports. Use the SEMCHK program to determine if any compounds were missed in quantitation. Use the QAREA program to integrate any missing compounds, or to check the integration of problem compounds.

3. Run CBUPD,CB827x,C to test the calibration.

4. Run CBRPT,CB827x,TRD to generate a hard copy of the results.

CONFIDENTIAL

5. Run CBCHK, CB827x to check the 50 ppm standard vs. the calibration.

6. If all CCC and SPCC compounds meet the specified criteria, update the response factors and retention times in the library by running QCAL, ^50 ppm standard, ID827x and QCAL, ^50 ppm standard, ID827x,T.

7. At this point, the calibration is complete and analysis of samples may begin and continue for twelve hours after the injection of the DFTPP. The RRF of the 50 ng/ul standard from the initial calibration is then used in sample quantitation.

D. Continuing Calibrations - After twelve hours have elapsed from the injection of DFTPP, a time check and calibration check shall be performed.

1. DFTPP must be reanalyzed and meet the criteria described in Table 3.

2. A calibration check standard of 50 ppm is analyzed and checked by using CBCHK, CB827x. All CCC compounds (see Table 4) must have a percent difference of less than 30 (see Calibration section of this method). All SPCC must have a minimum relative response factor of at least 0.050. If the criteria are met, analysis of samples may begin and continue for 12 hours after the injection of the DFTPP. The RRF of the 50 ng/ul continuing calibration standard is then used for sample quantitation. If either of these criteria are not met, a new five-point calibration must be generated.

CONFIDENTIAL

E. Analysis of Samples:

1. Set up a BLIST containing the samples to be analyzed.

2. Internal standard must be added to all samples.

   a. Sonicate an ampule of Supelco internal standard mix for 15 minutes.

   b. Add internal standard mix to each sample for an in-extract concentration of 40 ug/ml. If the sample is subsequently diluted, additional internal standard must be added to maintain the concentration of 40 ug/ml.

   c. Run BAMON.

F. Qualitative Analysis - A compound is identified by comparison of the sample mass spectra with the mass spectrum of a standard of the suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The sample component relative retention item must compare within ± 0.06 RRT units of the RRT of the standard component.

2. All ions in the reference spectra at greater than 10% of the most abundant ion must be in the sample mass spectra.

3. The relative intensities of the ions mentioned in Part F.2. must agree within ± 20% between the sample spectra and the reference spectra.

CONFIDENTIAL

G. Quantitative Analysis - Once a compound has been identified, quantitation will be based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion.

$$\text{Concentration (ug/l)} = \frac{A(x)*I(s)*V(t)}{A(is)*RRF*V(o)*V(i)}$$

where:

A(x) = Area of characteristic ion for compound being measured.

I(s) = Amount of internal standard injected (ng).

V(t) = Volume of total extract, taking into account dilutions.

A(is) = Area of characteristic ion for the internal standard.

RRF = Relative Response factor for the compound being measured.

V(i) = Volume of extract injected (ul).

V(o) = Volume of water extracted (ml).

Calculations:

1. Calculation of the relative response factor (RF):

RF = [A(x) * C(is))]/[A(is) * C(x)]

Where:

A(x) = Area of the characteristic ion for the compound being measured.

A(is) = Area of the characteristic ion for the specific internal standard.

C(x) = Concentration of the compound being measured.

C(is) = concentration of specific internal standard.

CONFIDENTIAL

2. Calculation of the percent difference:

$$\% \text{ Difference} = \frac{RRF(i) - RRF(c)}{RRF(i)} * 100$$

Where:

RRF(i) = Average response factor from the initial calibration.

RRF(c) = Response factor from current check standard.

Quality Assurance:

Each extraction batch will contain a method blank, a background sample, a matrix spike, a matrix spike duplicate and a quality control reference sample.

1. The method blank will be evaluated for each extraction batch. It will be used to determine if there are any interferences from the analytical system, glassware and reagents. The method blank must contain less than or equal to five times the CRQL (Contract Required Quantitation Limits) of the phthalate esters listed in Table 6. All other compounds must contain less than or equal to the CRQL of any single analyte. If these criteria are exceeded, the entire batch must be re-extracted and reanalyzed.

2. The matrix spike/matrix spike duplicate will be analyzed to assess accuracy. Each individual analyte must meet the criteria listed in Table 6. If any analyte falls outside the acceptance criteria, a quality control reference sample must be analyzed for that analyte. If, after analysis of the reference sample, the analyte is still outside the acceptance criteria, the entire batch must be re-extracted and reanalyzed.

CONFIDENTIAL

3. Each sample is spiked with surrogate standards. Acceptance criteria are listed in Table 8. If any surrogate recoveries do not meet the criteria listed in Table 8, the sample should be reanalyzed. If the recoveries still do not meet the criteria, the sample must be re-extracted and reanalyzed.

4. Each sample is spiked with six internal standards. If any one of these internal standard areas differs from the area of the internal standards in the 50 ppm standard by a factor of two (-50% to +100%), the injection must be repeated. If this criteria is not met upon re-injection, the analyst will decide whether to submit the original injection with a comment or to do a dilution. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration, the batch run must be terminated and the analytical system inspected for possible problems.

14241425.W51
EX METHODS #1
122292

CONFIDENTIAL

Prepared by: Marha Ratcliff Date: 1/4/93

Approved by: [illegible] Date: 1/5/93

Approved by: Susan B Shorter Date: 1/5/93

TABLE 3.
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a]

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	40-100% of mass 198
443	17-23% of mass 442

[a]See Referemce 4.

CONFIDENTIAL

TABLE 4.
CALIBRATION CHECK COMPOUNDS

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

CONFIDENTIAL

TABLE 6.
QC ACCEPTANCE CRITERIA[a]

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range p, ps (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27.133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl)ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl)ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl)phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octylphthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26.155

CONFIDENTIAL

TABLE 6.
(Continued)

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for x (ug/L)	Range p, p_s (%)
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.7	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in ug/L.

x = Average recovery for four recovery measurements, in ug/L.

p, p_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 625. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

CONFIDENTIAL

TABLE 8.
SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Low/Medium Water	Low/Medium Soil/Sediment
Nitrobenzene-d_5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
p-Terphenyl-d_{14}	33-141	18-137
Phenol-d_6	10-94	24-113
2-Fluorophenol	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122

CONFIDENTIAL

Analysis #1508, 1265, 1266,
1358, 1384, 1131,
3981, 4592, 5121,
5122, 5222, 5243,
5244
Initiated Date: 12/13/89
Effective Date: APR 26 1993

CONFIDENTIAL

Waters and Wastewaters for Priority Pollutant Volatile Target Compounds

Reference:

USEPA SW-846, 3rd edition, Method #8240, December 1987

Scope:

This method is suitable for the determination of compounds listed in USEPA SW-846 Method #8240 for aqueous matrices. The method also is appropriate for acquiring data files required to perform a search for tentatively identified compounds (TIC) in volatile organics GC/MS analyses. This analysis should be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by GC/MS purge and trap methodologies.

Basic Principles:

A 5 ml sample or a dilution of a sample is placed in a specially designed purge vessel. The sample is purged with an inert gas and the effluent gas passed through a sorbent trap where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and backflushed on to the head of a gas chromatographic column held at the appropriate initial temperature for the column in use. The gas chromatographic column is temperature programmed to separate the volatile compounds which are subsequently detected and identified using mass spectrometric techniques.

CONFIDENTIAL

Apparatus:

1. Gastight micro syringes - 10 ul and larger

2. 5 ml syringe

3. Analytical balance, capable of accurately weighing ± 0.001 g

4. Glassware - Volumetric flasks, class A with ground glass stopper

5. Purge and Trap Device - Consisting of the sample purger, the trap, and desorber; the Tekmar LSC-2 and LSC 2000 or equivalent meets the requirements of this method

6. GC/MS System - The HP 5970 MSD, Finnigan Incos 50, Finnigan 1020/DVA or equivalent GC/MS system meets the requirements for this method

Reagents:

1. Reagent water is defined as water in which an interferant is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory will meet this criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Methanol and other appropriate solvents (i.e., TGDE, CS_2) - For Purge and Trap analysis or equivalent

CONFIDENTIAL

3. Surrogate Standard Spiking Solution - Prepare stock standard solutions for toluene-d8, p-bromofluorobenzene, and 1,2-dichloroethane-d4 in methanol. Prepare the surrogate standard spiking solution from the stock standards at a concentration of 25 ug/ml in methanol. See Item 4 below for the preparation of stock standards.

4. Stock Standard Solutions - Prepared from assayed standard materials or purchased. Stock solutions must be prepared in methanol or an equivalent solvent.

   a. Place about 9.8 ml methanol or an equivalent solvent into a tared 10.0 ml glass stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

   b. Add the liquids using a syringe or pipet by adding two or more drops of the assayed material to the flask being careful that no drop hits the side of the flask. Bring the volume of solvent in the flask to 10.0 ml. Calculate the concentration of the standard.

   c. The gases bromomethane, chloromethane, chloroethane, vinyl chloride, and dichlorodifluoromethane are purchased from an outside vendor and used as received.

   d. The stock standard solutions are stored in teflon sealed screw capped vials at -10° to -20°C. The compound name, concentration, date prepared, and expiration date must appear on the bottle.

   e. Replace stock standard solutions every six months.

CONFIDENTIAL

5. Secondary Dilution Standards - Using the stock standard solutions prepare secondary stock solutions in methanol or an equivalent solvent containing the desired compounds. Table I lists the priority pollutant compounds analyzed under Analysis #1508. These standards are prepared by calculating the volume of each stock standard required to produce a given volume of a mixed working standard with a known concentration of each analyte. The working standard is tested according to SOP-MS-006, Dept. 21. The verified working standard is pipetted into 1.5 ml teflon lined screw capped GC vials and is stored at -10° to -20°C. A designator indicating the standard, month, and day of preparation must be on the standard vials. The designator and the calculations for the working standard preparation are to be recorded in the standards log book. Replace secondary dilution standards every six months.

6. Matrix Spiking Solution - Prepare solutions in methanol or an equivalent solvent that contain the compounds of interest at known concentrations in a similar manner as described above for the secondary dilution standards. These solutions serve as both the matrix spiking solution and the Quality Control Reference stock solution. Matrix spikes also serve as duplicates, therefore, two aliquots of the same sample need to be spiked for analysis with these solutions.

7. Internal Standards Solution - Prepare a solution in methanol or an equivalent solvent containing bromochloromethane, 1,4-difluorobenzene and

CONFIDENTIAL

chlorobenzene-d5 at a concentration of 25 ug/ml using stock standards as described in 4 above. Replace the internal standards solution every six months.

8. 1,4-Bromofluorobenzene (BFB) Standard - Prepare a 25 ug/ml solution of BFB in methanol. Replace this solution every six months.

9. Store all standard solutions at -10° to -20°C.

Preparation of Glassware:

All glassware is washed with soap water, rinsed with tap water, then rinsed with deionized water, and baked in a drying oven at 100°C for at least 4 hours.

Safety Precautions:

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, 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, such as the use of fume hoods, lab coats, and gloves.

Sample Handling:

The samples to be analyzed using USEPA SW-846 Method #8240, should be stored in a refrigerator between 2° and 6°C. The samples should be preserved to a pH of less than 2 in order to prevent degradation of aromatic compounds that may be present in the sample. The recommended preservative is 1+1 HCl, however,

CONFIDENTIAL

samples with other preservatives can be analyzed. In addition, all samples must be analyzed within 14 days of collection.

Procedure:

1. Tune the GC/MS system to meet the criteria in Table II following a 2 ul injection of BFB. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the temperature ramp may be increased and the initial temperature may be different. The BFB tune must be verified every 12 hours.

2. The purge and trap device should have the trap conditioned for 30 minutes at 180° to 200°C at a flow rate of 20 to 40 ml/min prior to initial use.

3. The recommended gas chromatographic operating conditions are listed in the table below:

	Method 8240 Packed Column	**Method 8240 Capillary Column**
Column packing	SP-1000 on Carbopack B	DB-624
Carrier gas	Helium	Helium
Carrier gas flow	30 ml/min	9-10 ml/min
Initial temperature	45°C	35°C
Initial hold time	3 min	5 min
Temperature ramp	8°C/min	10°C/min
Final temperature	220°C	180°C
Final hold time	15 min	2.5 min

CONFIDENTIAL

4. The recommended mass spectrometer operating conditions are listed below:

Ions	Positive
Electron energy	70 volts
Mass range	35 - 300 amu

Scan time: Packed

INCOS Systems: Total Scan Time 1.5 sec

H-P Systems: Number A/D Samples 2^4 (16)
Integration Time/Sample 50 microsec
Total Time 2.3 sec

Scan time: Capillary

INCOS Systems: Total Scan Time 0.7 sec

OWA System: Total Scan Time 1.0 sec

H-P Systems: Number A/D Samples 2^2 (4)
Integration Time/Sample 50 microsec
Total Time 0.6 sec

5. Internal standard calibration consists of analyzing standards at five distinct levels of analyte and producing response factors for each compound. The relative standard deviation of the response factors determine the suitability of the average relative response factor for calculation of the analyte concentration.

CONFIDENTIAL

a. All aqueous matrices should be prepared in the following manner. Remove the plunger from a 5 ml glass "luerlock" syringe and fill the syringe barrel to overflowing with the aqueous matrix. Replace the plunger and compress the aqueous matrix such that no air is trapped in the syringe. Adjust the syringe volume to 5.0 ml. Add an appropriate volume of the surrogate spiking solution and the internal standard solution to the syringe through the syringe tip.

b. Add the spiked aqueous matrix to the purge vessel through the "luerlock" assembly at the top of the impinging assembly. Do these steps quickly to minimize the loss of volatiles from the aqueous matrix and to minimize the possibility of airborne contamination of the aqueous matrix.

c. The suggested Purge and Trap conditions are listed below:

Purge Flow	20-60 ml/min
Purge Temperature	ambient temp.
Purge Time	11 min
Desorb Temperature	160 to 250°C
Desorb Time	1-4 min
Bake Temperature	180 to 220°C
Bake Time	5 to 20 min

d. Empty and rinse the purging chamber twice with reagent water to minimize the possibility of carry-over contamination, prior to loading another sample into the vessel.

CONFIDENTIAL

e. Continue collecting GC/MS data until the end of the GC run.

f. Prepare the calibration standards at appropriate levels; an example of a five point calibration is listed below:

Standard Concentration (ug/l)	Gas Mix (ul)	ul Secondary Dilution Stock Standard	Final Volume (ml)
20	20	4	200
50	25	5	100
100	25	5	50
150	37.5	7.5	50
200	25	5	25

Due to poor purging efficiency or poor chromatography, certain analytes (Table I) require calibration at higher concentrations. To prevent confusion and assure proper calibration, a Theoretical Standard Concentration (TSC) sheet is completed for each calibration (Figure II). The TSC sheet contains the theoretical concentration for each analyte in the calibration at each of the five levels.

g. Each level is analyzed as described above by substituting five milliliters of the calibration standard as the aqueous matrix. Next, tabulate the area response of the characteristic ions against concentration for each analyte, surrogate standard,

CONFIDENTIAL

and internal standard and calculate Relative Response Factors (RRF) for each compound using the calculation below:

$$RRF = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

Where: Ax = Area of the characteristic ion for the analyte

Ais = Area of the characteristic ion of the appropriate internal standard

Cis = Concentration of the internal standard

Cx = Concentration of the analyte to be measured

h. Calculate the average relative response factors for all analytes. Five analytes are checked for minimum average response factors. These compounds are "System Performance Check Compounds" or SPCCs. The compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. Six additional compounds are evaluated on the basis of the relative standard deviation of the RRF values (% RSD). These compounds are known as Calibration Check Compounds (CCCs). The CCCs are 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. Minimum average RRF value for each SPCC compound is 0.30 except for bromoform which is 0.25. The % RSD for the CCC compounds must be less than 30%. Failure to meet either of the criteria for the SPCC and CCC

CONFIDENTIAL

compounds invalidates the calibration and the calibration must be redone after proper corrective action to the system is taken.

i. The RRF values for the 12-hour check standard (50 ug/l) is used to calculate values for method blank and sample analyses. The calibration is valid for 12 hours, at which time a new tune check and a calibration check must occur prior to the analysis of additional samples.

j. The calibration check involves analyzing the 50 ug/l standard and calculating the RRF for each compound. Minimum RRF values as described under the calibration section for the SPCC compounds must be met. Also the percent difference between the initial calibration average RRF and the RRF for each compound in the 50 ug/l check standard are calculated. The percent difference for the CCC compounds must be less than 25%. If any SPCC or CCC compound fails to meet the criteria listed above the calibration is considered invalid. Corrective action must be taken and the calibration redone. If the criteria are met the response factors from the check standard are used for the sample calculations for the next 12-hour period.

6. A method blank is then analyzed as described above in Step 5a through e using reagent water as the aqueous matrix. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. Methylene chloride, acetone, toluene, and

CONFIDENTIAL

2-butanone must not exceed five times the quantitation limit in the blank. All other compounds may not exceed the quantitation limit. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met. Also all surrogate compound recoveries must meet the criteria listed in Table III.

7. Sample analysis

   a. Sample analysis for water and wastewater proceeds as described in Section 5a-e above.

   b. For each set of 20 field samples one sample shall be analyzed in triplicate, spiking each duplicate with each of the Quality Control reference concentrates. Results of the unspiked and spiked duplicates shall be compared to Table IV.

   c. All samples must also meet the surrogate recoveries listed in Table III. If a sample surrogate recovery falls outside the criteria, the sample shall be reanalyzed. If the reanalysis shows the same surrogate results, the sample matrix is assumed to be interfering and the initial results are reported. If the reanalysis meets the recovery criteria, the first analysis is assumed to have been outside of limits due to a laboratory error and the second analysis is reported.

CONFIDENTIAL

8. Data Analysis

a. Sample chromatograms are analyzed both qualitatively and quantitatively. First a determination of the identity of a sample peak as a compound on the priority pollutant list (Table I) is made through the use of computerized analysis. Guidelines for the qualitative determination are as follows:

(1) The relative retention time (RRT) of the sample peak is within 0.06 of the RRT of the most recent check standard.

(2) Each ion with a relative intensity of greater than 10% of the base ion of the mass spectra of the sample must be present in the sample spectrum produced on the same mass spectrometer.

(3) The relative intensities of the ions in the sample mass spectrum must be within 20% of the intensities of the standard mass spectrum.

(4) Peaks greater than 10% relative intensity in the sample mass spectrum but not present in the standard mass spectrum from the same mass spectrometer must be accounted for by the analyst.

(5) If a compound fails any of the criteria listed above but in the judgment of the mass spectral interpretation specialist is a correct

identification, the identification is used and the quantitation of the peak is performed.

b. Quantitation of identified priority pollutant compounds is performed using the equations listed in the Calculations section of this procedure. All calculations should report concentrations in values of ug/l. Any analyte with a calculated concentration above the highest standard must be reanalyzed at a dilution which will bring the concentration in solution within the calibration curve. It is desirable to have the dilution within the top half of the calibration curve but not required.

Calculations:

$$Concentration\ (ug/l) = \frac{(Ax)\ (Is)}{(Ais)\ (RRF)\ (Vo)}$$

Where: Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Amount of internal standard added in nanograms

Vo = Volume of sample purged

RRF = Relative Response Factor from the current calibration check standard (50 ug/l)

CONFIDENTIAL

CONFIDENTIAL

Quality Assurance:

1. The GC/MS system must be tuned to meet the criteria in Table II following BFB injection. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the rate of temperature ramping may be increased and the initial temperature may be different. The BFB tune must be verified every twelve hours.

2. Internal standard calibration requires analysis of a minimum of five levels of analyte concentration. Response factors for each compound are calculated. The average response factors and the relative standard deviation of the response factors determine the suitability of the average relative response factor for calculation of the analyte concentration.

   a. Five compounds are checked for minimum average response factors. These compounds are "system performance check compounds" or SPCCs. The compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum average RRF value for each SPCC compound is 0.30 except for bromoform which is 0.25. If these values can not be attained there is a problem with the purge and trap or the chromatographic system. Corrective action needs to be taken.

   b. Six additional compounds are evaluated on the basis of the percent standard deviation of the RRF values (%RSD). These compounds are known as calibration

check compounds (CCCs). The CCCs are 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. %RSD values for these compounds on the initial calibration must be less than 30.0%. For continuing calibration to be valid the percent difference between the RRF of each CCC in the 50 ug/l check standard and the average RRF for the CCC compounds in the initial calibration must be within ± 25.0%.

3. Abundances for the 3 internal standards must be within +100% and -50% of the abundances of the internal standards of the current calibration check standard (50 ug/l). The retention time for each internal standard must be within 30 seconds of the value for the last calibration check. Values outside these limits indicate a change in system performance or a matrix effect. Samples with internal standard abundances outside of limits are to be reanalyzed. If the reanalysis is within limits, the data from the reanalysis is reported. If the reanalysis confirms the first analysis the sample matrix is interfering and the data from the first analysis should be reported.

4. Surrogate recoveries are calculated for each sample and blank analyzed. Surrogate recovery limits are listed in Table III. If any surrogate is outside of limits, the sample is to be reanalyzed. If surrogates in the reanalysis are within limits, the reanalysis data are reported. If surrogate recoveries are again outside of limits on the reanalysis, the first analysis is reported.

CONFIDENTIAL

CONFIDENTIAL

5. The method blank must meet all the above criteria for internal standard recoveries and surrogate recoveries. In addition the method blank may not contain any target compound above the quantitation limit except that methylene chloride, acetone, toluene, and 2-butanone may be present up to five times the quantitation limit. All method blanks must meet these criteria otherwise the system is considered out of control and corrective action must be taken.

6. The matrix spike and matrix spike duplicate pair are analyzed for each set of 20 field samples. The recoveries and the percent differences of the recoveries for each spiked compound are calculated and compared to the values in Table IV or from the statistical results of multiple analysis of the spiked samples. The results of the matrix spike/matrix spike duplicate must be within the windows outlined in Table IV. For samples with results outside the statistical windows, analysis of a Quality Control Reference Sample is required. The Quality Control Reference Sample is a method blank spiked with each of the matrix spiking solutions. The Quality Control Reference sample must yield recoveries for all analytes within the specification of Table IV. If the Quality Control Reference Sample fails to meet the criteria, the analysis must stop and the cause of the problem found. If the analysis meets the values in the table, the results are reported.

7. All data is reviewed by a data review specialist prior to release of the data from the analytical laboratory with respect to quality assurance and data interpretation. The data review specialist will return

CONFIDENTIAL

data to the analyst for correction as necessary. The specialist will also report the results of the review and corrections on a form to be kept with the raw data. Any noncompliant data that can not be corrected will be referred to the group leader for further action.

Additional Analyses:

The following methods are also performed using USEPA SW-846 Method 8240: 1508, 1131, 1265, 1266 1384, 3981, 4592, 5121, 5122, 5222, 5243, and 5244. The target compound lists for each of these analyses are listed in Table V through Table XVI, respectively. Any additional compounds should be added to the theoretical standards concentration sheet when performing these analyses. Standards containing additional analytes should be prepared as described in the reagents section of this document. Both secondary stock solutions and matrix spike solutions should be prepared for use in analyzing for additional compounds.

15081265.W51
MS Methods #4
032993

Prepared by: Susan M. Ayh Date: 3/30/93

Approved by: Robin U. Eaton Date: 4/15/93

Approved by: Susan B. Shorter Date: 4/23/93

CONFIDENTIAL

Table I

Practical Quantitation Limits (PQL) for Volatile Organics[a]

Volatiles	Practical Quantitation Limits Water/Wastewater (ug/l)
Acrolein	100
Acrylonitrile	100
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
Carbon tetrachloride	5
Chlorobenzene	5
Chlorodibromomethane	5
Chloroethane	10
2-Chloroethyl vinyl ether	10
Chloroform	5
Chloromethane	10
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1 Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	10
Xylene (total)	5

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependent PQLs.

CONFIDENTIAL

Table II

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

CONFIDENTIAL

Table III

Surrogate Spike Recovery Limits for Water and Wastewater Samples

Surrogate Compound	Water/Wastewater
4-Bromofluorobenzene	86-115
1,2-Dichloroethane-d4	76-114
Toluene-d8	88-110

CONFIDENTIAL

Table IV

Calibration and QC Acceptance Criteria[a]

Parameter	Range for Q (ug/l)	Range p, p_2 (%)
Acrolein	Not Defined	Not Defined
Acrylonitrile	Not Defined	Not Defined
Benzene	12.8-27.2	37-151
Bromodichloromethane	13.1-26.9	35-155
Bromoform	14.2-25.8	45-169
Bromomethane	2.8-37.2	D-242
Carbon tetrachloride	14.6-25.4	70-140
Chlorobenzene	13.2-26.8	37-160
Chloroethane	7.6-32.4	14-230
2-Chloroethylvinyl ether	D-44.8	D-305
Chloroform	13.5-26.5	51-138
Chloromethane	D-40.8	D-273
Dibromochloromethane	13.5-26.5	53-149
1,1-Dichloroethane	14.5-25.5	59-155
1,2-Dichloroethane	13.6-26.4	49-155
1,1-Dichloroethene	10.1-29.9	D-234
trans-1,2-Dichloroethene	13.9-26.1	54-156
1,2-Dichloropropane	6.8-33.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	D-227
trans-1,3-Dichloropropene	10.0-30.0	17-183
Ethyl benzene	11.8-28.2	37-162
Methylene chloride	12.1-27.9	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	46-157
Tetrachloroethene	14.7-25-3	64-148
Toluene	14.9-25.1	47-150
1,1,1-Trichloroethane	15.0-25.0	52-162
1,1,2-Trichloroethane	14.2-25.8	52-150
Trichloroethene	13.3-26.7	71-157
Trichlorofluoromethane	9.6-30.4	17-181
Vinyl chloride	0.8-39.2	D-251
Xylenes (total)	Not Defined	Not Defined

Q = Concentration measured in QC check sample, in ug/l.

p, p_2 = Percent recovery measured (20 ug/l spike)

[a]Criteria from 40 CFR Part 136 for Method 624 and were calculated assuming a QC check sample concentration of 20 ug/l.

CONFIDENTIAL

Table V

Analysis 1131

Compound	POL
Dichlorodifluoromethane	5.
3-Chloro-1-propene	5.
2-Butanone	100.
1,2-Dibromoethane	5.
1,1,1,2-Tetrachloroethane	5.
4-Methyl-2-pentanone	50.
1,2,3-Trichloropropane	5.
1,3-Dichlorobenzene	10.
1,2-Dichlorobenzene	10.
1,4-Dichlorobenzene	10.

CONFIDENTIAL

Table VI

Analysis 1265

Compound	PQL
Chloromethane	10.
Bromomethane	10.
Vinyl Chloride	10.
Dichlorodifluoromethane	5.
Chloroethane	10.
Methyl Iodide	5.
Acrolein	100.
Acrylonitrile	100.
Acetonitrile	100.
Methylene chloride	5.
Acetone	100.
Trichlorofluoromethane	5.
Carbon Disulfide	100.
Propionitrile	100.
1,1-Dichloroethene	5.
Allyl Chloride	5.
1,1-Dichloroethane	5.
trans-1,2-Dichloroethene	5.
Chloroform	5.
1,2-Dichloroethane	5.
Methacrylonitrole	100.
2-Butanone	100.
Dibromoethane	5.
1,1,1-Trichloroethane	5.
1,4-Dioxane	

CONFIDENTIAL

Table VII

Analysis 1266

Compound	PQL
Carbon Tetrachloride	5.
Isobutyl Alcohol	250.
Vinyl Acetate	50.
Bromodichloromethane	5.
2-Chloro-1,3-butadiene	5.
1,2-Dichloropropane	5.
trans-1,3-Dichloropropene	5.
Trichloroethene	5.
Dibromochloromethane	5.
1,1,2-Trichloroethane	5.
1,2-Dibromoethane	5.
Benzene	5.
cis-1,3-Dichloropropene	5.
Methyl Methacrylate	5.
1,1,1,2-Tetrachloroethane	5.
Bromoform	5.
trans-1,4-Dichloro-2-butene	100.
1,2,3-Trichloropropane	5.
2-Hexanone	50.
4-Methyl-2-pentanone	50.
Tetrachloroethane	5.
1,1,2,2-Tetrachloroethane	5.
Toluene	5.
Ethyl Methacrylate	5.
Chlorobenzene	5.
Pentachloroethane	10.
Ethylbenzene	5.
1,2-Dibromo-3-chloropropane	100.
Styrene	5.
Xylene (total)	5.

CONFIDENTIAL

Table VIII

Analysis 1358

Compound	PQL
Benzene	5.
1,2-Dibromoethane	5.
1,1-Dichloroethane	5.
1,1-Dichloroethene	5.
1,2-Dichloroethane	5.
trans-1,2-Dichloroethene	5.
Ethylbenzene	5.
Methylene Chloride	5.
Tetrachloroethene	5.
Toluene	5.
1,1,1-Trichloroethane	5.
Trichloroethene	5.
Vinyl Chloride	10.
Xylene (total)	5.

CONFIDENTIAL

Table IX

Analysis 1384

Compound	PQL
Benzene	5.
1,2-Dibromoethane	5.
1,1-Dichloroethane	5.
1,1-Dichloroethene	5.
1,2-Dichloroethane	5.
trans-1,2-Dichloroethene	5.
Ethylbenzene	5.
Methylene Chloride	5.
Tetrachloroethene	5.
Toluene	5.
1,1,1-Trichloroethane	5.
Trichloroethene	5.
Trichlorofluoromethane	5.
Vinyl Chloride	10.
Xylene (total)	5.

CONFIDENTIAL

Table X

Analysis 3981

Compound	POL
Benzene	5.
Toluene	5.
Ethylbenzene	5.
Xylene (total)	5.

CONFIDENTIAL

Table XI

Analysis 4592

Compound	POL
Chloromethane	10.
Bromomethane	10.
Vinyl Chloride	10.
Chloroethane	10.
Methylene Chloride	5.
Acetone	100.
Carbon Disulfide	100.
1,1-Dichloroethene	5.
1,1-Dichloroethane	5.
trans-1,2-Dichloroethene	5.
Chloroform	5.
1,2-Dichloroethane	5.
2-Butanone	100.
1,1,1-Trichloroethane	5.
Carbon Tetrachloride	5.
Vinyl Acetate	50.
Bromodichloromethane	5.
1,2-Dichloropropane	5.
cis-1,3-Dichloropropene	5.
Trichloroethene	5.
Dibromochloromethane	5.
1,1,2-Trichloromethane	5.
Benzene	5.
trans-1,3-Dichloropropene	5.
Bromoform	5.
4-Methyl-2-pentanone	50.
2-Hexanone	50.
Tetrachloroethene	5.
1,1,2,2-Tetrachloroethane	5.
Toluene	5.
Chlorobenzene	5.
Ethylbenzene	5.
Styrene	5.
Xylene (total)	5.

CONFIDENTIAL

Table XII

Analysis 5121

Compound	PQL
Chloromethane	10.
Vinyl Chloride	10.
Bromomethane	10.
Chloroethane	10.
Trichlorofluoromethane	5.
Ethyl Ether	5.
Acrolein	100.
1,1-Dichloroethene	5.
Acetone	100.
Carbon Disulfide	100.
Isopropyl Alcohol	100.
Acetonitrile	100.
Methylene Chloride	5.
t-Butyl Alcohol	100.
Acrylonitrile	100.
Hexane	5.
1,1-Dichloroethane	5.
1,2-Dichloroethene (total)	5.
2-Butanone	100.
Tetrahydrofuran	5.
Chloroform	5.
1,1,1-Trichloroethane	5.
Carbon Tetrachloride	5.
Benzene	5.
1,2-Dichloroethane	5.
Heptane	5.
Isopropyl Acetate	5.
n-Butanol	250.
Trichloroethene	5.
1,2-Dichloropropane	5.
Bromodichloromethane	5.

CONFIDENTIAL

Table XIII

Analysis 5122

Compound	POL
Monochloroacetone	250.
2-Chloroethyl Vinyl Ether	10.
cis-1,3-Dichloropropene	5.
Toluene	5.
trans-1,3-Dichloropropene	5.
1,1,2-Trichloroethane	5.
Tetrachloroethane	5.
Dibromochloromethane	5.
Chlorobenzene	5.
Ethylbenzene	5.
Xylene (total)	5.
Bromoform	5.
1,1,2,2-Tetrachloroethane	5.

CONFIDENTIAL

Table XIV

Analysis 5222

Compound	PQL
Acetone	100.
n-Pentane	10.
Ethyl Ether	5.
1,3-Diethylbenzene	5.
1,4-Diethylbenzene	5.
1,2-Diethylbenzene	5.

CONFIDENTIAL

Table XV

Analysis 5243

Compound	PQL
2-Butanone	100.
Allyl Chloride	5.
2-Chloro-1,3-butadiene	5.
Bromomethane	10.
Chloromethane	10.
Dibromomethane	5.
Methyl Iodide	5.
Trichlorofluoromethane	5.
Vinyl Chloride	10.
Dichlorodifluoromethane	5.
Chloroethane	10.
Methylene Chloride	5.
Acetone	100.
Carbon Disulfide	100.
1,1-Dichloroethene	5.
1,1-Dichloroethane	5.
1,2-Dichloroethene (total)	5.
Chloroform	5.
1,2-Dichloroethane	5.
1,1,1-Trichloroethane	5.
Carbon Tetrachloride	5.
Bromodichloromethane	5.
1,2-Dichloropropane	5.
Trichloroethene	5.
Benzene	5.
2-Chloroethyl Vinyl Ether	10.
Ethyl Ether	5.
Freon 113	5.
Ethyl Acetate	5.
2-Nitropropane	10.

CONFIDENTIAL

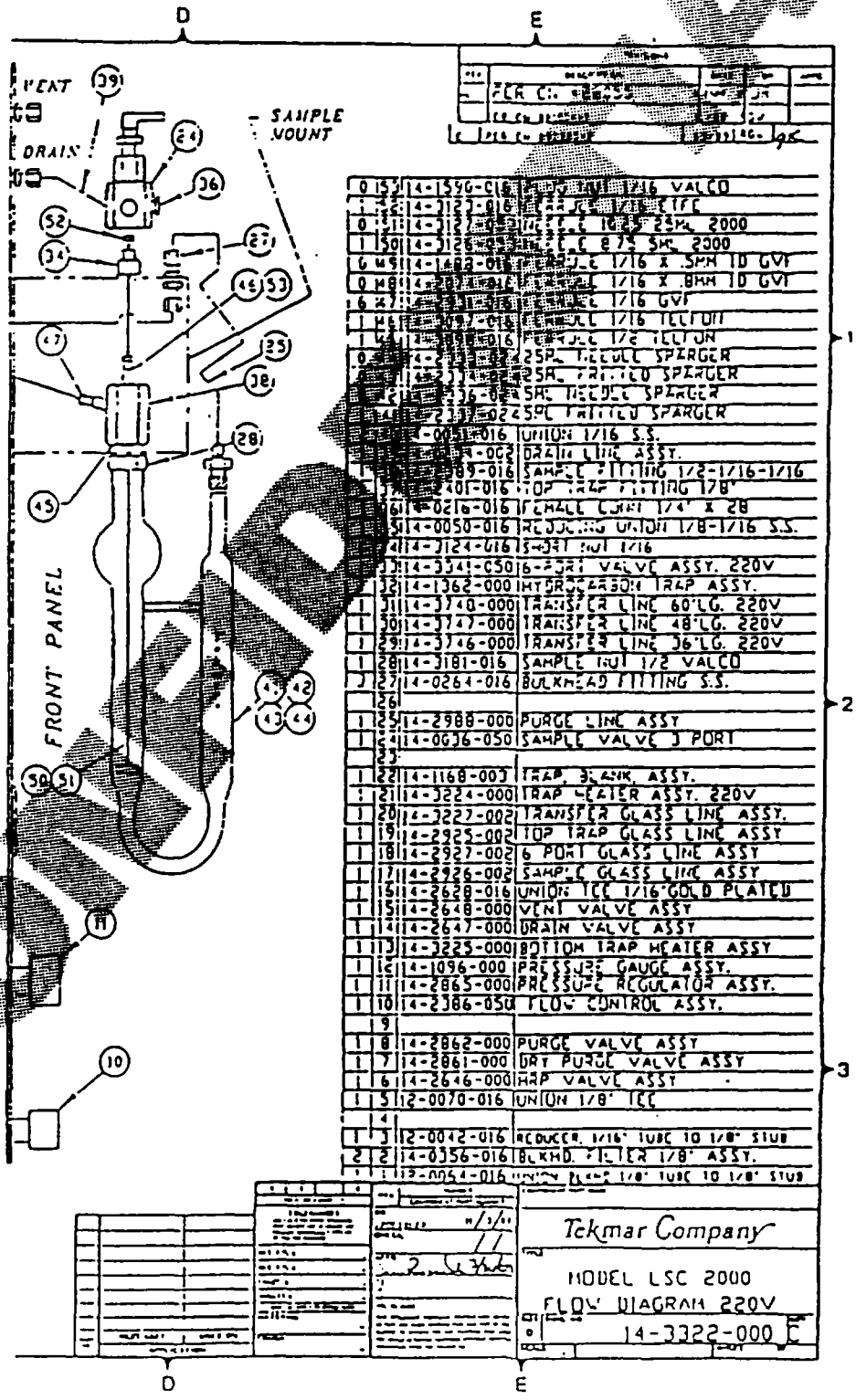
Table XVI

Analysis 5244

Compound	PQL
Cyclohexanone	100.
trans-1,4-Dichloro-2-butene	100.
1,2-Dichlorobenzene	10.
trans-1,3-Dichloropropene	5.
Dibromochloromethane	5.
1,1,2-Trichloroethane	5.
1,2-Dibromomethane	5.
cis-1,3-Dichloropropene	5.
1,1,1,2-Tetrachloroethane	5.
Bromoform	5.
1,2,3-Trichloropropane	5.
4-Methyl-2-pentanone	50.
Tetrachloroethene	5.
1,1,2,2-Tetrachloroethane	5.
Toluene	5.
Chlorobenzene	5.
Ethylbenzene	5.
1,2-Dibromo-3-chloropropane	100.
Xylene (total)	5.

Figure 1

Tekmar®



CONFIDENTIAL

Figure 2

THEORETICAL STANDARD CONCENTRATIONS Rev. 03/20/92
INITIAL CALIBRATION
PACKED COLUMN
EPA SW846 METHOD 8240 - APPENDIX IX/FORM 8

Note: A charcoal trap must be used to calibrate for these compounds.

DATE:

INSTRUMENT:

stock mix name	V1____ V2____ V3____	V4____ V5____	V6____ V7____	V8____	DCDEM MX RESTEK MIX(200)	FLASK ml
200ppb std	5 ul	12.5 ul	5 ul	5 ul	25 ul	25
150ppb std	7.5 ul	18.75ul	7.5 ul	7.5 ul	37.5 ul	50
100ppb std	5 ul	12.5 ul	5 ul	5 ul	25 ul	50
50ppb std	5 ul	12.5 ul	5 ul	5 ul	25 ul	100
20ppb std	4 ul	10 ul	4 ul	4 ul	20 ul	200

concentration compound name	stock ppm	200 ppb	150 ppb	100 ppb	50 ppb	20 ppb
Bromodichloromethane	1000	200	150	100	50	20
Bromoform	1000	200	150	100	50	20
Carbon Tetrachloride	1000	200	150	100	50	20
Chlorobenzene	1000	200	150	100	50	20
Chloroform	1000	200	150	100	50	20
Dibromochloromethane	1000	200	150	100	50	20
1,1-Dichloroethane	1000	200	150	100	50	20
1,2-Dichloroethane	1000	200	150	100	50	20
1,1-Dichloroethene	1000	200	150	100	50	20
trans-1,2-Dichloroethene	1000	200	150	100	50	20
1,2-Dichloropropane	1000	200	150	100	50	20
Styrene	1000	200	150	100	50	20
1,1,2-Trichloroethane	1000	200	150	100	50	20
cis-1,3-Dichloropropene	1000	200	150	100	50	20
trans-1,3-Dichloropropene	333	66.6	50	33.3	16.6	6.7
Ethylbenzene	1000	200	150	100	50	20
Methylene Chloride	1000	200	150	100	50	20
Tetrachloroethene	1000	200	150	100	50	20
1,1,2,2-Tetrachlorethane	1000	200	150	100	50	20
1,1,1-Trichloroethane	1000	200	150	100	50	20
Trichloroethene	1000	200	150	100	50	20
Trichlorofluoromethane	1000	200	150	100	50	20
Toluene	1000	200	150	100	50	20
o-Xylene	1000	200	150	100	50	20
Benzene	1000	200	150	100	50	20
Acrolein	5000	1000	750	500	250	100
Acrylonitrile	5000	1000	750	500	250	100
2-Chloroethyl Vinyl Ether	1000	200	375	100	50	20
Vinyl Acetate	1000	500	375	250	125	50
Carbon Disulfide	1000	500	375	250	125	50
Acetone	1000	500	375	250	125	50
2-Hexanone	1000	500	375	250	125	50
2-Butanone	1000	500	375	250	125	50
4-Methyl-2-pentanone	1000	500	375	250	125	50

UNOFFICIAL

Figure 2 - Continued

compound name	stock ppm	200 ppb	150 ppb	100 ppb	50 ppb	20 ppb
1,2-Dibromo-3-chloropropane	2500	500	375	250	125	50
1,2-Dichlorobenzene	1000	200	150	100	50	20
1,3-Dichlorobenzene	1000	200	150	100	50	20
1,4-Dichlorobenzene	1000	200	150	100	50	20
1,4-Dioxane	5000	1000	750	500	250	100
1,2-Dibromomethane	1000	200	150	100	50	20
1,2,3-Trichloropropane	1000	200	150	100	50	20
Methyl t-Butyl Ether	1000	200	150	100	50	20
t-Butyl Alcohol	10000	2000	1500	1000	500	200
Di-isopropyl Ether	1000	200	150	100	50	20
Allyl Chloride	1000	200	150	100	50	20
Cumene	1000	200	150	100	50	20
Dibromomethane	1000	200	150	100	50	20
trans-1,4-Dichloro-2-butene	2500	500	375	250	125	50
Ethyl Methacrylate	1000	200	150	100	50	20
Isobutyl Alcohol	12500	2500	1875	1250	625	250
Methacrylonitrile	2500	500	375	250	125	50
Methyl Iodide	1000	200	150	100	50	20
Methyl Methacrylate	1000	200	150	100	50	20
Pentachloroethane	1000	200	150	100	50	20
Propionitrile	5000	1000	750	500	250	100
1,1,1,2-Tetrachloroethane	1000	200	150	100	50	20
Acetonitrile	5000	1000	750	500	250	100
2-Chloro-1,3-butadiene	1000	200	150	100	50	20
Dichlorodifluoromethane	200	200	150	100	50	20
Chloromethane	200	200	150	100	50	20
Vinyl Chloride	200	200	150	100	50	20
Bromomethane	200	200	150	100	50	20
Chloroethane	200	200	150	100	50	20

page 2 of 2

ppb of analytical standard = stock ppm x ul stock/flask ml

Analyst _____.

CONFIDENTIAL

Analysis #200
Initiated Date: 2/27/85
Effective Date: AUG 14 1992

CONFIDENTIAL

pH (WW)
(electrometric)

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 150.1.

2. Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989, 4500-H+B, pp. 4-95 - 4-102.

3. Lange's Handbook of Chemistry, 13th Edition, 1985, pp. 5-97 - 5.99.

Scope:

This procedure is applicable to aqueous samples.

The sensitivity limit for this technique is 0.01 pH units. Interferences may be caused by oily or particulate matter adhering to the electrodes and reducing the response. Gentle wiping or rinsing with deionized water will usually correct this problem. Temperature effects can be compensated for by calibrating the pH meter at the temperature of the sample or using a pH meter equipped with temperature compensators. Temperature effects caused by shifts in ionic equilibria of the sample cannot be controlled.

Basic Principle:

The electrometric pH is a measure of the activity of hydrogen ions in a sample using a glass electrode and a reference electrode.

Apparatus and Reagents:

1. pH meter

2. Solution C Stock Solution - Dissolve 33.870 g potassium phosphate monobasic (KH_2PO_4), dried at 105°C for one hour, plus 35.330 g sodium phosphate dibasic (Na_2HPO_4) (do not dry), in deionized water and dilute to 1 liter (0.25 M KH_2PO_4; 0.25 M Na_2HPO_4). Stable four months at 4°C ± 2°C.

3. Solution C Working Standard (pH = 6.87) - Pipet 100 ml of stock solution into a 1 liter volumetric flask and dilute to volume with deionized water. Stable four weeks at 4°C ± 2°C.

4. Solution H Working Standard (pH = 10.01) - Dissolve 2.10 g sodium bicarbonate ($NaHCO_3$), plus 2.65 g sodium carbonate (Na_2CO_3) in deionized water and dilute to 1 liter (.025 M $NaHCO_3$; .025 M Na_2CO_3). Store in a plastic container. Stable four weeks at 4°C ± 2°C.

5. Combination electrodes, or equivalent

6. Stir bar and stir plate

7. Appropriate pH electrode filling solution for electrode

Safety Precautions:

There are no special safety precautions for this method. Use routine laboratory safety procedures.

CONFIDENTIAL

Procedure:

1. Water pH analysis should be performed at time of sample collection as specified in 40 CFR Part 136. Samples will be analyzed as soon as possible after the sample is received in the laboratory.

2. Calibrate the pH meter as described in SOP MC-WQ-007.

3. Place the sample in a clean beaker, enough to immerse the sensing elements of the electrodes (approximately 30 ml).

4. Rinse and shake off any water on the electrode. Immerse the electrode in the sample and stir at a constant rate, being careful not to let the stir bar bump the electrode.

5. Note the pH after the meter equilibrates. If the pH is less than 9.0, record the pH and repeat the procedure with the next sample. If the pH is greater than 9.0, the sample will need to be reread while the meter is sloped with the pH 7.0 and pH 10.0 buffers. Solution H should be run before any samples when the meter is sloped with the pH 7.0 and pH 10.0 buffers.

   NOTE: If pH fluctuates, add 5 drops of filling solution to the sample.

Statistical Information:

For statistical information, refer to EPA Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-070 p. 150.1-3.

CONFIDENTIAL

Quality Assurance:

A standard should be run at the beginning of each batch, after every ten samples, before and after resloping the meter, and at the end of the day's run. One duplicate (±20% reproducibility) should be run per bath (20 samples). The pH standards must be within ±1.90% of their true values.

NO200.W51
WQ Methods #2
080692

CONFIDENTIAL

Prepared by: Eric J Frederick Date: 8/6/92

Approved by: Beth Ebg Date: 8/6/92

Approved by: Susan B. Shorter Date: 8-13-92

Analysis #280
Initiated Date: 10/23/85
Effective Date: MAY 1 1 1992

CONFIDENTIAL

Specific Conductance

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 120.1.

2. Standard Methods for the Examination of Water and Wastewater, 17th edition, 1989, Method 2510B, p. 2-59 - 2-61.

Scope:

The limit of quantitation for this method is 4 umhos/cm at 25°C.

Basic Principles:

Conductivity is a numerical expression of the ability of aqueous solution to carry an electric current. This ability depends on the presence of ions, their total concentration, mobility, valence, and relative concentrations. The specific conductance of a sample is measured by the use of a conductivity meter.

Sample Handling:

If analysis is not completed within 24 hours of sample collection, the sample should be filtered through a .45 micron filter and stored at 4°C ± 2°C. The holding time is 28 days.

CONFIDENTIAL

Apparatus and Reagents:

1. Conductivity meter (Hach Model 44600), or equivalent.

2. Solution D Stock Standard: .01 M KCl solution (1413 umhos/cm). Dissolve .7456 g predried KCl (two hours @ 105°C) in deionized water in a one liter volumetric flask. Dilute to volume. (Stable three months at 4°C ± 2°C.)

3. .001 M KCl Working Standard (147 umhos/cm): Dilute 10 ml of stock in a 100 ml volumetric with deionized water. (Prepare fresh daily.)

Safety Precautions:

Use caution when handling any chemicals, solvents, or reagents. Avoid inhalation and skin contact.

Procedure:

Note: All standards and samples must be at room temperature prior to analysis.

1. Rinse cell with deionized water until reading is .002 or lower on the 2 mS/cm range scale.

2. Rinse cell with sample or standard being analyzed.

3. Dip conductivity cell in standard (147 umhos/cm). Dip and stir until conductivity stabilizes. Record range and reading. Rinse cell with deionized water until reading is .002 or lower on the 2 mS/cm range.

4. Repeat steps 2 and 3 with blank (deionized water) and samples, recording the range and value for each sample.

5. If the reading is greater than can be read on the 2 mS/cm scale, use the 20 mS/cm scale. If it is still greater than 12.90 mS/cm, then dilute and reread.

6. If the mS/cm value is greater than 2.70, then calculate the value using the high level curve in the computer statistics program.

Statistical Information:

For statistical information, refer to EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 120.1.2, and Standard Methods for the Examination of Water and Wastewater, 17th edition, p. 2-60.

Calculations:

Enter reading as meter reading value in raw data entry. Calculate cell constant quarterly, according to MC-WQ-004 and replace constant for analysis #280 and #1215.

Quality Assurance:

Run .001 M working standard and blank each day specific conductance is run. Run duplicate per batch. The standard must be within ± 20% true value. The duplicate reproducibility must be ± 20%.

CONFIDENTIAL

Prepared by: Eric Frederick Date: 5/1/92

Approved by: Beth Eby Date: 5/1/92

Approved by: [illegible] Date: 5/8/92

Lancaster Laboratories
Where quality is a science.

Procedural Amendment #1

Number: Analysis #280

Title: Specific Conductance

Effective Date (listed on procedure): 05/11/92

Section(s) affected by change: References

Reason for addition(s) or change(s): Updated reference

Change will be effective from (date): 06/03/93

Samples or project affected: All

List change(s) or addition(s) (specify which section):

References:

Change indicated reference to read as follows:

2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992, Method 2510B, pp. 2-45 - 2-46

NO280.W51
WQ Methods #1
060793

CONFIDENTIAL

Prepared by: Erik Friedrich Date: 6/25/93

Approved by: Beth Eby Date: 7/5/93

Approved by: Susan B. Shorter Date: 7/6/93

Analysis #212, 389
Initiated Date: 11/12/84
Effective Date: JUL 01 1993

CONFIDENTIAL

Total Dissolved Solids

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, p. 160.1.

2. Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989, Method 2540C, p. 2-74.

Scope:

The sensitivity limit for this technique varies with sample volume:

Sample Volume (ml)	Sensitivity (mg/l)
200	25
100	50
50	100
25	200

Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and/or sulfate may require prolonged drying. Waters with excessive residue may form a water-trapping crust and, therefore, should be limited to no more than 400 mg residue. Analysis temperature is increased to 180° ± 2°C in order to convert bicarbonate to carbonate in the sample. Holding time is seven days.

Basic Principles:

A well-mixed sample is filtered through a glass fiber filter, the filtrate is then placed in a tared beaker or evaporating dish, evaporated to dryness on a steam bath or equivalent, and dried to a constant weight at 180° ± 2°C. The increase in dish weight represents the total dissolved solids.

Apparatus and Reagents:

1. Glass fiber filters

2. Filtration apparatus: Suction flask, 500 ml capacity (or equivalent)
   Filter Holder
   Buchner Funnel

3. 150 ml, 250 ml beaker, or evaporating dishes

4. TDS/TS Stock Standard (4000 mg/l). Dissolve 4.0 g KCl (dried approximately one hour at 105° ± 2°C, desiccate approximately one hour) in deionized water. Dilute to 1000 ml final volume. Stable six months at 4° ± 2°C.

5. TDS/TS Working Standard (200 mg/l). Dilute 50 ml stock standard up to 1000 ml with deionized water. Stable one month at 4° ± 2°C.

6. Hot water bath, or equivalent

CONFIDENTIAL

Procedure:

1. Place glass fiber filters on the bottom of a Buchner funnel. Apply vacuum and wash with three 20 ml portions of deionized water. Continue suction to remove all traces of water. Discard washings.

2. Dry clean evaporating dish, 150 ml, or 250 ml beaker in an oven for one hour at 180° ± 2°C. If fixed or volatile dissolved solids are to be performed on sample, use an evaporating dish dried in a muffle furnace for one hour at 550° ± 50°C.

3. Cool in a desiccator for two hours.

4. Check to see if balance had been calibrated that day. Record this and the balance number in tare weight book. Weigh beakers and/or evaporating dishes.

5. Shake sample well. (Use a blender, if necessary, to form a more homogeneous sample.)

6. Using a graduated cylinder (or equivalent), measure out an appropriate aliquot, sufficient to contain between 2.5 mg and 400 mg total dissolved solids. Rapidly transfer the aliquot to the Buchner funnel.

7. Filter the sample under vacuum and wash the filter with three 10 ml portions of deionized water (rinsing inside of graduated cylinder and funnel). Allow complete drainage between washings. Continue suction until filtration is complete.

8. Transfer the filtrate to either beaker or evaporating dish prepared as described in Step 2. Rinse flask with two small portions of deionized water. Transfer rinses to beaker or flask.

CONFIDENTIAL

9. Evaporate the sample to dryness on a steam bath or equivalent.

10. When evaporation is complete, place in an oven at 180° ± 2°C.

11. Samples should remain at this temperature for at least one hour.

12. Cool in a desiccator two hours, check to see if balances have been calibrated that day, if so then weigh samples. Repeat drying cycle until weight loss is less than 0.5 mg.

13. If an aliquot of insufficient amount was selected, repeat process using more sample; lesser volume if residue exceeded .400 g (400 mg).

Safety Precautions:

There are no special safety precautions for this procedure. Follow routine laboratory safety steps.

Calculations:

$$mg\ total\ dissolved\ solids/l = \frac{(A - B) \times 1000 \times 1000}{Sample\ Volume,\ ml}$$

where: A = weight of dried residue and beaker or dish, g
B = weight of beaker or dish, g

Occasionally weight gain is exhibited during the analysis due to oxidation of the sample. When this occurs the lowest weight, prior to oxidation, is used in the calculation.

CONFIDENTIAL

Statistical Information:

For statistical information refer to Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989, Method 2540C, p 2-74.

Quality Assurance:

A blank (<LOQ or sample values ≥10 x blank value), standard (± 20%), duplicate (± 20% RPD), and spike (± 25%) should be analyzed with each batch of 20 samples.

Comments:

For Analysis #389, use Comment 292 to report quadruplicate determinations.

NO212.W51
WQ METHODS #1
062893

CONFIDENTIAL

Prepared by: Sandra J. Miller Date: 6/30/93

Approved by: Beth Elg Date: 6/30/93

Approved by: Susan B. Shorter Date: 6/30/93

Analysis #206
Initiated Date: 11/09/84
Effective Date: JUL 08 1993

CONFIDENTIAL

Total Suspended Solids

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, 160.2.

2. Standard Methods for the Examination of Water and Wastewater, 18th edition, 1992, Method 2540D, p. 2-56.

Scope:

This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.

The limit of quantitation (LOQ) for this technique varies with the sample volume. The laboratory mainframe computer may round the LOQs during the calculation:

Sample Volume (ml)	LOQ (mg/l)
250	7
100	18
50	35
25	70
2	875

The practical range of determination is 4 mg/l to 20,000 mg/l. Samples high in dissolved solids may yield positive interferences.

Basic Principles:

A well-mixed sample is filtered through a glass fiber filter and the residue on the filter is dried to a constant weight at 103° to 105°C. The filtrate may be used for Total Dissolved Solids. The holding time is seven days.

Apparatus and Reagents:

1. Glass fiber filters

2. Filtration apparatus:

   a. 500 ml suction flask, or equivalent

   b. filter holder

   c. Gooch crucible

3. TSS Working Standard (150 mg/l). Dissolve .1500 ± .005 g Napthalimide (dried approximately one hour at 105° ± 2°C desiccated approximately one hour) into deionized water. Dilute to 1000 ml. Stable one month at 4° ± 2°C. Napthalimide can be weighed out and stored prior to use.

Safety Precautions:

There are no special safety precautions for this procedure. Follow routine laboratory safety steps.

Procedures:

1. Downloading a batch of samples to be performed using the LLENS system (consult SOP-WQ-014).

CONFIDENTIAL

a. Downloading a sample list for analysis #206.

(1) When downloading samples for analysis 206, all incomplete samples for analyses 206, 207, and 208 will appear. Choose the appropriate samples using the "Insert" key, and press PF10 to save.

(2) A table will appear labeled "Sample Table Editing."

(3) There is no need to enter a batch standard or blank. The LLENS system adds these automatically.

(4) If the batch chosen contains data package samples with client-submitted QC, use client QC for batch duplicate. If the batch chosen does not contain client submitted QC, choose a sample for a duplicate. Duplicates are selected by pressing PF5 when the cursor is on the background sample.

(5) Press PF10 to save.

b. Hand entering a sample table for analysis 206.

(1) Using the P.C. WATERM, hand enter a sample table for incomplete 206 samples. Select the duplicate by pressing PF5.

2. Printing a worksheet.

a. Move to the P.C. WTRSLD. From the Main Menu execute "Examine Data."

CONFIDENTIAL

b. Select the batch which has the incomplete samples entered (using the "Insert" key and pressing "Enter").

c. Press PF2 to print.

d. To choose data to print, select "Tare Weights" and "Sample Volumes"; press "Insert" key.

e. Execute PF10 to print worksheet.

f. Press PF10 to return to Main Menu.

3. Taring crucibles.

a. Place a glass fiber filter on the bottom of a gooch crucible (wrinkled side up). Apply vacuum and wash with three 20 ml portions of deionized water. Continue suction to remove all traces of water.

b. Dry crucible and filter in an oven for one hour at 103° to 105°C. If fixed or volatile suspended solids are to be performed, ignite the crucible in a muffle furnace at 550° ± 50°C for 20 minutes.

c. Place crucible in desiccator, wait one hour before weighing. Check balance calibration--record the balance number in the tare weight book with crucibles and tare weight. Crucibles may be stored until needed.

4. Sample analysis.

a. Shake sample. Remove nonrepresentative particles. Use a blender, if necessary.

CONFIDENTIAL

b. Using a calibrated pipette or graduated cylinder, measure sample volume. Record sample volume and crucible ID on worksheet.

c. Apply vacuum and filter a small amount of deionized water. This is to seat the filter securely in the crucible. Filter the aliquot, then using three 10 ml portions of deionized water, wash residue from graduate into crucible. This step is important to ensure that dissolved solids are rinsed through, and not dried in the filter. Continue vacuum until all trace of water is gone. A final rinse may be done to remove any residue from the sides of the crucible. Vacuum until all trace of water is gone.

d. Record oven temperature on worksheet. Put samples in oven. Dry at 103° to 105°C overnight (or as little as an hour, if necessary).

Samples may remain in oven over a weekend based on a validation study performed on November 18, 1992.

5. Hand entering data (sample volumes and tare weights).

a. From Main Menu, execute "Hand Enter Data."

b. Select the batch which has the incomplete samples.

c. Select the "Tare Weights"; press "Enter."

d. Enter crucible tare weights from databook. Press PF10 to save.

e. Select the "Sample Volumes;" press "Enter."

CONFIDENTIAL

f. Hand enter sample volumes and crucible IDs from worksheet; press PF10 to save.

g. Record oven temperature on worksheet prior to removing samples. Desiccate for one hour and weigh. Check balance calibration prior to weighing samples and document on worksheet. (Check desiccant to be sure indicator crystals are still fresh.)

6. Data collection.

a. Record oven temperature on worksheet prior to removing samples. Check balance calibration prior to weighing samples and document on worksheet.

b. Desiccate for one hour. If unable to weigh promptly after one hour desiccation, reheat samples for at least one hour in the oven. Redesiccate for one hour. (If samples are removed prior to overnight, repeat drying cycle until a constant weight is obtained <0.5 mg.)

c. From Main Menu, execute "Balance Data Collection."

d. Execute batch with incomplete samples.

e. Execute "Final Dry Weights."

f. Weigh samples, press space bar to transfer weight from balance to LLENS system. (An asterisk will appear when the weight stabilizes.) Press PF10 to save.

CONFIDENTIAL

7. Calculating and transmitting the finished batch.

   a. Move to P.C. WATERM. From LLENS Main Menu, execute "Calculate Results" for analysis 206.

   b. Select incomplete batch to calculate. Execute PF10 to transmit data to the Wang system.

   c. Collect all printed reports.

   d. Use the Wang to enter duplicate sample as a trial 2 for the batch.

Calculations:

$$\text{mg total suspended solids/l} = \frac{(A - B) \times 1000 \times 1000}{\text{Sample Volume (ml)}}$$

where A = weight of filter, crucible, and residue, g
B = weight of filter and crucible, g

Statistical Information:

For statistical information, refer to Standard Methods for the Examination of Water and Wastewater, 18th edition, 1992, p. 2-56.

CONFIDENTIAL

Quality Assurance:

One duplicate (± 20% RPD), blank (<LOQ, or sample values ≥10 x blank value), and standard (± 20%) should be run at the beginning of each batch (20 samples).

CONFIDENTIAL

NO206.W51
WQ METHODS #1
062893

Prepared by: Sandra J. Miller Date: 6/30/93

Approved by: Beth Eby Date: 6/30/93

-d by: Susan B. Shorter Date: 7/7/93

Analysis #234
Initiated Date: 10/23/84
Effective Date: AUG 14 1992

CONFIDENTIAL

Chemical Oxygen Demand (COD)
(Dichromate Reflux Method)
(Titrimetric, Mid-Level)

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 410.1 (Titrimetric, Mid-Level).

2. Standard Methods for the Examination of Water and Wastewater, 17th edition, 1990, pp. 5-12.

Scope:

The limit of quantitation is 50 mg O_2/l for waters or 2000 mg O_2/kg COD for solid samples. The mid-level method is used for samples with concentrations greater than 250 mg/l. The low-level method is used for New Jersey and New York samples and samples with concentrations of less than 250 mg/l.

Interferences:

Interferences can be caused by trace organic material on the glassware or in the atmosphere. A blank performed with each batch of samples corrects for any error introduced. Difficulties caused by the presence of halides can be reduced by adding mercuric sulfate ($HgSO_4$) before refluxing. If the chloride concentrations of the sample exceeds 1,000 mg/l, a modified method for saline waters is required.

CONFIDENTIAL

Sample Handling and Preservation:

Collect the samples in glass bottles (plastic containers are permissible if free from organic contaminants). Water samples should be preserved with the addition of 2 ml concentrated sulfuric acid per liter to a pH ≤ 2, and refrigeration at 4°C ± 2°C. Solid wastes do not require preservation. The holding time for this analysis is 28 days for waters. Literature does not cite a holding time for solid samples.

Basic Principles:

Chemical oxygen demand is a measure of the total amount of oxygen required for oxidation of a waste to carbon and water. It represents the amount of oxygen that would be required from a receiving body of water if oxygen alone, without microorganisms, could oxidize organic waste materials to the same by-products.

A sample is refluxed in acidic solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion, the remaining $K_2Cr_2O_7$ is titrated with ferrous ammonium sulfate (FAS). The amount of $K_2Cr_2O_7$ consumed is determined, and the COD in mg O_2/l is calculated.

Apparatus and Reagents:

1. Reflux apparatus consisting of 500 ml flasks and condensers with ground glass joints.

2. Hot plates.

3. Concentrated sulfuric acid (H_2SO_4).

4. Silver sulfate (Ag_2SO_4), powder, reagent grade.

5. Mercuric sulfate ($HgSO_4$), powder.

6. Standard Potassium Dichromate Solution (0.250 N): Dry potassium dichromate at 103°C ± 2°C for two hours. Dissolve 12.259 g ± 0.0005 in deionized water and dilute to 1 liter. Stable for six months at room temperature.

7. Sulfuric Acid Reagent: Add 23.5 g ± 0.05 silver sulfate to 4 kg concentrated sulfuric acid. Put stir bar in bottle and place on stir plate until silver sulfate is dissolved. Complete dissolution may take over an hour. Stable for six months at room temperature.

8. Ferroin Indicator Solution: Dissolve 1.485 g ± 0.0005 1,10-phenanthroline monohydrate and 0.695 g ± 0.0005 ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) in deionized water and dilute to 100 ml. Stable for six months at room temperature.

9. Standard Ferrous Ammonium Sulfate (0.250 N): Dissolve 98.0 g ± 0.05 ferrous ammonium sulfate ($Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$) in deionized water. Add 20.0 ml concentrated sulfuric acid and dilute to 1 liter. Stable for six months at room temperature.

10. COD Standard (5,000 mg/l): Dissolve 0.2125 g potassium hydrogen pthalate (KHP) dried at 105°C ± 2°C for two hours in deionized water and dilute to 50 ml. Preserve with H_2SO_4 to a pH <2 and store at room temperature. Stable for one month.

11. COD Standard (500 mg/l): Dissolve 0.2125 g potassium hydrogen pthalate (KHP) dried at 105°C ± 2°C for two

CONFIDENTIAL

hours in deionized water and dilute to 500 ml. Preserve with H_2SO_4 to a pH <2 and store at 4°C (± 2°C). Stable for one month.

12. COD Standard (50 mg/l): Dilute 20 of the 500 mg/l standard to 200 ml with deionized water. Preserve with H_2SO_4 to a pH <2 and store at 4°C (± 2°C). Stable for one month.

Safety Precautions:

Dispose of digest and reagents in the acid waste container for Department 29. These contain mercury and concentrated sulfuric acid and should be handled with extreme care.

Standardization (Normality):

To approximately 200 ml deionized water in a 500 ml Erlenmeyer flask, add 25 ml 0.250 N $K_2Cr_2O_7$ solution. Add 20 ml concentrated H_2SO_4 and place in hood with samples for duration of the reflux. Titrate with samples in procedure section.

Procedure:

1. Wash down condensers with dilute H_2SO_4 and deionized water. Turn on water to condensers.

2. Place three to five glass beads in the reflux flask, followed by three level scoops of $HgSO_4$. (about 1 g $HgSO_4$)

3. Add 50 ml sample or an aliquot diluted to 50 ml to the flask. For solid samples, any appropriate amount $\leq$2.0000 g should be weighed on the analytical balance. Quantitatively transfer this amount to the reflux flask.

CONFIDENTIAL

4. Add 5 ml concentrated sulfuric (bottle dispenser or equivalent) and swirl until the $HgSO_4$ has dissolved.

5. Add 25 ml 0.250 N $K_2Cr_2O_7$ to the flask (bottle dispenser or equivalent) and swirl.

6. Add 70 ml sulfuric acid reagent to the reflux flask (two pumps of bottle dispenser or equivalent) while swirling. Attach to condenser immediately to limit loss of volatile organics.

CAUTION! Make sure contents of flask are well mixed to prevent superheating.

7. Turn hot plate to HIGH and reflux for two hours.

8. Allow flasks to cool while continuing water flow for 20 minutes.

9. Wash down the condensers with about 25 ml deionized water.

10. Add approximately 100 ml deionized water to the flasks and allow them to cool to about room temperature (an ice bath is recommended).

11. Add eight to ten drops ferroin indicator to the solution and titrate the excess dichromate with approximately 0.250 N FAS. The color change will be sharp, changing from a blue-green to a reddish-brown. If less than 6 ml FAS is used, repeat analysis with less sample.

CONFIDENTIAL

Calculations:

$$\text{Normality FAS} = \frac{\text{ml } K_2Cr_2O_7 \times 0.25}{\text{ml FAS}}$$

$$\text{COD mg/l or mg/kg} = \frac{(A - B) \times N \times 8{,}000}{\text{ml or g sample}}$$

A = volume FAS used for the blank

B = volume FAS used for the sample

N = normality of the FAS solution

Statistical Information:

For statistical information refer to EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 410.1.

Quality Assurance:

Batch QC for the Mid-level analysis should include the normality, blank (50 ml deionized water), a 50 mg/l standard (acceptance = ± 50%), a 500 mg/l standard (acceptance = ± 20%), a duplicate (acceptance ≤ 20% Relative Percent Difference), and a matrix spike (acceptance = 75 to 125% recovery).

NO234
WQ Methods #1
081192

CONFIDENTIAL

Prepared by: Robert Heisey Date: 8/11/92

Approved by: [illegible] Date: 8/13/92

Approved by: [illegible] Date: 8/14/92

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

Name of Curve: Dangerous
Corr. Coeff.: .99728
Slope: .0048
Y-intercept: -.00212

Analysis #4001
Chemical Oxygen Demand

Batch #: 9 2 2 0 4 | 6 0 5 | 9 0 0 | 4 0 0 1

Init./Employee #: SMC/605 Date Analyzed: 7-22-92

	Sample Number	Dilution Factor	Absorbance @ 600 nm	Conc. from Curve	COD mg/l	LOQ	Comments
1	B	1x	0.00	0.0	<50.	50 mg/l	
2	50 mg/l	1x	.028	54.0	50. (IS)		Raw: 54.0 (108%)
3	500 mg/l	1x	.239	493.0	490. (IS)		Raw: 493.0 (98%)
4	5-27-92	10x	.251	518.0	5180.		(104%)
5	6-3-92	10x	.236	487.0	4870.		(97%)
6	6-10-92	10x	.248	512.0	5120.		(102%)
7	6-24-92	10x	.252	521.0	5210.		(104%)
8	6-29-92	10x	.244	504.0	5040.		(101%)
9	7-7-92	10x	.256	529.0	5290.		(106%)
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							

Verified by: RGH/355 Date: 7/23/92

1401 7/17/91

CONFIDENTIAL

Analysis #235
Initiated Date: 3/86
Effective Date: AUG 25 1992

Determination of Biochemical Oxygen Demand in Water and Wastewater

CONFIDENTIAL

References:

1. Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020 March, 1983 Method 405.1.

2. Standard Methods for the Examination of Water and Wastewater; Method #5210B, 17th Ed., 1989, pp. 5-4 to 5-10.

Scope:

Biochemical Oxygen Demand is an empirical test which measures the dissolved oxygen consumed by microbes while assimilating and oxidizing organic matter in water and wastewater samples. It is used for determining the relative oxygen requirements of municipal and industrial wastes and often used by sewer authorities to assess surcharges on clients.

The BOD test determines the relative oxygen requirements of waters. The test measures the amount of oxygen required by bacteria over a period of five days at 20°C ± 1°C to consume biodegradable material. It also measures the amount of oxygen used to oxidize inorganic material such as sulfides and ferrous iron.

The method consists of placing a sample in a full, airtight bottle and incubating the bottle under specific conditions for a specific time. BOD is calculated as the difference between the initial D.O. and the D.O. after incubation.

In this test, conditions that inhibit bacterial growth must be avoided. Some conditions that minimize optimum growth of bacteria are as follows:

1. pH outside neutral range of 6.5-7.5

2. Toxic chemicals - chlorine

3. High or low temperature exposure

4. Low dissolved oxygen level

5. Insufficient nutrients

6. Insufficient organic matter as food source

pH

Samples are neutralized to pH 6.5 to 7.5 with a solution of sulfuric acid or sodium hydroxide before dilutions are made. Bacterial growth tends to lower pH which will inhibit further growth. A buffer is added to the dilution water to ensure that the pH of the incubated sample over five days remains in a range that is suitable for growth.

Toxic Chemicals

Chlorine in samples can reduce or inhibit bacterial growth causing erroneously low results. Chlorine will be neutralized using a sodium sulfite solution. The sodium sulfite solution should be added in sufficient quantity (as determined by a starch-iodine titration) to neutralize the chlorine but not in excess.

There are also other sources of toxicants, including heavy metals such as copper.

CONFIDENTIAL

CONFIDENTIAL

Temperature

Optimum temperature for bacterial growth is 20°C ± 1°C.

If the temperature is lower than 20°C, the reaction will be slowed. The decrease in biological activity will cause the oxygen uptake rate to decrease. Erroneously low results will be caused by the slowed biological growth.

If the temperature exceeds the optimum temperature of 20°C, the reaction rate increases. This will cause the microorganisms to reach their peak at a lower age, causing erroneously high results. High temperatures may also reduce biological activity, causing low results, due to a toxic effect on some species of microorganisms.

Dissolved Oxygen

The maximum dissolved oxygen content of water at 20°C is 9.2 mg/l. Higher concentrations of dissolved oxygen can exist but represent unstable conditions in which the excess oxygen may be lost physically and not by biological consumption.

Too low a dissolved oxygen content will cause bacteria to stop metabolising. Dissolved oxygen (at least 0.5 mg/l) must remain at the end of the five day incubation period, to ensure that the limiting factor of the biological growth was insufficient organic matter as a food source and not dissolved oxygen. Most wastewater contains more oxygen demanding materials than the amount of dissolved oxygen available in air saturated water. In this case, it may be necessary to dilute the sample before incubation to bring oxygen demand and supply into appropriate balance.

Once an initial dissolved oxygen content is established, it is important to limit the amount of dissolved oxygen added or lost physically.

A few precautions that can limit physically changing the dissolved oxygen content are as follows:

1. Siphon H_2O into BOD bottle

2. Water seals maintained during incubation (additional oxygen cannot enter bottle)

3. Dilution water at 20°C ± 1°C initially to prevent contraction of water (prevent drawing air into bottle)

Nutrients

Bacterial growth requires nutrients such as nitrogen, phosphorus and trace metals. These nutrients are added to the dilution water in excess to ensure that they do not become the limiting factor in biological growth.

Organic Matter as a Food Source

The only source of organic matter as a food source should be from the sample and the seed. The seed source of organic matter is compensated for in the seeded blank. The most reliable results are produced by dilutions that result in a dissolved oxygen residual of at least 0.5 mg/l and a dissolved oxygen uptake of at least 2.0 mg/l after the five day incubation.

CONFIDENTIAL

CONFIDENTIAL

Summary of Method:

An aliquot of the sample is placed in a 300 ml BOD bottle along with appropriate nutrients and allowed to incubate in the dark at 20°C ± 1°C for five days. The reduction of the dissolved oxygen content during incubation is used to calculate the BOD. The limit of quantitation is 2 mg/l.

Sample Handling:

Samples for BOD analysis may degrade significantly during storage between collection and analysis, resulting in low BOD values. Samples should be stored (around 4°C ± 2°C) and analyzed within 48 hours of collection. No preservatives should be added.

Reagents:

1. Phosphate Buffer Solution: Dissolve 8.5 g KH_2PO_4, 21.75 g K_2HPO_4, 33.4 g Na_2HPO_4-$7H_2O$ and 1.7 g NH_4CL in about 500 ml of water. (All weights ± 0.100 g.) Dilute to 1 liter. Check the pH to be sure it is 7.2 ± .1. If solution is not within this range, remake and check pH again. Store at 4°C ± 2°C. Hold Time = 2 months.

2. Calcium Chloride Solution: Dissolve 27.5 (± 0.10) g of $CaCl_2$ in distilled water and dilute to 1 liter. Store at 4°C ± 2°C. Hold Time = 2 months.

3. Magnesium Sulfate Solution: Dissolve 22.5 (± 0.10) g $MgSO_4 \cdot 7H_2O$ in distilled water and dilute to one liter. Store at 4°C ± 2°C. Hold Time = 2 months.

4. Ferric Chloride Solution: Dissolve 0.25 g (± 0.02) $FeCl_3 \cdot 6H_2O$ in distilled water and dilute to one liter. Store at 4°C ± 2°C. Hold Time = 2 months.

5. Nitrification Inhibitor: 2 chloro-6-(trichloromethyl) pyridine or Nitrification Inhibitor 2533, Hach Chemical Co. or equivalent.

6. Glucose-Glutamic Acid Standard: Dry reagent-grade glucose and reagent-grade glutamic acid for 1 hour at 105°C ± 2°C. Desiccate to room temperature. Add .150 g (± 0.002) of each to distilled water and dilute to one liter. Autoclave 15 minutes in five 250 ml bottles with caps taped on loosely and store at 4°C ± 2°C. Hold Time = 2 months.

7. Glucose-Glutamic Acid Spiking Solution: Dry reagent-grade glucose and reagent-grade glutamic acid for one hour at 105°C ± 2°C. Desiccate to room temperature. Add .375 g (± 0.002) of each to distilled water and dilute to 100 ml (theoretical 5,450 mg/l spike). Autoclave approximately 15 minutes with bottle cap taped on loosely and store at 4°C ± 2°C. Hold Time = 6 months.

8. Ortho Tolidine, purchased.

9. .025 N Sodium Sulfite: Dissolve 0.1575 g (± 0.0010 g) Na_2SO_3 in 100 ml distilled water. This solution is not stable; prepare daily.

10. Seed: Obtained from Ephrata Wastewater Treatment Plant (clarifier effluent) or equivalent.

11. Acetic acid (1 + 1 solution). Store at room temperature indefinitely.

12. Potassium iodide solution (20 g/200 ml deionized water). Store at room temperature until it turns yellow (approximately six months).

CONFIDENTIAL

Procedure:

Prepare dilution water in 20 liter Nalge carboys. Once per month, these containers should be soaked in dilute chemsolv four hours, then nitric acid rinsed, followed by deionized water rinsing (or equivalent cleaning procedure). This should be done more frequently if growth is observed in the containers. Place in an incubator and bring the water to 19 ± 1°C. Check this temperature with a thermometer. The carboy will supply enough water for 60-65 BOD bottles. Determine the number of samples that can be run from each carboy including blanks and standards. Do not use dilution water from more than one carboy for each batch of samples.

Add about 1 ml each of the phosphate buffer, $MgSO_4$, $CaCl_2$ and $FeCl_3$ solutions per liter of water. Do not add nutrients prior to day of use.

Aerate for 30 minutes with the fish tank pump to saturate the dilution water with O_2. Check all hoses before using -- air, water, and siphon, and bottles for cracks in the necks. Pour into two BOD bottles and measure the dissolved oxygen with the D.O. meter and probe. Fill bottles completely and place in the incubator promptly. These are the unseeded blanks.

Add about 2 ml of seed/liter to the remaining dilution water and aerate for another 10 minutes. Fill 3 more BOD bottles, record the initial D.O., fill bottles completely and place in the incubator. These are the seeded blanks.

Adjust pH of each sample to pH 7 ± 0.5 with NaOH or H_2SO_4. Check each sample, especially effluents, with ortho tolidine for chlorine residual. (A yellow color appears when one drop of ortho tolidine is added to a sample having a chlorine residual.)

CONFIDENTIAL

Determine required volume of 0.025 N Na_2SO_3 by adding 1 ml of potassium iodide (KI) solution, then 1 ml of 1 + 1 acetic acid per 10 ml of neutralized sample/dilution. Titrate with Na_2SO_3 solution to the starch-iodine endpoint. Continue adding Na_2SO_3 solution until the blue color stops reappearing. A slight excess of Na_2SO_3 is preferable to a deficiency.

Add to the neutralized sample/dilution the volume of Na_2SO_3 solution determined by the above test. Note in notebook amount of Na_2SO_3 solution added per amount of sample.

Determine the aliquot needed for each sample by referring to notebook which lists the aliquots of common samples. If it is a sample from a new source, the BOD may be estimated from the TOC or COD. From the stoichio-metric ration, the BOD should not be more than about 2.6 times the TOC. However, the BOD is often less. The BOD and COD can be equal, but usually the BOD is less than the COD. The BOD should not exceed the COD. Once the BOD has been estimated, refer to Table 1 to obtain the approximate aliquot to be used.

Table 1. BOD Measurable With Various Dilutions of Samples

Using Percent Mixtures		By Direct Pipetting Into 300 ml Bottles	
% Mixture	Range of BOD	ml	Range of BOD
0.01	20,000-70,000	0.02	30,000-105,000
0.02	10,000-35,000	0.05	12,000- 42,000
0.05	4,000-14,000	0.10	6,000- 21,000
0.1	2,000- 7,000	0.20	3,000- 10,500
0.2	1,000- 3,500	0.50	1,200- 4,200
0.5	400- 1,400	1.0	600- 2,100
1.0	200- 700	2.0	300- 1,050
2.0	100- 350	5.0	120- 420
5.0	40- 140	10.0	60- 210
10.0	20- 70	20.0	30- 105
20.0	10- 35	50.0	12- 42
50.0	4- 14	100	6- 21
100	0- 7	300	0- 7

CONFIDENTIAL

When the BOD bottles have been setup for the addition of aliquots, the data should be recorded in the BOD logbook or LLENS system. Consult the LLENS SOP (BOD Application) for instruction. Use a wide mouth pipette or a graduated cylinder to place the appropriate aliquot in each BOD bottle. Be sure to obtain an aliquot of a well-mixed sample. Aliquots less than 1.0 ml should be obtained from a primary dilution of the sample. For example, for a 1/2 ml aliquot, make a 10x primary dilution by taking 10 ml of sample and diluting to 100 ml with deionized water, then take a 5 ml aliquot of this dilution. Fill the BOD bottles with the seeded dilution water and stopper. Exclude large air bubbles with tapping if necessary and obtain a water seal. After putting on the special plastic caps to prevent evaporation of the water seal, place the BOD bottles in a dark incubator kept at 20 ± 1°C for five days.

After the five day incubation period, remove the bottles and measure the dissolved oxygen. Calibrate the D.O. meter according to the manual. Fine tune the meter by performing the Winkler Titration.

For sample aliquots less than 50 ml, an oxygen depletion >2 mg/l when subtracted from the seeded blank must be obtained. The dissolved oxygen reading on any bottle must be greater than 0.5 mg/l for the reading to be valid. According to <u>Standard Methods</u>, the unseeded blanks should not show a depletion of more than 0.2 mg/l.

The BOD of the sample is calculated using the following formula:

$$\text{BOD, mg/l} = \frac{(B_2 - D) \times 300 \times \text{dilution factor}}{V} \text{ (if applicable)}$$

where D = Final D.O. reading of sample, mg/l

B_2 = Final average D.O. reading of the seeded blanks, mg/l

CONFIDENTIAL

V = Aliquot used in ml

At least one of the aliquots must yield a valid D.O. reading for the BOD to be acceptable. If several aliquots are within range, the BOD values are averaged and reported.

If BOD's are to be determined with nitrification inhibition, add approximately 3.33 mg (two shots from the dispenser) 2-chloro-6(trichloro methyl) pyridine to each BOD bottle before adding the dilution water and proceed as above.

<u>20 Day BOD</u> - A BOD_{20} is incubated for 20 days along with blanks, and a 5 day BOD standard.

Safety Precautions:

All chemicals and samples should be handled with care. Avoid contact with skin and wash hands immediately after use.

Quality Control:

1. A glutamic acid/glucose standard should be run with every batch of BOD's. Run two BOD standards at the beginning of the batch and two BOD standards at the end of the batch. A standard consists of a 5 ml aliquot (use autoclaved pipets) of the glucose/glutamic acid reagent diluted to 300 mls with seeded dilution water. After a five day incubation, the acceptance criteria is 218 mg/l ± 20% for the average of each pair of standards.

2. A duplicate (acceptance ≤20% relative percent difference) should be done with every batch.

CONFIDENTIAL

3. For matrix spikes, using an autoclaved pipet, add an aliquot (usually 1 ml) of the glucose/glutamic acid spiking solution to an aliquot (usually 100 mls) of sample in a beaker. (For best results, use a sample whose expected value is <100 mg/l.) Mix well, then pipet aliquots of the spiked sample into BOD bottles and dilute with seeded dilution water until full. After five day incubation, the acceptance criteria relative to the sample background is 75 to 125% recovery.

Note: High chlorine residual may cause problems with spike samples.

4. If carbonaceous samples are included in the batch, run a pair of carbonaceous standards as described in #1, but add the inhibitor to the standards before diluting them with seeded dilution water. Acceptance criteria has yet to be established.

NO235.W51
WQ METHODS #2
081392

CONFIDENTIAL

Prepared by: Robert Heisey Date: 8/18/92

Approved by: Beth Eby Date: 8/21/92

Approved by: Susan B. Shorter Date: 8-24-92

Lancaster Laboratories
Where quality is a science.

Procedural Amendment #1

Number: Analysis #235

Title: Determination of Biochemical Oxygen Demand in Water and Wastewater

Effective Date (listed on procedure): 08/25/92

Section(s) affected by change: Procedure

Reason for addition(s) or change(s): Method reference and QA audit

Change will be effective from (date): 07/12/93

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Procedure: *Add the following sentence*

At least two bottles (dilutions) for each client sample should be prepared.

NO235.W51
WQ Methods #2
071593

CONFIDENTIAL

Prepared by: Jean Slaugh Date: 7/14/93

Approved by: Seth Eby Date: 7/15/93

Approved by: Susan B. Shorter Date: 7/17/93

Lancaster Laboratories
Where quality is a science.

Analysis #221
Initiated Date: 09/17/85
Effective Date: MAR 29 1993

Ammonia-Nitrogen for Waters
(Titrimetric distillation procedure)

CONFIDENTIAL

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 350.2.

2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 4500-NH_3B/4500-NH_3E, pp. 4-77, 4-78, 4-81.

Scope:

The sensitivity limit for this procedure is 1.0 mg/l NH_3-N. The following method covers the determination of ammonia-nitrogen in drinking, surface, and saline waters, and domestic and industrial wastes.

Basic Principles:

The sample is buffered at a pH of 9.5 with a borate buffer and is then distilled into a solution of boric acid. The ammonia in the distillate is determined titrimetrically with standard sulfuric acid with the use of a mixed indicator.

Apparatus and Reagents:

1. An all glass distillation apparatus with an 800 ml Kjeldahl flask

2. pH paper

3. Microburets with reservoir

4. Sodium sulfite, Na_2SO_3, purchased; see label for expiration date

5. Borate buffer solution - Dissolve 5.0 ± .1 g of $Na_2B_4O_7$ or 9.5 ± .1 g $Na_2B_4O_7 \cdot 10\ H_2O$ in 1 liter deionized water. Add 176.0 ± 1.0 ml of 0.1 N NaOH to the above solution and dilute to 2 liters with deionized water. Stable 6 months at room temperature.

6. Mixed indicator solution - Dissolve 0.100 ± .005 g methyl red in 50.0 ± .5 ml of 95% ethanol. Dissolve 0.050 ± .005 g methylene blue in 25.0 ± .5 ml of 95% ethanol. Combine the two solutions. Stable 1 month at room temperature.

7. Boric acid solution - Dissolve 40.0 ± .5 g boric acid (H_3BO_3) in deionized water. Add 20.0 ± .5 ml of mixed indicator solution and dilute to 2 liters. Stable 1 month at room temperature.

8. Standard Sulfuric acid, 0.1 N - Add 6 ml of concentrated sulfuric acid (H_2SO_4) to approximately 1900 ml of deionized water in a 2 liter volumetric flask. Dilute to volume with deionized water. Stable 3 months at room temperature.

CONFIDENTIAL

9. Standard Sulfuric acid, 0.02 N - Add 200 ml of 0.1 N H_2SO_4 to approximately 400 ml of deionized water in a 1 liter volumetric flask. Dilute to volume with deionized water.

   NOTE: See Standardization SOP-WQ-002 for acid standardization instructions.

10. Ammonia-Nitrogen standard, 500 mg/l NH_3-N - Dissolve 1.9095 ± .0002 g NH_4Cl (which has been dried at 105° ± 2°C for approximately 2 hrs.) in deionized water and dilute to 1 liter. Preserve the solution with concentrated sulfuric acid to a pH <2. Stable 6 months at 4° ± 2°C.

Safety Precautions:

Always use caution when handling any chemicals, solvents, or reagents. Avoid inhalation and skin contact.

Sample Preparation:

Preserve the sample with concentrated sulfuric acid to a pH <2 and store at 4° ± 2°C. This is to be done by sample administration (preservation).

Procedure:

1. Rinse tubes with dilute HCl. Add approximately 500 ml of deionized water to an 800 ml Kjeldahl flask and steam clean the distillation apparatus. Collect at least 100 ml.

CONFIDENTIAL

2. Measure 500 ml of sample or an aliquot diluted to 500 ml with deionized water. Adjust the pH of the sample to 9.5 to 12 with NaOH. Add 25.0 ± 1. ml of borate buffer solution.

3. Transfer the solution to an 800 ml Kjeldahl flask; add boiling chips and antifoam if necessary.

4. Distill the solution into a 400 ml beaker containing approximately 50 ml of boric acid solution making sure the delivery tube is below the level of the receiving solution.

5. Collect 325 ± 5 ml of distillate.

6. Titrate the distillate with standardized 0.1 N H_2SO_4 or 0.02 N H_2SO_4 to a purple endpoint. Sometimes this endpoint is very weak. Add H_2SO_4 until no further color change is observed.

7. If more samples are to be done, any tube that had a sample greater than the detection limit must be steam cleaned (see step 1).

Quality Assurance:

Distill and titrate a deionized water blank (<LOQ or sample results 10 times the blank value) and one ammonia-nitrogen standard (± 20%) as described above, with each day's distillations. Perform one matrix spike (75 to 125% recovery) and one duplicate (± 20%) per batch (20 samples).

CONFIDENTIAL

Calculations:

$$mg/l\ NH_3\text{-}N = N\ H_2SO_4 \times \frac{14}{ml\ sample} \times ml\ titrant \times 1000$$

Statistical Information:

For statistical information refer to EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 350.2 - 5.

NO221.W51
WQ Methods #2
032593

CONFIDENTIAL

Prepared by: _____ Date: 3/25/93

Approved by: _____ Date: 3/25/93

Approved by: _____ Date: 3/26/93

Analysis #231
Initiated Date: 10/23/84
Effective Date: MAR 11 1993

CONFIDENTIAL

Total Recoverable Oil and Grease
(Extraction, Gravimetric)

References:

1. EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 413.1.

2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992, Method 5520B, pp. 5-43 through 5-44.

3. SW-846, 3rd Edition, Method 9070A, November 1990.

Scope:

The range is from 5.0 to 1000 mg/l of extractable material. This method is not applicable for samples that contain light hydrocarbons that volatilize at temperatures below 70°C. In such cases, an infrared method is advisable. A Soxhlet extraction method is suggested for samples containing polar, heavy petroleum fractions, or levels of oils and greases that make solubility in trichlorotrifluoroethane impossible or very difficult.

Basic Principles:

"Oil and grease" is any material recovered as a substance soluble in trichlorotrifluoroethane. This material is extracted from water by the intimate contact with the solvent, and then measured gravimetrically.

Trichlorotrifluoroethane may dissolve organic substances other than oils and greases resulting in a positive interference. The nature of the sample itself may cause interferences. If volatiles or heavy petroleum residuals are present, an alternative method is advisable.

Apparatus and Reagents:

1. 2 liter separatory funnel with a Teflon stopcock

2. Pleated filter paper, or equivalent

3. 125 ml tared distilling flask

4. Concentrated hydrochloric acid (HCl), stable indefinitely at room temperature

5. Sodium sulfate (Na_2SO_4), anhydrous crystal, stable indefinitely at room temperature

6. Trichlorotrifluoroethane (residue checked at <2 mg residue or IR checked at <0.01 absorbance units), stable indefinitely when stored at room temperature

7. Vacuum Pump, or equivalent

8. Glass Funnels (no stem), or equivalent

9. Analytical Balance

10. Solvent Capture System, or equivalent

11. Desiccator

CONFIDENTIAL

12. 100% vegetable or corn oil, stable indefinitely at room temperature

13. N-Propanol, stable indefinitely at room temperature

14. 10,000 mg/l Standard - Pipet 2.500 ± .001 g vegetable or corn oil into a 250 ml volumetric flask. Dilute to volume with N-propanol. Store, well stoppered at 4° ± 2°C. Hold time = 1 month.

Safety Precautions:

Trichlorotrifluoroethane is heavier than air and reduces oxygen available for breathing. It should be used only in a well ventilated area.

Procedures:

1. Collect about 1000 ml of the sample or an aliquot diluted to 1000 ml and mark sample level on bottle for later determination of sample volume.

2. Acidify the sample to a pH <2 by adding 5 ml of concentrated hydrochloric acid if the sample has not been preserved or if 500 ml or more of deionized water has been added to a reduced aliquot of sample.

3. Transfer the sample to a 2 liter separatory funnel.

4. Rinse the graduate or sample container with approximately 30 ml of trichlorotrifluoroethane and then transfer to the separatory funnel.

CONFIDENTIAL

5. Load the separatory funnels onto the shaker with the stopcocks open. Shake at intensity 30 for 30 seconds, then close the stopcocks and shake at intensity 45 for 90 seconds. Be sure to vent the separatory funnels before removing from the shaker! This will prevent pent-up pressure, generated while shaking, from blowing out the teflon stopper, with resultant loss of sample. Allow the layers to separate.

6. Filter the bottom layer through solvent-moistened pleated filter paper containing approximately 1 g Na_2SO_4 into a clean tared 125 ml distilling flask. If a clear solvent layer cannot be obtained, slowly drain emulsified solvent onto the crystals. Add small portions of Na_2SO_4 if emulsion is excessive. Na_2SO_4 is used to selectively absorb the water from the emulsion.

7. Repeat steps 5 and 6 two more times with approximately 30 ml additions of solvent, but first rinse sample container with each solvent portion. Combine all three extracts in tared distilling flasks and wash filter paper with an additional 10 to 20 ml of solvent. Add more sodium sulfate if necessary.

8. Place a flask on solvent capture system that has been pre-warmed (set temperature at approximately 7). The water gauge for bath water flow (in ccm) should be set at approximately 15 ccm. The gauge for condenser flow (in GPH) should be set at 4 GPH per sample (32 GPH for a full set-up). The samples will take about three hours to boil down to about 1 ml of freon.

CONFIDENTIAL

9. Insert tubing connected to a vacuum source for one minute to remove any solvent vapor from flask. As one flask is being aspirated, remove the neck of the next flask from the condenser. This will let the last few drops of freon escape. Wipe outside of flask to remove fingerprints and moisture.

10. Cool the flask in a desiccator for 30 minutes and weigh. Redesiccate for 15 minutes and reweigh. If second weight is more than 1 mg lower than first weight, redesiccate for 15 minutes more and reweigh. Continue in this way until constant weight is achieved.

Calculations:

$$\frac{\text{mg oil and grease}}{\text{liter}} = \frac{(A - B) \times 1000}{\text{ml sample}} \times 1000 - C$$

Where A is the weight of the flask and the residue in grams, B is the original weight of the flask (in grams), and C is the value of the blank (in mg/l).

Statistical Information:

For statistical information refer to EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, p. 413.1

Quality Assurance:

A sample of known oil and grease concentration (5 ml of the 10,000 mg/l standard, added to 1 liter acidified deionized water), a blank (1 liter acidified deionized water), duplicate and a spike (3 ml of the 10,000 mg/l standard added to the

CONFIDENTIAL

sample) should be analyzed along with each batch of 20 samples or 5 days, whichever comes first. It is best when QC samples are extracted on the first setup of samples. Try to be sure the sample chosen as a duplicate contains some of the analyte to be determined. One batch is not to continue for a period greater than five days. The QC acceptance ranges are as follows:

Standard	-	80 to 120% Recovery
Blank	-	<LOQ, or samples 10X blank value
Spike	-	75 to 125% Recovery
Duplicate	-	≤20% RPD

NO231.W51
WQ Methods #2
030292

CONFIDENTIAL

Prepared by: Catherine Cammarata Date: 3/2/93

Approved by: Betty Ann Eby Date: 3/2/93

Approved by: Susan S. Shorter Date: 3/10/93

Lancaster Laboratory

Summa Canister - TO-14 Analysis Plan

Canister Analysis Plan - November 30, 1993 DRAFT

1) Summary of Analytical Method

Samples will be collected in evacuated SUMMA polished stainless steel canisters. The canisters will be sent to the laboratory for analysis. Upon receipt in the laboratory, the canisters will be pressurized to approximately 30 psi (positive pressure). Up to 250 mL will be withdrawn from the canister for analysis by GC/MS. The air will pass through a Nafion dryer to remove the water vapor, and the volatile organic compounds will be cryogenically trapped. The trap will be heated and the contents swept into a gas chromatograph/mass spectrometer. The analytes will be quantified. A library search may be performed as needed.

The method reference is EPA method TO-14 "Determination of Volatile Organic Compounds in Ambient Air Using SUMMA Polished Canister Sampling and Gas Chromatographic Analysis".

The target analytes are listed below.

Allyl Chloride
Benzene
Benzyl Chloride
Bromomethane
Carbon Tetrachloride
Chlorobenzene
Chloroethane
Chloroform
Chloromethane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethylene
1,2-Dichloroethylene (c)
1,2-Dichloropropane
1,3-Dichloropropylene (c)
1,3-Dichloropropylene (t)
1,2-Dichlorotetrafluoroethane
Ethyl Benzene
4-Ethyl toluene
Freon 11 (trichlorofluoromethane)
Freon 12 (dichlorodifluoromethane)
Freon 113
Hexachlorobutadiene
Methylene Chloride
Styrene
Tetrachloroethylene
1,1,2,2-Tetrachloroethane
1,1,1,2-Tetrachloroethane
Toluene
1,2,4-Trichlorobenzene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethylene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene
Vinyl Chloride
Xylene (s)

DRAFT

2) Canister Cleaning and Certification

Equipment List

• Six liter SUMMA Polished Canister equipped with a valve which will isolate the canister from other components in the sampling system. The canister will be cleaned and evacuated by the laboratory and submitted by the laboratory to the field ready to be used for collection of the sample. Each canister will be tagged with a unique identification number.

Cleaning of SUMMA Canisters

Each canister will be cleaned unless purchased or rented from a supplier which certifies the canister as clean. The canister will be attached to a manifold system for cleaning in a batch mode. The canisters will be evacuated, filled with zero air, and evacuated again. This process will be repeated three times. Additional cycles may be repeated if additional cleaning is needed. The canisters may be heated up to 140°C to speed the cleaning process. During this heating, the valves should not be subject to the heating.

Certification of SUMMA Canisters

SUMMA canisters will be certified as clean by checking one canister from each batch of canisters prepared. The canister, filled with zero air, will be analyzed collecting at least 250 milliliters of air. The air must contain no target VOCs at levels greater than the limit of quantitation. If the canister does contain levels greater than the analytical limit of quantitation, the batch of canisters must be recleaned and certified.

Each SUMMA canister will be pressure checked following the certification of cleanliness. The canister should be pressurized to 30 psi, the pressure noted, and stored for approximately 24 hours. The pressure will be read again at the end of the test period using the same gauge as was used for the initial reading. The pressure must not vary more than 2 psi.

DRAFT

3) Analysis

Equipment List

• Hewlett Packard Model 5890II Gas Chromatograph connected to a Model 5971 Mass Selective Detector or equivalent system. The gas chromatograph is equipped with a capillary column: DB-1, 60m x 0.32 mm ID; or DB-624, 60 m x 0.32 mm ID, or a capillary column which gives similar or better resolving power for the target compounds.

• Entech Model 2000 Ambient Air Concentrator or equivalent system. This system is equipped with a Nafion dryer to remove water present in the sample, and cryogenic traps to concentrate the VOCs present in the sample of air. The last focusing cryogenic trap is equipped with a heater capable of quickly heating the trap to allow for a "plug" injection of the VOCs onto the chromatographic column.

• Gas Dilution System with Electronic Flow Controllers to permit at least two sources of standards and a balance gas to be blended to within ± 10% accuracy.

Standards

• Stock VOC standards prepared in pressurized cylinders in air or nitrogen. These are commercially prepared standards (Scott Specialty Gases, Plumbsteadville, PA and Alpha Gas, Morristown, NJ) at ppm(v) levels. The standards are traceable to NIST standards or certified by the manufacturer for the accuracy.

• Working VOC standards prepared by blending the purchase stock standards with humidified zero air. The standards are prepared by the dynamic calibration process and directed to the air concentrator.

• Internal standards prepared in zero air or nitrogen and held in a canister.

• A Laboratory Control Stock Standard (LCS) prepared by a commercial vendor at approximately 2 ppm(v). The working LCS is prepared by a dynamic calibration process to approximately 10 ppb.

Calibration of the analytical system

An initial calibration of the system with three levels of standards which span the analytical range (approximately 1 to 50 ppb) will be performed. If necessary, and additional level of calibration standard above the 50 ppb level can be added. The average response factor will be calculated for each target compound. The relative standard deviation for the response factors must be less than 25%.

DRAFT

Continuing calibration standards (made up of the mid level calibration standard) will be analyzed every twelve hours. The calculated response factor must be within 25% of the average response factor calculated for the initial calibration standards.

Three internal standards, chlorobromomethane, 1,4-difluorobenzene, and chlorobenzene d5 are added to the cryogen trap prior to the concentration of the VOCs from air samples or standards.

Tuning of the GC/MS

4-Bromofluorobenzene (BFB) is injected into the system daily. The key ions and ion abundance criteria identified in the TO-14 method must be met for the system to be in tune.

Certification of the Analytical System

Prior to the calibration of the system, the concentrator and GC/MS system are shown to be free of VOCs by analyzing humidified zero air. The system must show VOCs less than the limit of quantitation before calibration or analysis of samples can proceed.

Maintenance of the Analytical System

The Nafion dryer is heated to 100° C for 20 minutes while purging with dry zero air or nitrogen. This will be performed every 10th analysis or every day, which ever is more frequent. If polar compounds are to be determined, the micro purge and trap system must also be cleaned as appropriate. Following any cleaning, the system must be shown to be free of VOCs and recalibrated.

Quantification of results

Analytes will be quantified based on the internal standard calibration. Correction will be made for sample volume (volume at STP trapped on the cryogenic trap), initial and final sample pressure.

4) Performance Criteria

Accuracy

To perform the analysis of whole air samples using EPA method TO14, a certified standard must be used to indicate that the analytical system is operating in control and allow for demonstration of the accuracy of the measurements.

DRAFT

The Environmental Protection Agency has audit cylinders which are available for selected projects, however, these are not suitable for day to day operations. To allow for the demonstration of accuracy of the analysis, a laboratory control sample (LCS) should be available which will indicate changes in the calibration conditions or performance of the analytical measurement of whole air samples.

A set of six components has been prepared for this purpose. Standard purchased with concentration of 10 ppm(v). Using dynamic dilution equipment a standard of approximately 10 ppb(v) are be made. This standard will be analyzed daily.

The six compounds in the LCS are listed below:

Benzene
1,4-Dichlorobenzene
Ethyl Benzene
1,1,1-Trichloroethane
Trichloroethylene
Vinyl Chloride

A single analysis of the LCS will be performed daily or with each batch of samples (which ever is first).

Blanks

The result of the system blank analysis must be less than the limit of quantitation for the analysis. At minimum one system blank will be performed per day. After high level samples a blank should be analyzed to assure that there is no carryover in the system.

Precision

Duplicate analyses from the same canister must agree within 25% RPD. If one result from a pair is reported as < LOQ and the other as > LOQ, the first result is listed as the LOQ to calculate the RPD. Duplicate analysis of canisters is not a standard QC analysis. This analysis will be performed on a project by project basis. Analysis of field duplicates (colocated sampling) will be performed as required for each field sampling plan.

Limit of Quantitation

The limit of quantitation for the compounds is 1 ppb(v) for samples collected with at least 10 psia pressure in the canister. If high levels of target or nontarget compounds are present, the sample may be diluted or smaller aliquots of air may be analyzed. Both of these circumstances will affect the limit of quantitation.

Lancaster Laboratory

Quality Assurance Operations Manual

Lancaster Laboratories
Where quality is a science.

Initiated Date: 03/87
Effective Date: APR 14 1993

QUALITY ASSURANCE OPERATIONS MANUAL

SOP-QA-102

Title: Sample Log-in

Purpose:

In order to provide accountability of our results and to prevent sample loss or mix-up, a unique identification number is assigned to each sample.

Scope:

This SOP will cover the procedures to log-in samples received for analysis.

Procedures:

1. All samples received by laboratory personnel shall be delivered to the Sample Administration Group immediately upon arrival at the laboratory. The only exception to this requirement will be samples which are not tracked using the computerized Sample Management System (SMS). There are only a few cases where samples will be not be tracked using the SMS. These include samples which will be stored for a long period of time prior to analysis, (e.g., stability storage) and samples for special projects that will be reported in a narrative R&D report instead of on the usual computerized analytical reports.

CONFIDENTIAL

The procedures for sample log-in described in this SOP apply only to samples which are logged into the SMS. However, a written procedure for tracking any samples not entered into the SMS must be developed by the technical department responsible for the processing or analysis of those samples.

2. All client correspondence relating to samples shall also be transferred to the Sample Administration Group. This includes purchase orders, quotes, letters and completed entry request forms.

3. Personnel of the Sample Support Group shall log the samples into the computer as soon as practical after receipt. The computer will assign a unique identification number to each sample. Samples shall be logged in on the same day they are received with the following exceptions:

   a. Samples received during a holiday or between 6 p.m. on Friday and 6 p.m. on Sunday. These samples shall be logged-in on the next normal work day.

   b. Samples submitted by clients without any indication of the tests to be performed or with unclear or incomplete information. Every effort shall be made to contact the client on the same day as sample receipt.

   If same day entry is not possible, any special storage requirements (e.g., refrigeration) should be observed.

CONFIDENTIAL

4. Upon assignment of a sample number, the computer will generate a label which shall be attached to the sample container. The information on the label will include the LLI sample number, the client name, the storage location, a list of analyses requested (by analytical method number), a bottle code indicating container and preservative type, and a unique bar code.

5. Addition of preservatives to unpreserved samples will be the responsibility of the Sample Administration Group. Preservation should be performed immediately after log-in. A list of preservatives required for routine analyses may be found in the Fee Schedule.

6. All entries in preservation notebooks and on client paperwork shall be made in ink. The error correction procedure given in SOP-QA-109 shall be followed for any changes made in the documentation.

7. After samples are logged-in (or preserved, if required) they shall be stored in the computer-assigned location. If the computer-assigned location is inappropriate for the sample, the location code may be changed by manually overriding the computer.

8. The sample number assigned to each sample shall be used to identify the sample in all records, including laboratory notebooks, instrument printouts, and

CONFIDENTIAL

laboratory reports. The sample number shall be used to identify all additional containers of the sample which may be created during the sample preparation and analysis. This includes subsamples, extracts and digestates.

SOPQA102.W51
SOP QA #1
032493

CONFIDENTIAL

Prepared by: M Louise Hess Date: 4/8/93

Approved by: J William Hershey Date: 4/8/93

Read and understood by: _____ Date: _____

Lancaster Laboratories
Where quality is a science.

Initiated Date: 3/87
Effective Date: OCT 23 1992

QUALITY ASSURANCE OPERATIONS MANUAL

SOP-QA-104

Title: Chain-of-Custody Documentation

Purpose:

In order to demonstrate reliability of data which may be used as evidence in a legal case or required by a regulatory agency, an accurate written record tracing the possession of the sample must be maintained from the time it is logged into the computer system until the last analysis is validated.

Scope:

Procedures for initiating and maintaining chain-of-custody (COC) documentation are described in this document.

Definition:

A sample is in custody if it is in any one of the following states:

1. In actual physical possession.

2. In view after being in physical possession.

3. In physical possession and locked up so that no one can tamper with it.

4. In a secured area, restricted to authorized personnel. (e.g., any one of the individual laboratories in the building)

CONFIDENTIAL

Procedures:

1. Chain-of-custody documentation shall be kept [illegible] request of the client or for any samples [illegible] known to be involved in a legal dispute [illegible] all analytical data, it is extremely important that documentation be filled out completely and accurately with every transfer. If changes to the form need to be made, the error correction procedure given in SOP-QA-109 shall be followed.

2. If requested by the client, chain-of-custody documentation will begin with the preparation of bottles. A form (see Attachment 1) will be initiated by the person packing the sample bottles for shipment to the client. If the delivery of bottles is via our Transportation Department, the driver shall sign the form when relinquishing the bottles. Drivers must also sign chain-of-custody forms when picking up samples which require such documentation.

3. When samples arrive at the laboratory, a member of the Sample Administration Group will receive them and sign the chain-of-custody form, if one is provided with samples. If the sample was picked up by our Transportation Department, the driver must sign to indicate relinquishing the sample to Sample Receipt.

4. Samples will be logged into the computer as described in SOP-QA-102. Sample Administration personnel shall enter the analysis number for "laboratory chain-of-custody." A lab note to inform analysts of the need for chain of custody will be automatically added to sample labels.

CONFIDENTIAL

5. Sample Administration personnel shall initiate an internal "Laboratory Chain-of-Custody" form (Attachment 2) for each type of container in the sample group and relinquish the samples to a sample custodian or designated key holder, who will store the samples in the assigned locked location. This change of custody from sample entry to storage shall be documented on the chain, as well as any interim exchanges for preservation, homogenization, or temporary hold storage. The internal chain-of-custody forms will then accompany the samples throughout the lab. A master list of chains started for each sample group should also be initiated at this time.

6. At this point, the original copy of the external client chain-of-custody will be filed with Accounts Receivable, to be returned to the client with the invoice. Other copies of the external COC (pink or yellow) will be filed with the client's paperwork file.

7. All signatures documenting changes of custody will use the following format: first initial, full last name, and employee number. Dates will include month, day, and year, and all time will be in military time. Black ink is preferred. Pencil or red ink is not acceptable. Attachment 2 shows examples of chain-of-custody documentation.

8. Sample handling should be kept to a minimum. Analysts requiring use of a sample will requisition it through the computer requisition program. During the hours where Sample Support is manned by sample custodians, the custodian will receive the computerized requisition, and remove the sample from storage. The custodian will ensure that the bottle type listed on

CONFIDENTIAL

the chain of custody matches the bottle type being relinquished, then sign the "released by" column to indicate the sample has been relinquished, and enter the date and time. The analyst shall sign the "received by" column and note the reason for change of custody <u>before</u> taking the samples to their work area. It will be a shared responsibility of technicians and sample custodians to ensure that forms are signed with each transfer.

9. All changes of custody must be documented on the form. The following changes of custody shall be handled as follows:

   a. Signatures involving transfers from one shift to another shall be the responsibility of the technician who originally acquired the sample from Sample Support. When samples are then returned to storage, the person returning the samples shall be responsible to sign the "released by" column, and to ensure that samples were properly received by the custodian with his/her signature in the "received by" column.

   b. Occasionally a sample container will be needed for analysis by a technician in a department while it has been signed out to a technician in another department. It will be the responsibility of the first technician who received the sample to see that the second technician needing the sample signs the COC for receipt and return of the sample to the first technician.

CONFIDENTIAL

c. In situations where a sample group must be split between departments working on different analyses, a supplemental chain of custody may be initiated by Sample Support. This supplemental chain will be used to accompany that portion of the group which is needed by a second department, when another department has part of the group and the chains of custody for the whole group. Initiating supplemental chains of custody may only be done by Sample Support and Expressmail, and should be used only when necessary to minimize paperwork and confusion. Sample Support will also document on a Masterlist, all chains and supplemental chains initiated for any sample group. This Masterlist of chains will be made available to Data Packages who collect all chains for packages.

d. Weekend workers do not always have a sample custodian available. During these times the Lancaster Labs security personnel function as key holders for the storage areas. Technicians requiring use of samples over these times must requisition samples the previous day. These samples will be placed in the sample support hold walkin by a sample custodian. It will be necessary to page the security staff on weekends to acquire access to the hold walkin. Technicians will sign the COC for their own sample release by recording "SSG Storage" in the "Released By" column, and again in the "Received By" column when the sample is returned to the hold walkin.

e. Some samples are released by Sample Support and stored temporarily in other areas of the laboratory e.g. GC/MS Volatiles. During this time they may be

CONFIDENTIAL

worked on by several people in that department. Each of these people must sign for change of custody. These samples when completed are then returned to Sample Support. It will be the responsibility of the department who is temporary storage to see that all necessary signatures are on the chain of custody form before transferring samples and forms, at the same time, to Sample Support. It is also important to return complete sample groups as soon as possible after verification of data, because the chains may be required for data packages.

10. Analysts in possession of samples shall remove the aliquot required for analysis and return the sample to storage, as described in 9 below, with a minimum of delay. During the time of possession, samples must remain in the analyst's view or be in a designated storage area within a secure lab restricted to authorized personnel.

11. If additional containers of the sample are created (e.g., subsamples, extracts, distillates, leachates, etc.) an additional chain-of-custody form marked with container type shall be initiated to accompany the new container. Each department in the lab has specifically designed chain-of-custody forms which must be used for the new containers they create. All changes of custody involving handling of new containers within the department (e.g, analysis, storage, vials on instruments, etc.) shall be documented on the department specific chain. Any special handling or documentation requirements for department chains that are specific to any one department, should be described in a department SOP.

CONFIDENTIAL

The only exception to the additional container form requirement will be for clients who specify chain of custody for the original sample only. In this case, no forms for sample preparation will be required.

After completion of new container sample analyses for department chains, the completed forms will be collected by the department's Data Package coordinator to be given to the Data Package department with the package data.

12. After analysis, samples shall be relinquished to a sample custodian who will return the samples to locked storage. The forms which remain in Sample Support shall be signed again upon separate storage, and the sample custodian will review the forms to ensure that all transfers are completely documented before filing the forms. Sample custodians will not return a sample to its storage location without signing an accompanying chain.

13. All completed forms for the original sample containers will be retained in files in Sample Support. The Data Package group will retrieve these forms to be copied for inclusion in the data packages. All original forms will either returned to the client or retained here, depending on the client's wishes.

14. All sample handlers in Sample Administration, Sample Support, and technical centers will make every attempt to ensure that all changes of custody are properly documented. Disciplinary action may be taken for employees who fail to comply with this important requirement.

CONFIDENTIAL

15. In the event that a signature or other information is not recorded on the chain of custody, the Sample Support and Data Package groups shall determine [illegible] information is missing by checking computer acquisition records, raw data, or Sample Support work schedules. The corrected information shall be added to the chain of custody and signed and dated with the current date of information entry.

SOPQA104.W51
SOP QA #1
102392

CONFIDENTIAL

Prepared by: [signature] Date: 10/23/92

Approved by: [signature] Date: 10/23/92

Approved by: Kathleen M. Lawson Date: 10/25/92

Read and understood by: _____ Date: _____

SOP-QA-104
Initiated Date: 3/87
Effective Date: OCT 23 1992
Page 9 of 10

Attachment 1

Analysis Request/Environmental Services

Chain of Custody

Lancaster Laboratories
Where Quality is Science

Please print. Instructions on reverse side correspond with circled numbers.

1 Client: _____ Acct. # : _____
Project Name/# : _____
Project Manager: _____
Sampler: _____

4 Matrix

5 Analyses Requested

For LLI use only
FSC: _____
SCR # : _____

2 Sample Identification

6 Remarks

7 Turnaround time requested (please circle): Normal Rush
(Rush TAT is subject to LLI approval and surcharge.)
Rush results requested by (please circle):
Fax Fax # : _____
Phone Phone # : _____

8 Data package options (please circle if requested):
QC Summary
Tier I (NJ)
Tier II (NJ)
EPA CLP

Site-specific QC required? Yes No
(If yes, indicate QC sample and submit triplicate volume.)

Data Package Chain of Custody required?
Yes No

9

Relinquished by	Date	Time	Received by	Date	Time
Relinquished by	Date	Time	Received by	Date	Time
Relinquished by	Date	Time	Received by	Date	Time
Relinquished by	Date	Time	Received by	Date	Time
Relinquished by	Date	Time	Received by LLI by	Date	Time

2425 New Holland Pike, Lancaster, PA 17601-5994 (717) 656-2301 Copies: White and Yellow copies accompany samples to lab. Pink is retained by client. 4/26/91 #2102

CONFIDENTIAL

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601-5994

Attachment 2
Locked Storage Chain of Custody

ORIGINAL SAMPLE

Client/Project: ABC Engineering

Preservative: none

Matrix: [illegible]

Sample # Range of Entry Group: 1716402 - 416

Bottle Type: 1/2 [illegible] plastic

SDG: ABC1

Sample Number(s)	Released by	Received by	[illegible]	Time	Reason for Change of Custody
1716402 -416	A. Hutchison 210	Ca Smith 266	[illegible]	1115	from entry to storage

CONFIDENTIAL

2016 Rev. 2/14/92

RMC

STANDARD OPERATING PROCEDURES

RMC'S AQUATIC TOXICOLOGY LABORATORY INTERNAL METHODS MANUAL

QUALIFICATION STATEMENT

The methods in this internal manual were derived from the US EPA acute toxicity test manual (EPA/600/4-90/027) and the US EPA chronic toxicity test manuals (EPA/600/4-87/028 and EPA/600/4-89/001). They are intended to provide RMC toxicity personnel guidance where specific regulatory procedures are not available or mandated. The methods in this internal manual are to be followed, except when NPDES permits dictate other protocols or specific changes.

REVISION SHEET

Rev. No.	Page	Section or Paragraph

TABLE OF CONTENTS

Page

LIST OF FIGURES 4

LIST OF APPENDICES 4

FIELD METHODS 5

A. Effluent Sample Collection 5
B Receiving Water Collection 6
C. Sediment Collection 6
D. Volumes of Sample Required and Collection Containers 6
E. Additional Information 7

LABORATORY METHODS 8

A. Organisms Used and Types of Toxicity Tests Conducted 8
B. General Information 9
C. Protocol Summary/Operating File 13
D. Screening Bioassay 13
E. Rangefinding Bioassay 14
F. Acute Definitive Bioassay 15
G. Chronic Effluent Toxicity Tests (seven-day test) 16
H. Sediment Toxicity Tests (ten-day test) 19
I. Statistical Methods 20
J. Quality Assurance/Quality Control 20

LIST OF TERMS USED IN BIOMONITORING 23

LIST OF REFERENCE MANUALS RECOMMENDED FOR USE IN BIOMONITORING . 27

LIST OF FIGURES

Figure 1 Data Flow Diagram for RMC Chronic Toxicity Testing Data Analysis
Figure 2 Flow Chart for Statistical Analysis of Fathead Minnow - Larval Survival Data
Figure 3 Flow Chart for Statistical Analysis of Fathead Minnow - Larval Growth Data
Figure 4 Flow Chart for Statistical Analysis of Ceriodaphnia - Survival Data
Figure 5 Flow Chart for Statistical Analysis of Ceriodaphnia - Reproduction Data

LIST OF APPENDICES

Appendix A Laboratory Training Curriculum Summary
Appendix B Sample Handling
Appendix C General Bioassay Laboratory Logs
Appendix D General Laboratory Protocols
Appendix E Bioassay Equipment and Instruction Manuals
Appendix F Water Analysis Parameter List
Appendix G Acute Toxicity Test Forms
Appendix H Chronic Toxicity Test Forms
Appendix I Sediment Test Forms

RMC ENVIRONMENTAL SERVICES, INC.
AQUATIC TOXICOLOGY LABORATORY

FIELD METHODS

A. Effluent Sample Collection

Effluent is collected at the point of discharge to the receiving water or at the NPDES permit specified sampling location in the discharge channel leading to the final discharge where no further addition to the effluent occurs. An individual with knowledge of the facility and NPDES permit specified sampling locations should direct the collection of the effluent samples.

The decision on whether to collect grab or composite samples is based on the objectives of the test and an understanding of the short and long term operations and schedules of the discharges.

Grab samples of effluent are collected using a sampling pump. A plastic bucket and nylon rope are used when a sampling pump is not appropriate. Grab samples are routinely used in screening, rangefinding, and definitive bioassays.

Composite samples of effluent are collected using commercially available automatic samplers. The samplers used by RMC Environmental Services, Inc. (RMC) are ISCO samplers or SIGMA samplers, which are designed for toxics sampling and are fitted with 2.5 gallon glass jars.

If the data from the samples are to be acceptable for use in the NPDES Program, the lapsed time from collection of a grab or composite sample and its first use for initiation of a test, or for test solution renewal, should not exceed 36 h. Composite samples should be chilled during collection, where possible, and maintained at 4°C until used.

Samples collected for off-site toxicity testing are to be chilled to 4°C when collected, shipped iced to the central laboratory, and then transferred to a refrigerator (4°C) until used. Every effort must be made to initiate the test with an effluent sample on the day of arrival in the laboratory.

Samples may be shipped in 4-L (1-gallon) CUBITAINERS® or new plastic "milk" jugs. All sample containers should be rinsed with source water before being filled with sample. After use, CUBITAINERS® and plastic jugs are punctured to prevent reuse.

Sample chain-of-custody forms should be completed at the time of collection with sample identification, date and time of collection, and contain the appropriate signatures. Sample bottles should also be labeled with the sample identification and the date and time of collection.

B. Receiving Water Collection

The source of the dilution water used in effluent toxicity tests will depend largely on the objectives of the study. If the NPDES permit requires use of receiving stream water as dilution water, a suitable collection site must be chosen. The sample location should be close to the outfall, but upstream from or beyond the influence of the effluent. The grab samples should be taken at mid-stream and mid-depth if accessible, or as close to this as possible. Before taking the actual sample, rinse the sample container(s) with the sample and then fill completely. Label the sample bottle, and the chain-of-custody form with the date and time of collection and the name of the person collecting the sample(s).

C. Sediment Collection

Field collection of sediments for use in toxicity tests should follow the methods outlined in the ASTM quidance document E1391-90 or the specific protocol required by the study.

D. Volumes of Sample Required and Collection Containers

1. Screening Bioassays

For screening tests, a minimum sample volume of one gallon of effluent is required. The type of sample, grab or composite, and the collection interval is permit specific. All containers should be rinsed with sample before being filled. All containers should be properly labeled. The samples are packed in insulated containers (coolers) with ice packs for transport.

2. Rangefinding Bioassays

A minimum sample volume of one gallon of effluent and two gallons of upstream receiving water, when used as diluent, are collected as grab samples and poured into labeled one-gallon plastic containers. Effluent is collected from the same location as samples for a screening bioassay. Samples for definitive bioassays are collected at the same time and locations as the rangefinding samples. The samples are packed on ice in insulated containers for transport.

3. Acute Definitive Bioassays

Two gallons of effluent and three gallons of receiving water, when used as diluent, are collected as composite and grab samples, respectively, and poured into labeled one-gallon plastic containers. The effluent and diluent samples are transported on ice in insulated containers. Grab samples of effluent are used occasionally if dictated by the permit.

4. Flow-through Bioassays

When flow-through testing is specified in the permit, a larger volume of effluent and receiving water is required. A minimum volume of thirty gallons of effluent and forty-five gallons of receiving water, when used as diluent, collected as composite and grab samples, respectively, are collected daily. The effluent is collected with an automatic sampler into 15 gallon polyethylene Del drums and the receiving water is collected with a sampling pump into 15 gallon polyethylene Del drums.

5. Chronic Seven-day Bioassays

Three collections of two gallons of effluent, and seven to ten gallons of receiving water, when used as diluent, are collected and poured into labeled one-gallon plastic containers. Composite effluent samples and receiving water grab samples are collected. The samples are packed on ice in insulated containers for transport.

6. Sediment Bioassays

The volume of sediment required is organism and test specific. Consult the appropriate ASTM guidance document, US EPA guidance, or Army Corps of Engineers' manuals for specific information.

E. *Additional Information*

Effluent and receiving water may be collected during a rainstorm but are not collected during flood conditions or other unsafe conditions. All sampling apparatus and containers are rinsed with source water prior to collecting the samples for the test. Bioassay sample containers are labeled with the sample source and date and time of collection. All samples are logged on chain-of-custody forms with the sample identification, the date and time of collection, and should contain the appropriate signatures.

In some instances, samples for chemical analyses of NPDES permit specified parameters are collected at the same time as the bioassay samples. If chemical sampling is requested in advance, the samples are collected in the appropriate containers, and properly preserved when required. The samples are packed on ice in insulated containers for transport.

For chemical analyses, an Analytical Request Form and Chain of Custody Form are completed at the time of sample collection.

Sample temperature and dissolved oxygen content are measured and recorded in a field notebook, if appropriate, at the time of each sample collection. Any additional pertinent information about the weather, operation of the facility, flow of the effluent, the receiving water, or the collection sites is recorded in the field book at the time of collection.

All samples for bioassay testing are transported to the Aquatic Toxicology Laboratory in Spring City, Pennsylvania. Upon receipt in the lab, the samples are logged into the sample log book with the sample temperature, dissolved oxygen, salinity (if appropriate) the sample type, client, job number, and the date and time of collection. Testing is usually initiated the same day or if this is not possible, the sample is stored at 4 C in the walk-in cooler and used within 24 hours of the time of collection. The walk-in cooler remains locked at all times and the temperature is recorded daily. All samples are placed in the areas designated for bioassay samples.

LABORATORY METHODS

A. Organisms Used and Types of Toxicity Tests Conducted

RMC routinely uses two species of aquatic organisms in the bioassays conducted using industrial and municipal effluents. These are:

1. fathead minnow (Pimephales promelas) (P. promelas)
2. the freshwater cladoceran Ceriodaphnia dubia (C. dubia)

On a nonroutine basis, RMC uses the following species of aquatic organisms:

1. rainbow trout (Oncorhynchus mykiss)
2. bluegill (Lepomis macrochirus)
3. white perch (Morone americana)
4. striped bass (Morone saxitalis)
5. mysid shrimp (Mysidopsis bahia)
6. the freshwater cladoceran Daphnia magna
7. the amphipod Hayalella azteca
8. the midge Chironomus tentans
9. the amphipod (Ampelisca abdita)
10. Asiatic clam (Corbicula fluminea)
11. Zebra mussel (Dreisseina polymorpha)
12. Other field collected or cultured organisms as specified or requested.

These test organisms are used in screening, rangefinding, definitive, seven-day, ten-day, fifteen-day, and twenty-eight day bioassays. The methods described for each type of bioassay are described using the most routinely used test organisms, fathead minnow and C. dubia, and are followed in routine toxicity testing of effluents. These methods may be applied to other recommended test species, and may be slightly modified to better address the reason for conducting a test. Any modification made will be consistent with the direction of changes in EPA protocols. The methods described in this document are meant to address the proper laboratory techniques as opposed to the specific test organisms and will be periodically revised and updated. Specific procedures are contained in RMC's Quality Assurance Program.

B. General Information

1. Chemical Cleaning

Any new or used item of glass, nalgene, or metal is chemically cleaned prior to being used in a toxicity test. The following procedures are used:

a. US EPA Glassware Cleaning Procedure

(1.) Soak 15 minutes in tap water, and scrub with detergent, or clean in an automatic dishwasher.
(2.) Rinse twice with tap water.
(3.) Carefully rinse once with fresh, dilute (10%, V:V) hydrochloric or nitric acid to remove scale, metals, and bases. To prepare a 10% solution of acid, add 10 ml of concentrated acid to 90 ml of deionized water.
(4.) Rinse twice with deionized water.
(5.) Rinse once with full-strength, pesticide-grade acetone to remove organic compounds (use a fume hood or canopy).
(6.) Rinse three times with deionized water.

- All test chambers and equipment should be thoroughly rinsed with the dilution water immediately prior to use in each test.

b. NJAC 7:18 - Glassware Cleaning Procedure

(1.) As soon as practical, rinse with acetone to remove organic compounds then at least twice with laboratory grade water.
(2.) Secondly, soak and wash with warm water and laboratory detergent (Alconox or equivalent) then rinse at least twice with 50 C or warmer laboratory grade water.
(3.) Finally, rinse with a fresh 5 percent hydrochloric acid or nitric acid solution for the removal of metals and bases than rinse again with 50 C or warmer laboratory grade water.
(4.) Allow to drain and then store under clean conditions.

Chemically cleaned items are inverted and stored on shelves in the laboratory until needed. After use in a toxicity test, a contaminated item is emptied of its contents (if applicable), rinsed with tap water, and placed in a designated area until the item may be chemically cleaned. Contaminated items are chemically cleaned as soon as possible after a toxicity test is terminated. Any new disposable plastic items are rinsed with distilled water before use. Sample collection containers are rinsed with sample before filling.

2. Aeration

Aeration for the test organism holding and culture units, and if needed, for test chambers is supplied by Whisper air pumps. These pumps supply oil-less air to the organisms. Adjustable gang valves are fitted to the air pumps. Airline tubing connects the gang valve to an airstone or small internal diameter glass tubing or pipette positioned in a test chamber when aeration is required. The airstone and the portion of airline which contacts the test solution are cleaned when a test is complete.

Test solutions are aerated when the dissolved oxygen concentrations is less than 60% of saturation or when a sample is supersaturated. Single bubble aeration may be maintained in the bioassay containers during a test when this is required. If it is not possible to aerate the test chambers, the dilution water will be aerated, and if necessary the effluent sample will be aerated before test solution preparation.

3. Lighting and Temperature

Acute screening and definitive tests, and chronic tests are conducted in a temperature controlled room. All testing areas have automatically controlled 16-hour light, 8-hour dark photoperiods. The temperature is automatically controlled to maintain the test temperature within $\pm$ 1 C. This is accomplished using a heat and air system which can be programmed for the desired temperature. The room temperature is recorded daily. A polypropylene water bath is also used for testing when needed. A Forma Scientific Materline 2095 Bath and Circulator is used to maintain the test temperature in the water bath. Water level in the bath is kept at a level slightly less than solution levels in the test containers. A standard temperature of 20 $\pm$ 2 C or 25 $\pm$ 2 C is maintained in the acute screening and definitive tests. The test temperature of 25 $\pm$ 1 C is maintained in chronic tests.

4. Care and Handling of the Organisms

Care is taken not to injure the organisms when placing them in the test chambers. All organisms are transferred using a pipette, eye dropper, or wet net, whichever is appropriate. Any organism that is inadvertently injured or that appears damaged or unhealthy is discarded.

C. dubia young to be used in rangefinding, screening, and definitive bioassays are obtained by maintaining adult females in individual 30 ml plastic beakers. Each beaker receives 0.1 ml of YCT (Yeast/Cerophyll/trout chow) suspension and 0.1 ml of a concentration of the green algae Selenastrum capricornutum. Neonates are transferred to another beaker containing DMW, YCT, and algae prior to use in a bioassay. Eye droppers with a bore size of approximately 2 mm are used to transfer the organisms. Neonates used in screening, rangefinding, and definitive acute toxicity tests are less than 24 hours old.

C. dubia young for use in 7-day toxicity tests are obtained by maintaining adult females in individual beakers for one to two weeks prior to test initiation. Each 30 ml plastic beaker contains 15 ml of DMW and one C. dubia, receives 0.1 ml of YCT, and 0.1 ml of Selenastrum daily. The tapered cups are 40 mm in diameter and 45 mm in height; the solution depth is 15 mm. The cups are rinsed with distilled water and DMW prior to use. Handling a number of cups and viewing their contents is facilitated through the use of a multi-holed tray and a light-box. Neonates are pooled and transferred to a separate beaker containing DMW, YCT, and algae prior to use in a bioassay. Eye droppers used to transfer adults and neonates have a bore size of approximately 2 mm. Less than twelve hour old neonates, all produced within the same eight hour time period, are used in the 7-day chronic toxicity test.

Young fathead minnows P. promelas (< 14 days old) are used in screening, rangefinding, and definitive tests. They are purchased from a commercial laboratory and shipped overnight in cubitainers containing culture water. The fish are then placed in a five gallon aquarium or two gallon rubbermaid container, one to five days prior to test initiation for acclimation purposes. All handling of fish is done using a wet aquarium net, or large bore pipette, if appropriate.

Less than 24 hour old fathead minnow larvae are used in the 7-day toxicity test. They are purchased from a commercial laboratory where they are hatched the morning prior to test initiation, placed in a cubitainer containing culture water and shipped overnight. Minnows are acclimated to laboratory environmental conditions for several hours before test initiation. Transfer pipettes have a bore size of approximately 5 mm and are used to transfer individual larva from the holding pan to the test containers.

5. Beginning of Tests

Bioassays are begun as soon as possible after sample collection, usually within 24 hours. The samples are temperature adjusted in a water bath to 20 ± 2 (acute tests) or 25 ± 1 C (acute or chronic tests). Fathead minnows for the acute and chronic tests are randomly netted or pipetted out of an aquarium or holding pan and placed in the test vessels. C. dubia neonates from broods of eight or more are used in the 7-day chronic test. All neonates released during the day of test initiation are pooled and used to initiate the test. The C. dubia are randomly placed in the test vessels.

6. Controls

A control uses the same species in the same number and size of the test treatments, however, the solution does not contain any of the effluent being tested. Control survival must meet the acceptability criteria in order for the test to be valid. Unacceptable control survival will usually result in the test being repeated. If

control survival is unacceptable when upstream or receiving water is used, the data may still be used if upstream point or nonpoint source(s) could have been responsible for the mortality. Laboratory controls consist of DMW, receiving stream controls consist of water collected immediately upstream from the discharge of concern but outside the zone of receiving water-effluent interaction.

7. Dilution Water

The primary objective of NPDES permit-related toxicity testing is to estimate the inherent chronic toxicity of the effluent, therefore, a standard dilution water (moderately hard) is prepared and used. This water is prepared according to procedures outlined in EPA/600/4-89/001. Distilled water is decanted through a deionizing column (Barnstead mixed bed) and diluted in a ratio of two parts Perrier water to eight parts deionized water. The 20% DMW is aerated for at least 24 hours prior to use as either acclimation water, dilution water, or control water.

The culture and holding water for P. promelas and C. dubia is 20% DMW. The water is prepared according to the procedures cited above. The 20% DMW is aerated for 24 hours prior to use. Sufficient volumes of 20% DMW are mixed and kept available at test temperatures at all times. Culture water is kept no longer than fourteen days.

8. Standard Length and Weight Measurements of Fish

Standard length is measured by positioning a fish on its right side with the head pointing to the left of the person making the measurement. Standard length, the distance from the tip of the snout to the end of the caudal peduncle, is measured to the nearest 0.1 mm through the use of a ruler. Individual fish are blotted with a paper towel to remove excess moisture and weighed to the nearest 0.1 gram on an American Scientific Products S/P Model 120 electronic balance.

9. Physical/Chemical Analytical Methods

Several physical/chemical parameters are routinely measured before, during, and after bioassays are conducted. The following list details the standard methods used by RMC procedure number. These procedures are issued to personnel on a controlled copy basis; use only the most current revision of each.

Procedure Number	Parameter
EB4	Dissolved Oxygen: Membrane Electrode
EB7	Temperature
EB8	pH
EB11	Conductivity
EB17	Alkalinity as mg/l $CaCO_3$
EB18	Hardness as mg/l $CaCo_3$
EB19	Total Residual Chlorine

At a minimum the following measurements are made for each bioassay: temperature, dissolved oxygen, and pH are measured at the beginning and end of each test day. Conductivity is measured in each new sample. Alkalinity and hardness are measured in high test concentration and control solution on each new sample.

C. Protocol Summary/Operating File

Prior to initiating any toxicity studies, a Protocol Summary must be completed by the Project Manager. The Protocol Summary contains all pertinent information needed in order to conduct the required testing.

A copy of the Protocol Summary should be placed in the job Operating File. The Operating File should contain the Protocol Summary, an Analytical Request Form (if applicable), a copy of a previous report (if applicable), and a copy of the client's permit (if applicable). The Operating File is also completed by the Project Manager and is the "working file" that is given to the bioassay lab technician for conducting the testing. At test completion, all job Operating Files are placed in the file cabinet in the Aquatic Toxicology Laboratory for future reference. Acute and chronic Protocol Summary Forms are located in Appendices D and E, respectively.

D. Screening Bioassay

1. Description

A screening bioassay consists of a 24 to 48 hour exposure of one species of test organisms to a single sample of effluent and a control. The effluent concentration is 100 percent (undiluted) effluent. The control is the same species, number, and size of test organisms but uses DMW culture water or receiving water as specified.

A screening bioassay is used to determine if a sample is acutely toxic to the test organisms. Separate screening bioassays utilizing samples from the same discharge collected at different times of day can be used to detect variability in acute toxicity of the wastewater.

A single control for each test organism is used when more than one screening bioassay is begun within a short period of each other on a particular day. Two controls are used when there is a longer time period between times of test initiation.

2. Test Organisms

Consult the appropriate procedure for the type of test organism, number of replicates, number of organisms per concentration, and test solution volume. Usually a minimum of twenty test organisms per concentration are required. The organisms remain in the original solution for the duration of the test, and are not fed during the test period.

Date recorded for the organisms are: life or death, motility or immotility, and exhibition of normal equilibrium or the loss of equilibrium. All dead individuals are removed from the test containers. Data for each test are tabulated at the conclusion of a test.

3. Other Organisms

The species of test organisms used depends upon the objectives of the study. All protocols will follow US EPA and/or state guidelines.

4. Data Reported

Survival by 90% or more of the organisms in a control for acute toxicity tests is considered acceptable and the test deemed to be valid. More than 10% mortality of the control organisms is considered excessive mortality and results in the test being considered invalid.

Endpoints for static acute tests are: death, immotility, and loss of equilibrium. Data for each species of test organism are expressed as the percent of organisms in each test solution that exhibit any combination of endpoints. The point at which a sample is considered acutely toxic is generally permit and state specific. If the sample is found to be acutely toxic, a definitive test is usually then required.

E. Rangefinding Bioassay

1. Description

A rangefinding bioassay is a 24 hour exposure of the test organism to three to five widely spaced concentrations of effluent and a control. The control consists of the same species and same number and size of test organisms placed in the same control water as will be used for conducting the test. A rangefinding bioassay is used to determine the range of effluent concentrations to be used in a definitive

test. Rangefinding bioassays are used prior to a flow through bioassay due to lack of flexibility in changing the concentrations after the test is underway, or if no recent toxicity information on the discharge is available.

Effluent is collected from the same location as for the screening bioassays.

2. Test Organisms

The test organism will be the same species, age, and size as the organism used in the definitive test.

The organisms are exposed to the solutions for 24 hours. At the end of the exposure period, an observation is made of the following: the number of organisms dead, immotile, exhibiting loss of equilibrium, or that are unaffected.

3. Data Reported

Data for each species of test organism are expressed as the percent of organisms in the effluent or control that exhibit any combination of end points. The data is then used to determine the effluent concentrations in the definitive test.

F. Acute Definitive Bioassay

1. Description

A definitive bioassay consists of exposures of 48 hours for invertebrates or 48 to 96 hours for vertebrates to a series of five effluent concentrations and a control. The effluent concentrations are based on a dilution factor and approximate a geometric series, such as 100%, 50%, 25%, 12.5%, and 6.25%. A definitive bioassay is used to determine the effluent concentration that is lethal to 50 percent of the organisms within the prescribed period of time. Acute tests can either be static renewal, with replacement of effluent every 24 hours, or static nonrenewal, where the original solution is used for the duration of the test.

2. Test Organisms

The type of test organism, age, and size will be specified in the NPDES permit.

Data recorded for each organism are: life or death, motility or immotility, and exhibition of normal equilibrium or loss of equilibrium. All dead individuals are removed from the test. Data for the controls and each effluent are tabulated at the conclusion of each test.

3. Data Reported

Survival by 90% or more of the organisms in the controls for acute toxicity tests is considered acceptable and the test is considered valid. More than 10% mortality is considered excessive mortality, and results in the test being considered invalid. An invalid test must be repeated.

Data for each species of test organism are expressed as the percent of organisms adversely affected in each test dilution. This is referred to as the median lethal concentration (LC_{50}), and median effective concentrations (EC_{50}), for cumulative 24-hour exposure periods. An LC_{50} is the statistically, or graphically derived estimate of the effluent concentration that will cause death for 50% of the exposed organisms in a stated interval of time. An EC_{50} is obtained in the same manner as an LC_{50}, however, it includes all organisms recorded as dead, immotile, or with loss of equilibrium.

The statistical estimates of the LC_{50} and EC_{50} values and 95% confidence limits are calculated using computer software designed by the United States Environmental Protection Agency (US EPA). The values are calculated by use of the moving average-angle, binomial probability and probit methods. A graphical interpolation is also used to estimate the LC_{50} and EC_{50}. Confidence intervals are not obtained when the graphical interpolation is used.

G. Chronic Effluent Toxicity Tests (seven-day test)

1. Description

A seven-day chronic toxicity test consists of a seven-day exposure of the test organism to a control and a series of five effluent concentrations. Chronic bioassay tests are used to estimate the safe or "no effect" concentration of effluents (NOEC) which will permit normal propagation of fish and other aquatic life in the receiving waters.

2. Ceriodaphnia dubia

All C. dubia brood stock females are reared individually for one to two weeks prior to the start of a test. The total number of young per female must be 15 or greater in seven days for their offspring to be used in the test. To qualify as brood stock the mortality among all individually reared females must be less than 10%.

All C. dubia neonates that are used to begin the seven-day test must be less than 24 hours old and must have been produced within an eight hour time period. Ten 30-ml plastic beakers each containing 15 ml of solution and one organism are used for each control, and each effluent concentration. The cups are rinsed with

distilled water before use. Each organism is transferred to fresh solution daily using an eye dropper.

Each C. dubia is fed 0.1 ml of unicellular green alga (Selenastrum capricornutum) concentrate and 0.1 ml of yeast/CEROPHYLL/trout chow (YCT) suspension. The food is dispensed in the test chamber with a micropipette immediately after transfer of the test organism to fresh solution. The stock food containers are gently agitated prior to each use.

A Nikon dissecting microscope and a light box facilitates handling the original female, counting and removing neonates produced. Neonates are counted and discarded after the adult has been transferred.

Data recorded for each original individual at each observation time are life or death, motility or immotility, exhibition of normal equilibrium or loss of equilibrium, and the number of young produced if reproduction has occurred. All containers with dead individuals are removed from the test. Data for each effluent concentration and controls are tabulated at the conclusion of the test.

Survival data is tabulated at the end of the test for each solution and the controls. This data is used to calculate the acute LC_{50}'s and EC_{50}'s and to determine the chronic NOEC (no observed effect concentration) for survival. The number of young produced in each effluent concentration and control are tabulated at the conclusion of the test. The mean number of young produced are calculated for each effluent concentration and control, and are used to determine the NOEC for reproduction.

3. Fathead Minnow (Pimephales promelas)

Larval fathead minnows used in testing are obtained from a commercial laboratory. The minnows are hatched the morning prior to test initiation at the commercial laboratory, placed in a cubitainer containing culture water, and shipped overnight to RMC. Minnows are acclimated to laboratory environmental conditions for several hours before test initiation. The larvae are approximately 24 hours old at test initiation.

Four 7.6 cm x 16.0 cm x 8.0 cm glass aquaria (or 500 ml plastic beakers) each containing 250 ml of test solution and ten larvae are used for each effluent concentration and control.

Pimephales promelas larvae are fed freshly hatched artemia nauplii twice daily. The nauplii suspension is rinsed with DMW and 0.1 ml is micropipetted to the test chambers, twice daily, a minimum of six hours apart.

Data recorded for each container are the number of larvae alive or dead, motile or immotile, and exhibiting normal equilibrium or loss of equilibrium. Dead organisms are removed and discarded when found.

All surviving larvae within each replicate are counted, rinsed with deionized water, and transferred as a group to pre-weighed pans at the end of seven days. Minnows are dried in an oven at 104 C for at least eight hours and no more than 24 hours, and immediately transferred to a dessicator. Each pan containing minnows is weighed to the nearest 0.1 mg on an American Scientific Products Electronic Analytical Balance and the total dry minnow weight divided by the number of minnows to obtain the average minnow weight per replicate exposure. The means are used to calculate the NOEC for growth.

Survival data is also tabulated at the end of the test for each solution and the controls. This data is used to calculate the acute LC_{50}'s and EC_{50}'s and the chronic NOEC for survival.

4. Test Acceptability Criteria

Survival by 80% or more of the organisms in the controls for chronic toxicity tests is considered acceptable and the test is considered valid. More than 20% mortality of the organisms in a control is considered excessive mortality and results in the test being invalid.

A fathead minnow chronic growth test is considered acceptable when the average dry weight of the control organisms equals or exceeds 0.25 mg. C. dubia chronic tests are considered acceptable when control organisms have produced an average of 15 young or greater per surviving female and at least 60% have produced three broods.

5. Data Reported

Acute test endpoints are death, immotility, and loss of equilibrium. Data for each species of test organism are expressed as the percent of organisms adversely affected in each test solution and the median lethal concentration (LC_{50}) or median effective concentration (EC_{50}) for the effluent test for cumulative 24-hour exposure periods. An LC_{50} is the statistically, or graphically, derived estimate of the effluent concentration that will cause the death of 50 percent of the exposed organisms in the stated period of time. An EC_{50} is obtained in the same manner as an LC_{50}, however, it includes all organisms recorded as dead, immotile, or with loss of equilibrium.

C. dubia chronic test results are analyzed to determine a statistically significant reduction in the numbers of young produced in an effluent concentration as compared to the controls, and to determine a statistically significant reduction

in survival in an effluent concentration as compared to the controls. Data are expressed as the NOEC and EC_{50} for 50 percent reduction. Pimephales promelas larvae chronic tests are analyzed to determine a statistically significant reduction in larvae weight for larvae in an effluent concentration as compared to the controls, and to determine a statistically significant reduction in survival in an effluent concentration as compared to the controls. Data are expressed as the NOEC and EC_{50} for 50 percent reduction.

H. Sediment Toxicity Tests (ten-day test)

1. Description

Protocols for conducting sediment toxicity tests are based on ASTM guidance documents and US EPA methods described in a regional document. A US EPA standardized protocol is anticipated in the fall of 1993.

2. Solid Phase Tests

Short term (ten day) tests are performed with the freshwater species Hyalella azteca (amphipod) and Chironomus tentans (midge) for survival and survival/growth, respectively. Other species can be utilized by consulting the appropriate guidance document.

3. Hyalella azteca

Seven to ten day old organisms are used to begin a ten day test. Eight 300 ml beakers each containing 100 ml of sediment and 150 ml of overlying water are prepared. Ten organisms are placed in each beaker for a total of eighty organisms per sediment. The overlying water is renewed daily.

Each test chamber is fed 1.5 ml YCT per day and the test temperature is 23 ± 2°C.

Survival data is recorded at the end of the test for each test sediment and the controls. This data is used to calculate the acute LC_{50}s and EC_{50}s.

4. Chironomus tentans

Second instar, light to twelve day old organisms, are used to begin a ten day test. Eight 300 ml beakers each containing 100 ml of sediment and 150 ml of overlying water are prepared. Ten organisms are placed in each beaker for a total of eighty organisms per sediment. The overlying water is renewed daily.

Each test chamber is fed Tetrafin® at 1 ml/day/beaker.

Survival data is recorded at the end of the test for each test sediment and the controls. This data is used to calculate the acute LC_{50}s and EC_{50}s.

I. Statistical Methods

1. Description

At the completion of a toxicity test, all raw data is entered into RMC's Bioassay Database. Summary sheets of the raw data are sent to RMC's Information Analyst for data analysis. The flowchart for RMC's data analysis is provided in Figure 1.

2. Methods

The LC_{50} and EC_{50} values and confidence limits are statistically calculated by use of the moving average, binomial probability, probit and Spearman-Karber methods. Straight-line graphical interpolation is also used to estimate LC_{50} and EC_{50} values. Confidence limits may not be obtained when the graphical procedure is used. The NOEC, LOEC, and Chv for C. dubia survival and reproduction, and Pimephales promelas survival and growth (weight) are determined by statistical analysis following U.S. EPA flowcharts (EPA/600/4-89/001). Chronic EC_{50} values for C. dubia reproduction and fathead minnow growth are determined by inverse linear regression. The flowcharts for statistical analysis of the chronic data are provided in Figures 2 through 5.

J. Quality Assurance/Quality Control

1. Standard Reference Toxicants

A standard reference toxicant (SRT) is used to ensure the validity of the results obtained in a bioassay. The analysis of SRT data, in conjunction with bioassay data, minimizes the chance of accepting bioassay results which have been biased through the use of diseased or stressed organisms. RMC's laboratory uses reagent grade potassium chloride as the SRT. Acute and chronic SRTs are performed on a routine basis. Chronic SRTs are performed monthly on fathead minnow and C. dubia. The results from the SRTs are recorded on a control chart and are available upon request.

2. Employee Training

All new laboratory technicians are formally trained in all bioassay techniques and the results of this training documented. See Appendix A for example training program.

3. Laboratory Operations

Internal laboratory audits are conducted at the discretion of RMC's QA Manager. Results of internal audits are available upon request. Audits are conducted on a non-routine basis by state regulatory agencies. Results of these audits are available upon request.

4. Record Keeping

Toxicity testing follows a standard process from sample collection to data processing. See Appendix B for example bioassy tracking forms, and Appendix C for logbook forms, and data sheets.

5. Support Procedures

Detailed procedures for conducting the tests outlined in this document are contained in the following procedures in RMC's Quality Assurance Program. The most current procedure revisions are to be used.

EB 1	Water Quality Sampling
EB 4	D.O. Meter
EB 7	Temperature
EB 8	pH Determination
EB 11	Specific Conductance
EB 16	Chronic Toxicity Testing
EB 17	Alkalinity - Hach
EB 18	Hardness - Hach
EB 19	Low Range Total Residual Chlorine
EB 45	Acute Bioassay Test - New Jersey
EF 44	Acute Toxicity Testing
EG 3	Compositing Environmental Samples
EG 4	Hazardous Waste Safety
GES 1	Corporate Health and Safety Plan
Q 5	Training and Certification
QAM	QA Manual

6. Support Instructions/Equipment Lists

Support instructions for performing routine tasks in the laboratory are contained in Appendix D. A list of bioassay equipment is contained in Appendix E.

7. Equipment Calibration

Prior to conducting water quality analyses, all meters must be calibrated. All meters are located on a moveable cart in the laboratory. Instructions on frequency of calibration are located on each meter, and a log book for recording calibration is located on the cart.

8. Water Analysis

Every time deionized water is picked up from RMC's Environmental Chemistry Laboratory, RMC Analaytics, the pH and conductivity of the water must be recorded. All other parameters are kept on record at RMC Analytics.

Monthly water analysis must be performed on both the laboratory tap water and laboratory deionized water. A list of required analysis is located in Appendix F.

A biannual water analysis must be completed on the laboratory tap water as part of the requirements for New Jersey Certification. A complete list of these parameters is located in Appendix F.

9. Biomonitoring Report Forms

When testing is completed for compliance purposes, a Biomonitoring Report Form should be completed and included in the formal test report. Acute and chronic Biomonitoring Report Forms are located in Appendices G and H, respectively.

10. Sediment Testing

Data sheets for sediment bioassays are located in Appendix I.

LIST OF TERMS USED IN BIOMONITORING

Acute - Involving a stimulus severe enough to rapidly induce a response; in aquatic toxicity tests, a response measuring lethality observed in 96 hours or less is typically considered acute.

Acute-Chronic Ratio (ACR) - The ratio of the acute toxicity of an effluent or a toxicant to its chronic toxicity. It is used as a factor for estimating chronic toxicity on the basis of acute toxicity data, or for estimating acute toxicity on the basis of chronic toxicity data.

Ambient Toxicity - Toxicity manifested by a sample collected from a waterbody.

Bioaccumulation - The process by which a compound is taken up by an aquatic organism, both from water and through food.

Bioaccumulation Factor (BAF) - Measure of a chemical's tendency to bioaccumulate.

Bioassay - A test used to evaluate the relative potency or toxicity of a chemical or aqueous solution by comparing its effect on a living organism with the effect of a standard preparation on the same type of organism.

Bioavailability - A measure of the physicochemical access that a toxicant has to the biological processes of an organism. The less the bioavailability of a toxicant, the less its toxic effect on an organism.

Bioconcentration - The process by which a compound is absorbed from water through gills or epithelial tissues and is concentrated in the body.

Bioconcentration factor (BCF) - Measure of a chemical's tendency to concentrate in tissues of aquatic organisms.

Biocriteria - Numerical measures or narrative descriptions of the biological integrity of unimpaired natural systems.

Ceriodaphnia dubia - A small cladoceran crustacean. It is found throughout most of North America and attains a maximum size of approximately 1 mm. This organism has been adopted for aquatic toxicity testing because of its small size, ease of culture under laboratory conditions, stability of genetic strains, and sensitivity to toxic substances. It is used in both acute (48-hr) and chronic (7-day) static toxicity testing with endpoints of survival and reproduction.

Chronic - Involving a stimulus that lingers or continues for a relatively long period of time, often one-tenth of the life span or more. Chronic should be considered a relative term depending on the life span of an organism. The measurement of a chronic effect can be reduced growth, reduced reproduction, etc., in addition to lethality.

Chronic Toxicity Endpoints (CTEs) - Include NOEC, LOEC, EC, and IC; based on observations of reduced reproduction, growth, etc., in addition to lethality.

Chronic Value (ChV) - A numeric value representing the geometric mean of the numeric values of concentrations analyzed as the No Observed Effect Concentration (NOEC) and the Lowest Observed Effect Concentration (LOEC) by chronic testing. The chronic value is an estimate of the toxicant concentration that will be the actual no effect concentration based on the chronic effect tested. ChV = Antilog ((Log 10 LOEC + Log 10 NOEC) / 2).

Coefficient of Variation (CV) - A measure of the amount of variation within a population, calculated as the standard deviation(s) expressed as a percentage of the mean (x); CV = S x 100 / x.

Composite - A sample or method of sampling used to obtain data on a substance which may vary over time or space. For example, a time or temporal composite of a stream would be one collected at intervals of time at the same location. This is frequently accomplished by the use of automatic sampling devices.

Criteria Continuous Concentration (CCC) - The EPA national water quality criteria recommendation for the highest instream concentration of a toxicant or an effluent to which organisms can be exposed indefinitely without causing unacceptable effect.

Criteria Maximum Concentration (CMC) - The EPA national water quality criteria recommendation for the highest instream concentration of a toxicant or an effluent to which organisms can be exposed for a brief period of time without causing mortality.

Critical Life Stage - The period of time in an organism's lifespan in which it is the most susceptible to adverse effects caused by exposure to toxicants, usually during early development (egg, embryo, larvae). Chronic toxicity tests are often run on critical life stages to replace long duration, life cycle tests since the most toxic effect usually occurs during the critical life stage.

Dilution (water) - The water used in aquatic toxicity tests to dilute the wastewater to various concentrations (expressed as a percent). This can be upstream receiving water, synthetic water, or any other department approved source.

Effective Concentration (EC) - A point estimate of the toxicant concentration that would cause an observable adverse effect such as death, immobilization, or serious incapacitation) in a given percentage of the test organisms. Calculated from curve fitting.

Effluent Biomonitoring - The measurement of the biological effects of effluents (such as toxicity, biostimulation, and bioaccumulation).

Fathead Minnow (Pimephales promelas) - A small fish which occurs throughout much of North America. It attains a maximum size of approximately 100 mm and is raised commercially as a bait fish. The fathead minnow has been raised for numerous generations in a number of laboratory cultures for use in toxicity testing. The species can produce eggs year-round in the laboratory environment under suitable conditions to provide test organisms as needed.

Flow-through - The flow through toxicity test utilizes a mechanical dilutor which either continuously or occasionally replaces the effluent/toxicant and dilution water in the test chambers with fresh solutions throughout the test in an attempt to sample the variability of effluent toxicity throughout the test period.

Inhibition Concentration (IC) - A point estimate of the toxicant concentration that would cause a given percent reduction (e.g., IC25) in a non-lethal biological measurement of the test organisms, such as reproduction or growth. Determined using curve fitting with an assumption of a continuous dose-response relationship.

Instream Waste Concentration (IWC) - The exposure concentration of a toxicant or the parameter toxicity in the receiving water after mixing. (Also known as the Receiving Water Concentration or RWC).

Lethal Concentration (LC) - The toxicant concentration killing a certain percentage (e.g., LC50) of exposed organisms at a specific time of observation.

Lowest Observed Effect Concentration (LOEC) - The lowest concentration of an effluent or toxicant that results in observable adverse affects in the aquatic test population.

Neonate - Roughly translated into newborn. In reference to cladocerans, the neonate refers to the life stage in the first and early second instar, generally the first 24 hours of its life.

No Observed Effect Concentration (NOEC) - The highest concentration of an effluent or a toxicant at which no adverse effects are observed on the aquatic test organisms. Determined using hypothesis testing with the assumption of a noncontinuous threshold model of the dose-response relationship.

Receiving Water Concentration (RWC) - The exposure concentration of a toxicant or the toxicity parameter in the receiving water after mixing. (Also termed "Instream Waste Concentration," IWC.)

Relative Toxicity - The toxicity of the effluent when it is mixed with the receiving water, or a dilution water of similar composition for toxicity testing.

Q7-10 - The measurement of a stream's lowest average daily flow over a 7-day period during a ten year span, generally stated as flow in cubic meters per second.

Static - Refers to an aquatic toxicity test in which toxicant/effluent concentrations are set up at the beginning of the test and not changed or replaced for the rest of the test. This is generally a short term test as compared to a flow-through or replacement test because of potential degradation of the toxicant/effluent. These tests can be either renewal or non-renewal in nature.

Sublethal - Involving stimulus below the level that causes death.

Threshold Effects - Acute, subacute, or chronic human health effects.

Toxicity Identification Evaluation (TIE) - Set of procedures to identify the specific chemical(s) responsible for effluent toxicity.

Toxicity Reduction Evaluation (TRE) - A site-specific study conducted in a step-wise process designed to identify the causative agents of effluent toxicity, isolate the sources of toxicity, evaluate the effectiveness of toxicity control options, and then confirm the reduction in effluent toxicity.

Toxicity Test - A procedure to determine the toxicity of a chemical or an effluent using living organisms. A toxicity test measures the degree of response of exposed test organisms to a known concentration of a specific chemical or effluent.

Toxic Units (TUs) - 100 divided by the toxicity endpoint measured; expressed as acute toxicity units (TUa) or chronic toxicity units (TUc).

Toxic Unit Acute (TUa) - The reciprocal of the effluent concentration that causes an acute effect by the end of the acute exposure period (e.g., 100/LC50).

Toxic Unit Chronic (TUc) - The reciprocal of the effluent concentration that causes no observable effect on the test organisms by the end of the chronic exposure period (e.g., 100/NOEC).

Water Quality Criteria - Scientifically derived ambient concentration developed by EPA or States for various pollutants of concern. Criteria developed by EPA are recommended levels which should not be exceeded in a body of water in order to protect aquatic life and human health; all states have both chemical-specific numeric criteria for individual toxic pollutants and narrative "free from toxics in toxic amounts" criteria; criteria are subjected to the regulatory process or regional, state, and public comment.

Water Quality Standard - A law or regulation that consists of the beneficial designated use or uses of a waterbody, the water quality criteria that are necessary to protect the use or uses of that particular waterbody, and an antidegration statement.

Whole Effluent Toxicity - The aggregate toxic effect of an effluent measured directly with a toxicity test.

LIST OF REFERENCE MANUALS RECOMMENDED FOR USE IN BIOMONITORING[1]

1. Peltier, W. and C.I. Weber. 1985. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Third Edition. Office of Research and Development, Cincinnati, OH. EPA/600/4-85/013. (New manual due out in 1991).

2. Weber, C.I., et al. (ed.). 1989. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Second Edition, Office of Research and Development, Cincinnati, OH. EPA/600/4-89/001.

3. Weber, C.I., et al. (ed.). 1988. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Office of Research and Development, Cincinnati, OH. EPA/600/4-87/028.

4. U.S. Environmental Protection Agency. 1991. Technical Support Document for Water Quality-Based Toxics Control. Office of Water, Washington, DC. EPA-505/2-90-001.

5. U.S. Environmental Protection Agency. 1988. Methods for Aquatic Toxicity Identification Evaluations. Phase I. Toxicity Characterization Procedures. ERL-Duluth. EPA/600/3-88/034.

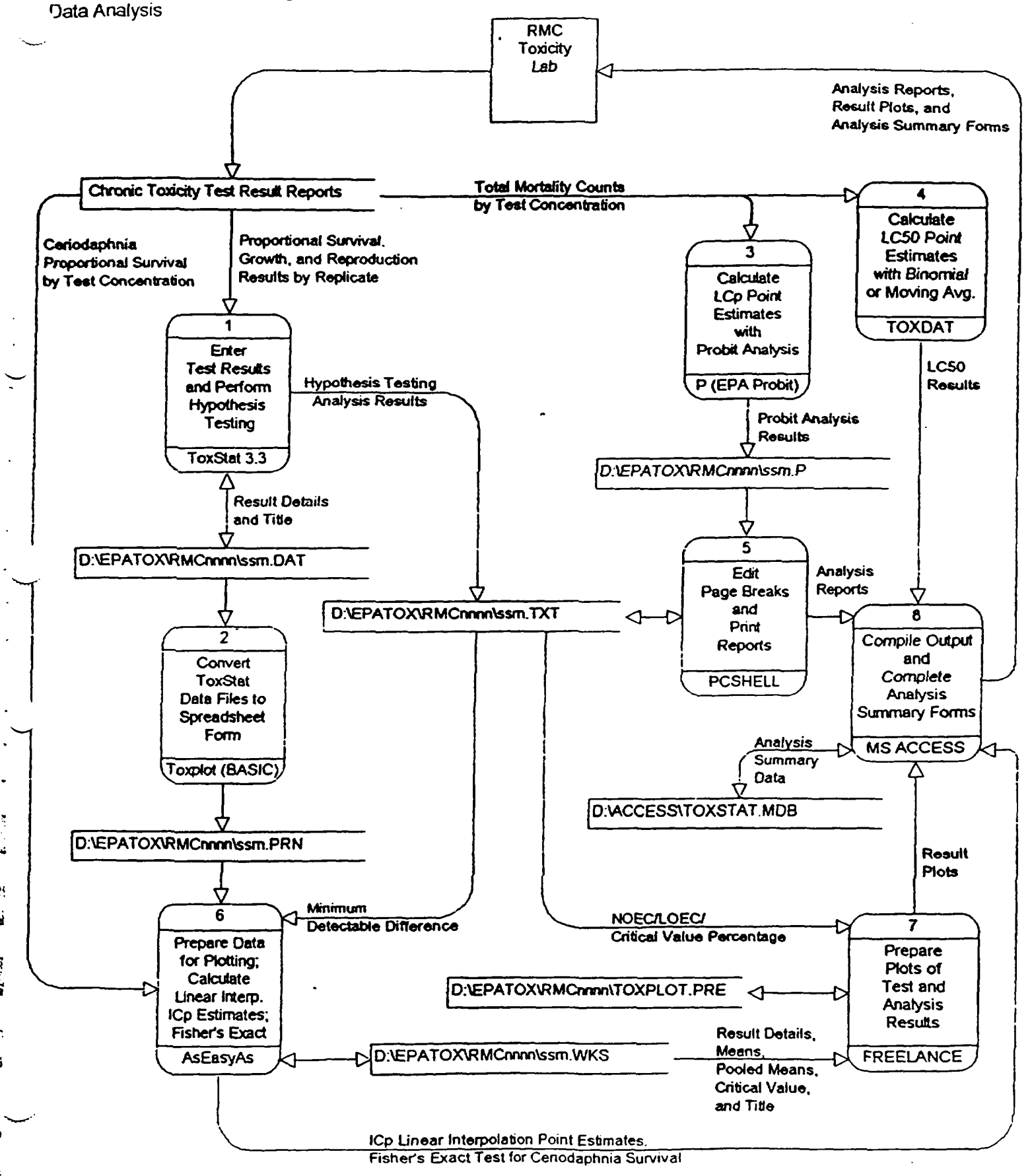
6. U.S. Environmental Protection Agency. 1989. Methods for Aquatic Toxicity Identification Evaluations. Phase II. Toxicity Identification Procedures. ERL-Duluth. EPA/600/3-88/035.

7. U.S. Environmental Protection Agency. 1989. Methods for Aquatic Toxicity Identification Evaluations. Phase III. Toxicity Confirmation Procedures. ERL-Duluth. EPA/600/3-88/036.

8. U.S. Environmental Protection Agency. 1989. Toxicity Reduction Evaluation Protocol for Municipal Wastewater Treatment Plants. Risk Reduction Engineering Lab, Cincinnati, OH. EPA/600/2-88/062.

[1]Pennsylvania Department of Environmental Resources. 1991. From Protocol For Conducting Whole Effluent Toxicity Tests. M.D. Bilger and C.M. Hersh, July 1991.

Figure 1.
Data Flow Diagram for
RMC Chronic Toxicity Testing
Data Analysis



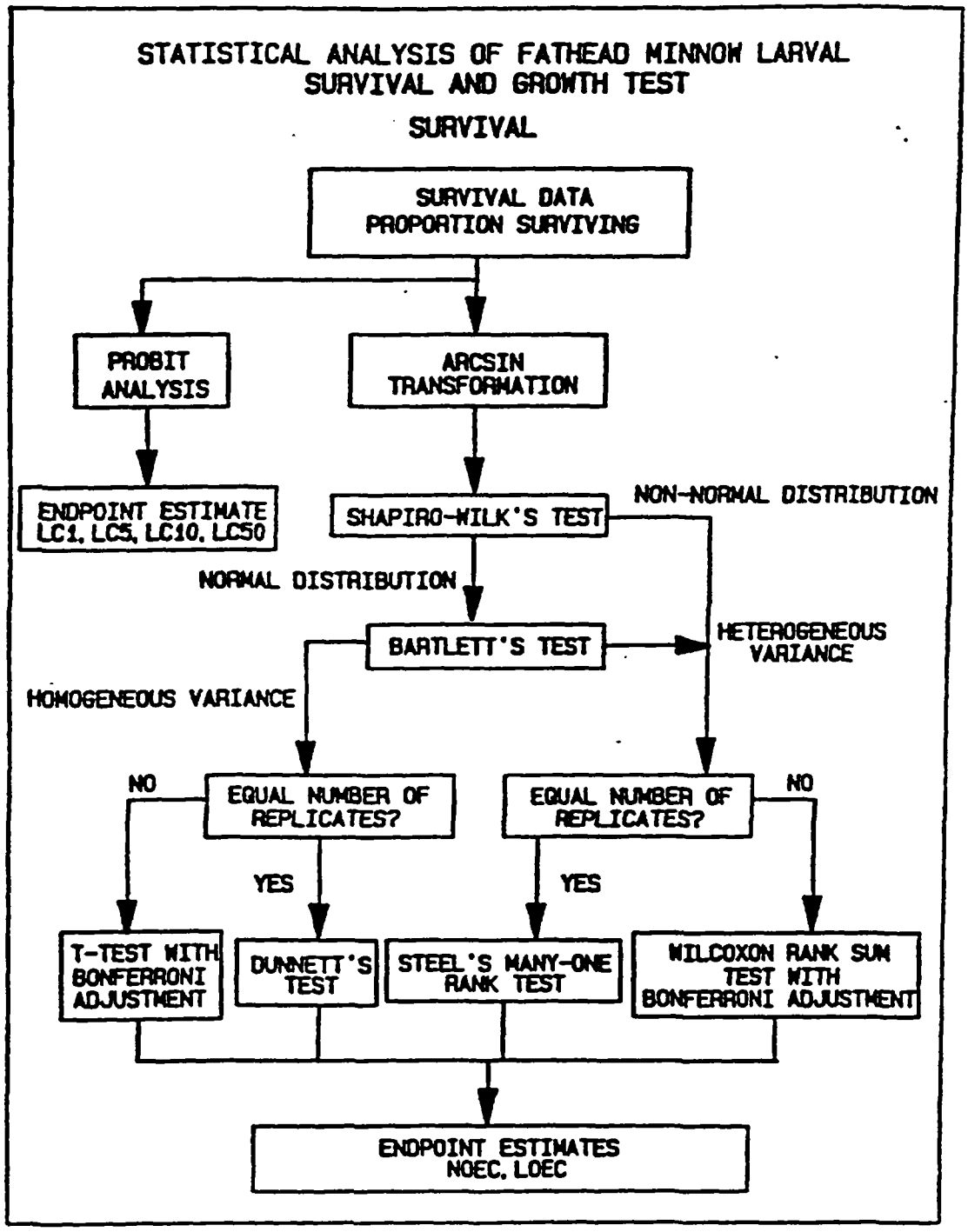


Figure 2. Flow chart for statistical analysis of fathead minnow larval survival data.

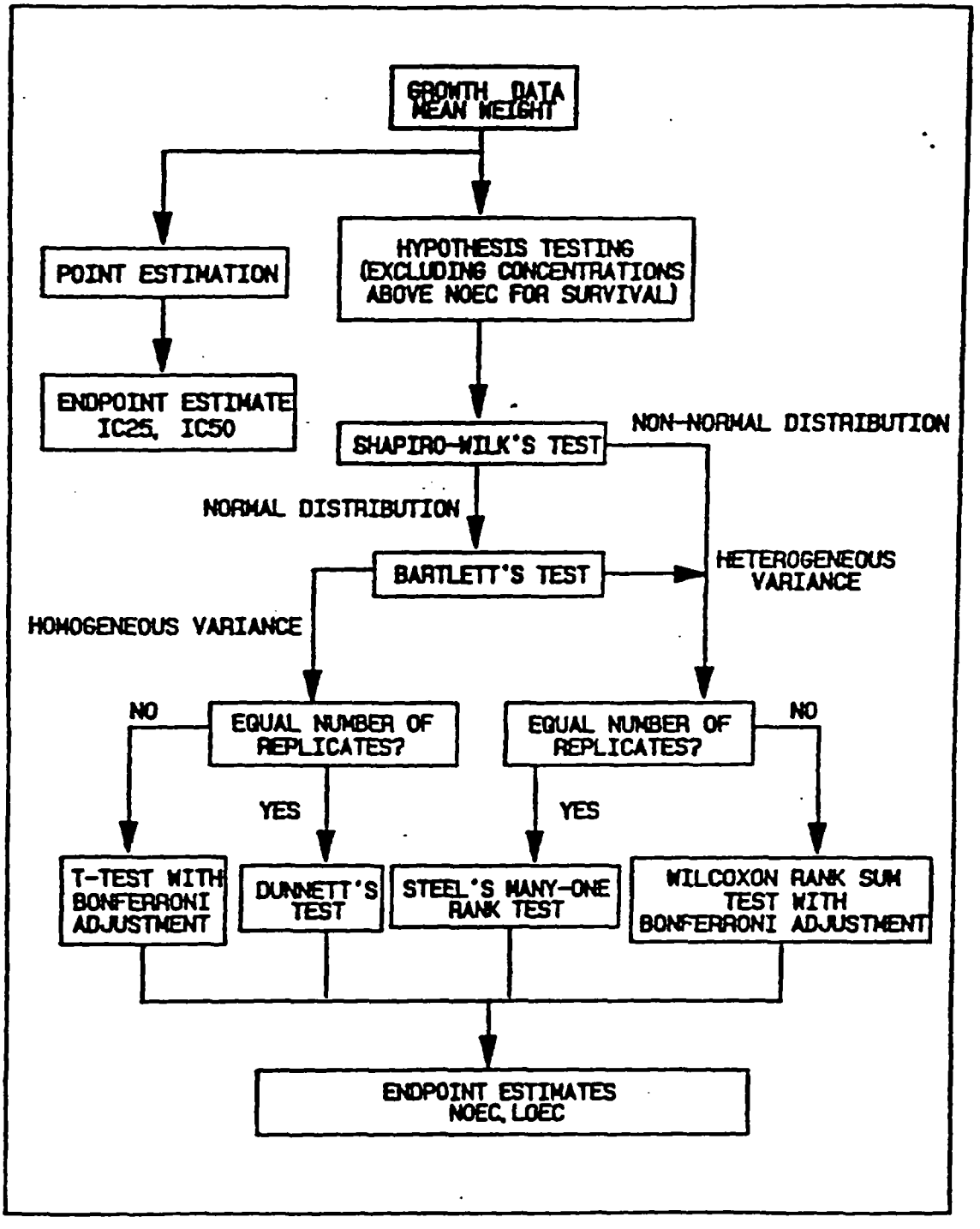


Figure 3. Flow chart for statistical analysis of fathead minnow larval growth data.

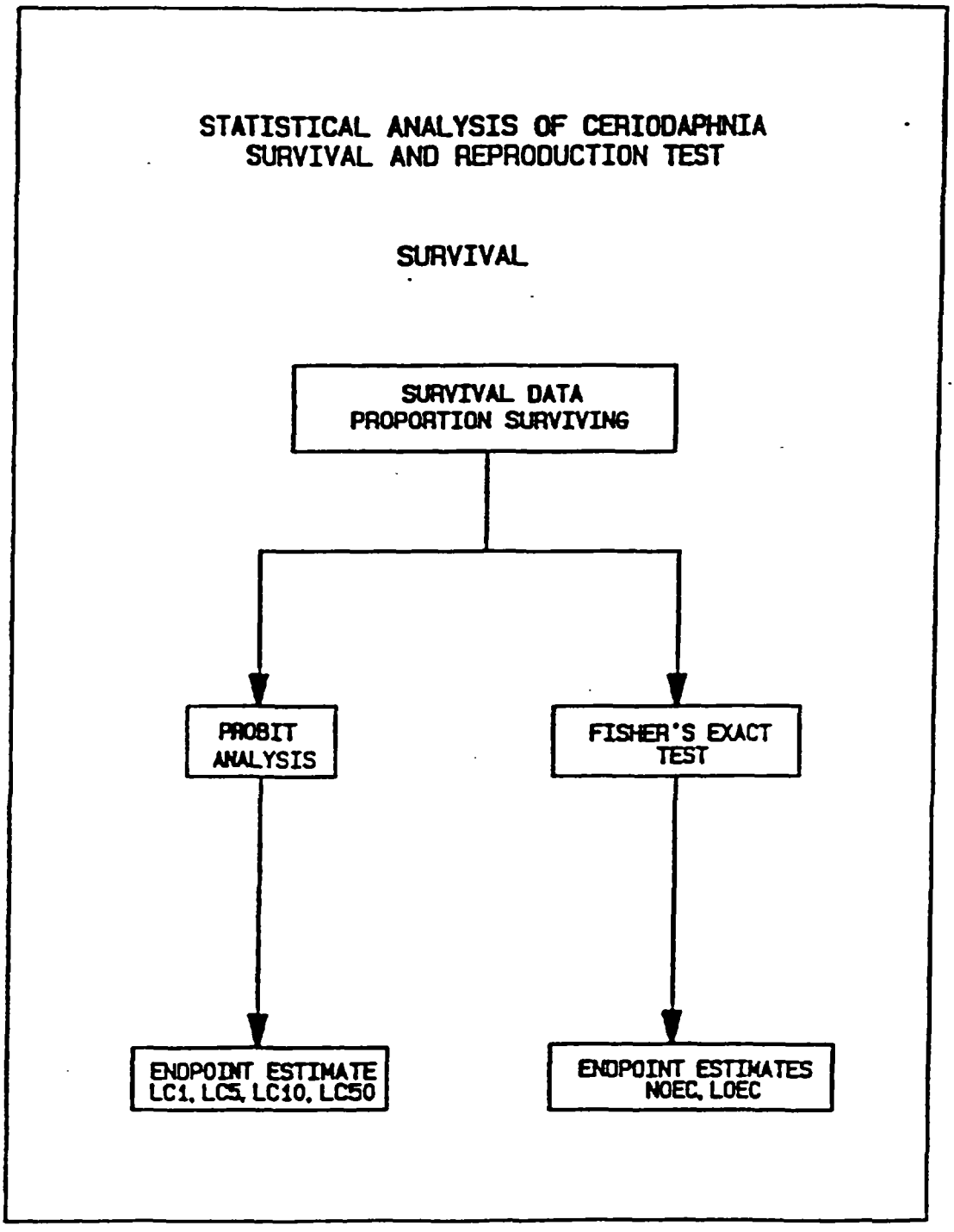


Figure 4. Flow chart for statistical analysis of *Ceriodaphnia* survival data.

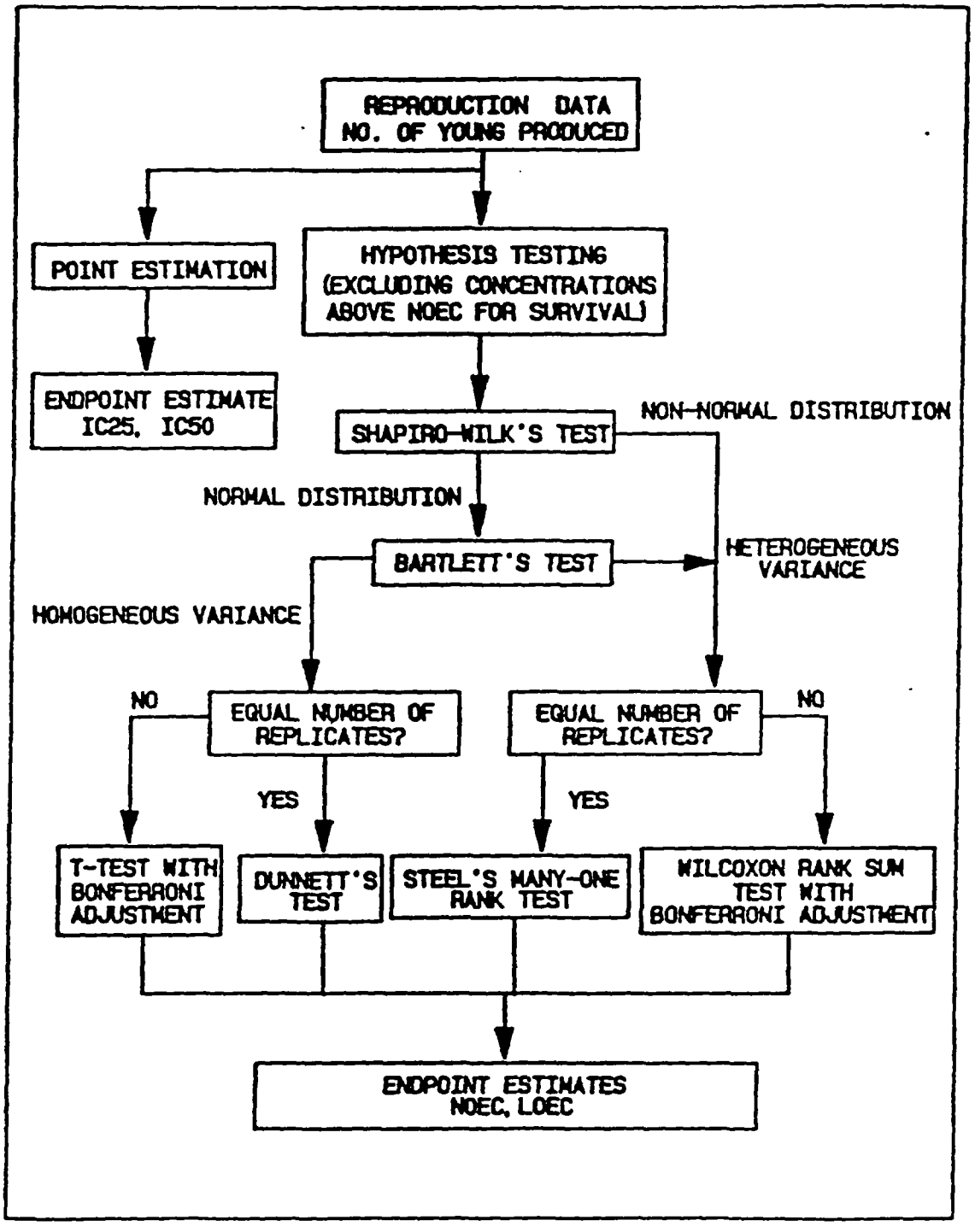


Figure 5. Flow chart for statistical analysis of Ceriodaphnia reproduction data.

APPENDIX A

On the Job Training Program

RMC Procedure Nos. EF44 and EB45

Determination of Acute Toxicity to Aquatic Organisms
and Acute Bioassay Testing of Effluents under
New Jersey Annotated Code (NJAC) Chapter 7:18-6

Training Program Item	Trainee Initials	Date(s)	Trainer Initials
1. Bioassay Procedure Manual reviewed	_____	_____	_____
2. Read and understand Procedure No. EF44	_____	_____	_____
3. Read and understand Procedure No. EB45	_____	_____	_____
4. EPA manual Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms (most recent edition) reviewed and understood	_____	_____	_____
5. Identify and become familiar with US EPA and state regulatory agency methodology	_____	_____	_____
6. Discuss Quality Control requirements	_____	_____	_____
7. Become familiar with laboratory apparatus	_____	_____	_____
8. Become familiar with test organism handling	_____	_____	_____
9. Become familiar with test organism culture	_____	_____	_____
10. Conduct Quality Control Task (see attached)	_____	_____	_____
11. Discuss problem solving	_____	_____	_____
12. Process test data (LC_{50} calculation)	_____	_____	_____
13. Prepare test report	_____	_____	_____

On the Job Training Program

Quality Control Task for

RMC Procedure Nos. EF44 and EB45

Acute Reference Toxicity Test

Training Program Item	Trainee Initials	Date(s)	Trainer Initials
1. Obtain two species of test organisms of proper age	_____	_____	_____
2. Mix stock solutions	_____	_____	_____
3. Prepare test concentrations	_____	_____	_____
4. Perform appropriate physical/chemical measurements	_____	_____	_____
5. Perform titrations	_____	_____	_____
6. Place test organisms in test chambers using proper handling techniques	_____	_____	_____
7. Record test organism mortality at proper intervals	_____	_____	_____
8. Complete data sheets	_____	_____	_____
9. Process test data (LC_{50} calculation)	_____	_____	_____
10. *All supporting procedures understood* EB4, EB7, EB9, EB11, EB17, EB18, EB19	_____	_____	_____

On the Job Training Program

RMC Procedure No. EB16

Determination of Chronic Toxicity to Aquatic Organisms

Training Program Item	Trainee Initials	Date(s)	Trainer Initials
1. Bioassay Procedure Manual reviewed	_____	_____	_____
2. Read and understand Procedure No. EB16	_____	_____	_____
3. EPA manual Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms (most recent edition) reviewed and understood	_____	_____	_____
4. US EPA videotapes detailing chronic test methods for fathead minnows and C. dubia viewed and understood	_____	_____	_____
5. Identify and become familiar with US EPA and state regulatory agency methodology	_____	_____	_____
6. Discuss Quality Control requirements and test acceptability criteria	_____	_____	_____
7. Become familiar with laboratory apparatus	_____	_____	_____
8. Become familiar with test organism handling	_____	_____	_____
9. Become familiar with test organism culture	_____	_____	_____
10. Conduct Quality Control Task (see attached)	_____	_____	_____
11. Discuss problem solving	_____	_____	_____
12. Process test data (data entry)	_____	_____	_____
13. Prepare test report	_____	_____	_____

On the Job Training Program

Quality Control Task for RMC Procedure No. EB16

Chronic Reference Toxicity Test

Training Program Item	Trainee Initials	Date(s)	Trainer Initials
1. Obtain two species of test organisms of proper age	_____	_____	_____
2. Mix stock solutions	_____	_____	_____
3. Prepare test concentrations	_____	_____	_____
4. Perform appropriate physical/chemical measurements at proper intervals	_____	_____	_____
5. Perform titrations	_____	_____	_____
6. Place test organisms in test chambers using proper handling techniques	_____	_____	_____
7. Record test organism mortality at proper intervals	_____	_____	_____
8. Prepare test organisms' food and feed proper amounts/types (brine shrimp, YCT)	_____	_____	_____
9. Prepare two types of dilution water (DMW, DMW + Se/B_{12})	_____	_____	_____
10. Perform renewals	_____	_____	_____
11. Meet test acceptability criteria	_____	_____	_____
12. Complete data sheets	_____	_____	_____
13. Process test data (data entry)	_____	_____	_____
14. All supporting procedures understood EB4, EB7, EB9, EB11, EB17, EB18, EB19	_____	_____	_____

APPENDIX B

(REV. 7/93)

AQUATIC TOXICOLOGY LABORATORY

RECEIPT OF SAMPLES FOR TESTING

1. All samples must be accompanied by Chain-of-Custody forms.

2. Upon receipt of samples, log sample into Sample Log Book (on meter cart) with the following information:

   - Job Number/Client
   - Sample Date/Type
   - Date of Receipt/Time
   - Temperature
   - Dissolved Oxygen
   - Salinity
   - Initials

3. After the sample has been logged in, obtain a Sample Tracking form. Complete the form as appropriate and place in the job operating file.

4. When you are finished using the sample, it must be stored in the walk-in freezer. Record appropriate information on the Sample Tracking Form.

5. The cooler temperature must be recorded daily in the Sample Log Book.

6. At test termination, record disposal of the sample(s) on the Sample Tracking form. The original Sample Tracking form should be placed in the main job file, and a copy in the operating file.

Aquatic Toxicology Laboratory

Sample Tracking

Client:_____ Test Date:_____

Job Number/Task:_____ Initials:_____

Test Type:_____

	Sample No. 1	2	3	4	5
Date sample received	_____	_____	_____	_____	_____
Logged on COC?	_____	_____	_____	_____	_____
Logged in sample receipt book?	_____	_____	_____	_____	_____
TRC determined?	_____	_____	_____	_____	_____
Sample type	_____	_____	_____	_____	_____
Number of samples/volume	_____	_____	_____	_____	_____
Sample I.D.	_____	_____	_____	_____	_____
Sample Expiration	_____	_____	_____	_____	_____
Initial storage date	_____	_____	_____	_____	_____
Cooler temp/thermometer I.D.	_____	_____	_____	_____	_____
Total storage time	_____	_____	_____	_____	_____
Method of Disposal	_____	_____	_____	_____	_____

Comments: _____

_____

_____

_____

_____

Analytics

Analytical Request Form

Client:	If Reporting Address is Different Report Results to:
Client Address:	Address:
Client Contact:	Contact:
Phone ( ) -	Phone ( ) -
Authorized signature:	Report Results via: (Circle all that apply)
Purchase Order Number:	Verbal Hardcopy Fax ( ) -
Job Number:	Report Results Due:
Sample Date:	Lab Contact:

SALES ORDER NO:	**QA/QC Requirements:(circle one) Standard Tier CLP Other (Specify)**

Sample Identification	Matrix	Parameters	Special Instructions

**** RMC cannot guarantee turnaround time faster than 2 weeks without 24 hour prior notice.**
*****RMC cannot guarantee bottle deliveries for large orders without 24 hour prior notice, bottles to be sent UPS must have notice by 11 AM and bottles to be sent Federal Express must have notice by 2 PM .**

The following information is extremely important to assist in determining the final disposal of lab samples. Results of laboratory tests requisitioned by the client as well as the following information will help to categorize the waste. Please indicate in this section your preference for disposal of samples.

( ) Please return samples to the client _____ (person to contact).
( ) Samples are not known to be hazardous, please dispose of samples as non hazardous.
( ) Samples contain hazardous materials, please dispose of and invoice accordingly.
( ) The source from which the samples were taken is known to contain hazardous materials.

Proper disposal of lab samples is the responsibility of both the laboratory and the client.

COMPLETING THE ANALYTICAL REQUEST FORM

The item numbers below correspond to the blanks on the Analytical Request Form. The form should be filled out as completely as possible.

1. The Client Name that will appear on the Certificate of Analysis.
2. The address to which the Certificate of Analysis is to be mailed (unless right-hand side is filled out.)
3. Your VIP # (Obtained from your Client Services Representative).
4. The person who will be contacted if there are any questions about the samples.
5. Telephone number of 3. above.
6. Signature of person authorizing analyses.
7. **Purchase Order number. RMC billing procedures require that our clients provide us with a hard copy purchase order or written authorization to proceed prior to acceptance of analytical work. A signed Analytical Request Form will be used in lieu of a purchase order.**
8. Job Number or project name.
9. Person whom client contacted at RMC Analytics.
10. The number which is located on the Analytical Quotation and identifies the sales order.
11. Any type of QA/QC requirements that are needed for the project and/or samples.
12. The sample description that will appear on the Certificate of Analysis.
13. From the matrix codes provided on the Chain of Custody.
14. Detailed list of analyses to be run on each sample. A list may be attached.
15. Any additional special information about the sample.
16. How you would like the samples disposed.

*** PLEASE NOTE: The Chain of Custody and Analytical Request Form completed and submitted with your sample.

RMC Analytics

ANALYTICAL REQUEST FORM

1	Client: Acme Waste	3 VIP #: 15555	If Reporting Address is Different Report Results to:
2	Client Address: 1234 Waste Way		Address:
4	Client Contact: Joe Smoe		Contact
5	Phone (215) 112-1234		Phone ( )
6	Authorized signature: Pete Toutsuite		Report Results via: (Circle all that apply)
7	Purchase Order Number:		Verbal Hardcopy Fax ( )
8	Job Number:		Report Results Due:
	Sample Date:	9	Lab Contact: Heidi Krueger

10	SALES ORDER NO: 1111	11	QA/QC Requirements:(circle one) Standard Tier CLP Other (Specify)

Sample Identification	Matrix	Parameters	Special Instructions
12 Soil Pile 1	13 S	14 TPVOA, Cyanide, Sulfide, pH, TDS	15

** RMC cannot guarantee turnaround time faster than 2 weeks without 24 hour prior notice.
***RMC cannot guarantee bottle deliveries for large orders without 24 hour prior notice. bottles to be sent UPS must have notice by 11 AM and bottles to be sent Federal Express must have notice by 2 PM .

16 The following information is extremely important to assist in determining the final disposal of lab samples. Results of laboratory tests requisitioned by the client as well as the following information will help to categorize the waste. Please indicate in this section your preference for disposal of samples.

- ( ) Please return samples to the client _____ _____ (person to contact).
- ( ) Samples are not known to be hazardous, please dispose of samples as non hazardous.
- ( ) Samples contain hazardous materials, please dispose of and invoice accordingly.
- ( ) The source from which the samples were taken is known to contain hazardous materials.

Proper disposal of lab samples is the responsibility of both the laboratory and the client.

CHAIN OF CUSTODY

RMC Analytics
A Division of RMC Environmental Services, Inc.
88 Robinson Street
Pottstown, Pa. 19464
(215) 327-4850

Client: _____ VIP #: _____
Address: _____
Phone: _____
Samplers: _____
Job No.: _____

Lab Contact: _____
Sales Order No.: _____
P.O. Number: _____

Turnaround Time	Normal [ ]	Rush [ ]
Sample Disposal	Return to Client	[ ]
	Disposal by Lab	[ ]

RMC USE ONLY

RMC only Lab ID	Sample Identification	Matrix Code *	Sample Date	Sample Time	VOA	BNA	Pest/ PCB	Herb				Metals	Cn	NH3	Wet Chem			
					ORGANICS							INORGANICS						
					No. Container/Size													
					Analysis Requested													

Potential Hazard Identification:
Non-Hazard [ ] Flammable [ ] Unknown [ ]
Skin-irritant [ ] Poison [ ]

QA/QC Reporting Requirements (Circle one)
None Standard Tier II CLP
Other (specify):

Relinquished By:	Received By:	Time:	Date:	Relinquished By:	Received By:	Time:	Date:

*** Matrix Codes:**

S - soil	WW- waste water
SE - sediment	PW - potable water
SO - solid	SW - surface water
WS - solid waste	GW - ground water
DS - drum solids	DL - drum liquids
SL - sludge	ST - stormwater
O - oil	A - air
WI - wipe	F - fish
BI - biological	X - other

Special Instructions: _____

RMC Use Only

Samples were:

1. Shipped or Hand-delivered — Notes:
2. Chilled or Ambient — Notes:
3. Received broken/leaking — Yes No
4. Properly preserved — Yes No
5. Received within holding times — Yes No

COC Tape Was:

1. Present on outer package — Yes No
2. Unbroken on outer package — Yes No
3. Present on sample — Yes No
4. Unbroken on sample — Yes No
5. Discrepancies between sample labels and COC record? — Yes No

CHAIN OF CUSTODY

COMPLETING THE CHAIN OF CUSTODY

The item numbers below correspond to the blanks on the Chain of Custody.

1. The Client Name and address as it should appear on the final report. Also please show your VIP account number if known.
2. Telephone number of 1. above.
3. Job Number or Project Name.
4. Person whom the client contacted at RMC Analytics.
5. The number which is located on the Analytical Quotation.
6. When results are needed; normal turnaround is two weeks.
7. How you would like samples disposed. There is a fee for laboraroty disposal.
8. List the number and capacity of containers.
9. The analysis GROUP. This is a general grouping; all analyses included in a given container type can be grouped (CN=Cyanide, Herb=Herbicides, NH3=Ammonia, Wet Chem=Unpreserved).
10. The sample description that will appear on the Certificate of Analysis.
11. From the matrix codes provided on the form ("s" for soil, "ww" for wastewater).
12. The date and time that each sample point is collected.
13. Hazards that are known, if any.
14. Any type of QA/QC requirements that are needed for the project and/or samples.
15. The sign-off blocks should be filled out as necessary in the field; when the sample is delivered to the lab, the Sample Administrator will receive the sample for RMC.
16. Provide any special instructions.

RMC Analytics
A Division of RMC Environmental Services Inc
88 Robinson Street
Pottstown, Pa. 19464
(215) 327-4850

CHAIN OF CUSTODY

1 Client: Acme Waste Company VIP #: 11111
Address: 1234 Waste Way, Clean PA 19999

2 Phone:
Samplers:
3 Job No.

4 Lab Contact: Heidi Krueger
5 Sales Order No.: 2222
P.O. Number:

6 Turnaround Time Normal [X] Rush [ ]
Sample Disposal 7 Return to Client [X] Disposal by Lab [ ]

RMC USE ONLY

RMC only Lab ID	Sample identification	Matrix Code *	Sample Date	Sample Time	VOA	BNA	Pest/PCB	Herb			Metals	Cn	NH3	Wet Chem		
8 No. Container/Size																
9 Analysis Requested					ORGANICS						INORGANICS					
	10 Soil Pile 1	11 S	12 1/1/93	1400	X									X		

Potential Hazard Identification: 13
Non-Hazard [X] Flammable [ ] Unknown [ ]
Skin-irritant [ ] Poison [ ]

QA/QC Reporting Requirements (Circle one)
14 None Standard Tier II CLP
Other (specify):

Relinquished By	Received By	Time	Date	Relinquished By	Received By	Time	Date
15 Pete Toutquite							

* Matrix Codes:

S - soil	WW - waste water
SE - sediment	PW - potable water
SG - solid	SW - surface water
WS - solid waste	GW - ground water
DS - drum solids	DL - drum liquids
SL - sludge	ST - stormwater
O - oil	A - air
Wi - wipe	F - fish
BI - biological	X - other

16 Special Instructions:

RMC Use Only

Samples were:
1. Shipped or hand delivered — Notes
2. On ice or Ambient — Notes
3. Received broken/leaking — Yes No
4. Properly preserved — Yes No
5. Received within holding times — Yes No

COC Tape Was:
1. Present on outer package — Yes No
2. Unbroken on outer package — Yes No
3. Present on sample — Yes No
4. Unbroken on sample — Yes No
5. Discrepancies between sample labels and COC record? — Yes No

APPENDIX C

DISTILLED WATER LOG

Date	Container #	pH	Conductivity	Analyst

AQUATIC TOXICOLOGY LABORATORY

DAILY TEMPERATURE LOG

Date	Initials	Thermostat Heat	Setting Cool	Thermostat Reading	Date	Initials	Thermostat Heat	Setting Cool	Thermostat Reading

AQUATIC TOXICOLOGY LABORATORY

STANDARD REFERENCE TOXICANT

STOCK SOLUTION WEIGH/MIX RECORD

Date	Initials	Toxicant	Balance Calibration Remarks	Measured Amount	Stock Volume (g/l) DI	Final Stock Concentration	Chem Analysis mg/l K/mg/l Cl

AQUATIC TOXICOLOGY LABORATORY

DAILY METER CALIBRATION LOG

Date	Analyst	YSI Model 51B YSI 4992		Corning Checkmate 90			Thermistor Check -		Corrections		Remarks
		D.O. Probe # 90L4767	Temperature Probe # 90L4767	pH 5845	Temperature (pH probe)	Conductance 4033	NBS#	Reading	YSI	Corning	

Remarks:
1 - D.O. probe replacement
2 - D.O. probe maintenance
3 - Temperature adjustment
4 - pH probe replacement
5 - pH probe maintenance
6 - Addition of KCl solution to pH probe
7 - Battery replacement

Aquatic Toxicology Laboratory

Model S/P 120

American Scientific Analytical Balance

Serial No. 3501916

	<----	--------	--------	---------	---------	Standards	-------	-------	-------	-------	----->		
Date	10 mg	20 mg	50 mg	100 mg	200 mg	500 mg	1 g	5 g	10 g	50 g	100 g	Initials	Comments

DAPHNIA FEEDING RECORDS

	Stock Cultures	Ceriodaphnia dubia 1	2	3	4	5	6	Daphnia magna 1	Brood Board A	Brood Board B
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									
Date_____	YCT									
Init._____	Algae									

C. dubia Culture Media

Preparation:			Quantity		Culture Use:									
Date	Time	Initials	DI	Perrier	Date	Time	Initials	Total Aeration (hrs)	Temp (C)	DO mg/l	pH	Alkalinity mg/l ($CaCO_3$)	Hardness mg/l ($CaCO_3$)	Conductivity µmhos/cm

AQUATIC TOXICOLOGY LABORATORY

BRINE SHRIMP NAUPLII CULTURE RECORD SHEET

Culture Initiation			Original Open Date for Culture Material	Culture Solution	
Date	Time	Analyst		Hydrated Eggs (teaspoons)	Total Volume (ml) Eggs and Water

Brood Board Log

Analyst	Brood Board #	Brood Start Date	Parental Start Date	Renewal Date/Analyst	Date Culture Ended

Aquatic Toxicology Laboratory
Monthly Water Analysis

Initials	Date	Source		Temp (°C)	D.O. (mg/l)	pH	Conductivity		Total Residual Chlorine
_____	July ____	DI		_____	_____	_____	_____		_____
	19____	Tap		_____	_____	_____	_____		_____
			Meter #	_____	_____	_____	_____	Method	_____
_____	August ____	DI		_____	_____	_____	_____		_____
	19____	Tap		_____	_____	_____	_____		_____
			Meter #	_____	_____	_____	_____	Method	_____
_____	September ____	DI		_____	_____	_____	_____		_____
	19____	Tap		_____	_____	_____	_____		_____
			Meter #	_____	_____	_____	_____	Method	_____
_____	October ____	DI		_____	_____	_____	_____		_____
	19____	Tap		_____	_____	_____	_____		_____
			Meter #	_____	_____	_____	_____	Method	_____
_____	November ____	DI		_____	_____	_____	_____		_____
	19____	Tap		_____	_____	_____	_____		_____
			Meter #	_____	_____	_____	_____	Method	_____
_____	December ____	DI		_____	_____	_____	_____		_____
	19____	Tap		_____	_____	_____	_____		_____
			Meter #	_____	_____	_____	_____	Method	_____

AQUATIC TOXICOLOGY LABORATORY

WEEKLY NON-ROUTINE WORK CHECKLIST

Week of	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday

AQUATIC TOXICOLOGY LABORATORY
WEEKLY ROUTINE WORK CHECKLIST

Initial and Date When Completed

Week of	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday
	Brood Board Renewals _____		Brood Board Renewals _____	Start New Brood Board _____	Brood Board Renewals _____		
	Organism Feedings _____	Organism Feedings _____	Organism Feedings _____	Organism Feedings _____	Organism Feedings _____		
	Daily Temperature Log _____	Daily Temperature Log _____	Daily Temperature Log _____	Daily Temperature Log _____	Daily Temperature Log _____		
	Daily Cooler Temperature Log _____	Daily Cooler Temperature Log _____	Daily Cooler Temperature Log _____	Daily Cooler Temperature Log _____	Daily Cooler Temperature Log _____		
			Stock Culture Renewals _____				

APPENDIX D

(REV. 7/93)

TEST DILUTION MIXING VOLUMES

Concentration	Effluent (ml)	Diluent (ml)	Concentration	Effluent (ml)	Diluent (ml)
3 liters			**1.5 liters**		
100%	3,000	0	100%	1,500	0
50%	1,500	1,500	50%	750	750
25%	750	2,250	25%	375	1,125
12.5%	375	2,625	12.5%	187.5	1,312.5
6.25%	187.5	2,812.5	6.25%	93.7	1,406.3
0%	0	3,000	0%	0	1,500
1.25 liters			**1 liters**		
100%	1,250	0	100%	1,000	0
50%	625	625	50%	500	500
25%	312.5	937.5	25%	250	750
12.5%	156.3	1,093.7	12.5%	125	875
6.25%	78.1	1,171.9	6.25%	62.5	937.5
0%	0	1,250	0%	0	1,000
750 ml			**400 ml**		
100%	750	0	100%	400	0
50%	375	375	50%	200	200
25%	187.5	562.5	25%	100	300
12.5%	93.7	656.3	12.5%	50	350
6.25%	46.8	703.2	6.25%	25	375
0%	0	750	0%	0	400

STORAGE COLOR CODE KEY

Fisher Products

Red (R): **Flammable.** Store in area segregated for flammable reagents.

Blue (B): **Health Hazard.** Toxic if inhaled, ingested, or absorbed through skin. Store in secured area.

Yellow (Y): **Reactive and Oxidizing Reagents.** May react violently with air, water, or other substances. Store away from flammable and combustible materials.

White (W): **Corrosive.** May harm skin, eyes, mucous membranes. Store away from red, yellow, and blue coded reagents.

Gray (G): Presents no more than moderate hazard in any of categories above. For General Chemical Storage.

Exception: Reagent incompatible with other reagents of the same color bar. Store separately.

Mallinckrodt Products

White:	Contact Hazard (Corrosive)
Yellow:	Reactivity Hazard
Red:	Flammable Hazard
Green:	Minimal or No Hazard
Blue:	Health Hazard
Gray Stripes:	Incompatible substance

Baker Products

Red:	Flammable
Orange:	General Storage Minimal or No Hazard

ALWAYS CONSULT MSDS FOR PROPER STORAGE AND DISPOSAL PROCEDURES!

STANDARD REFERENCE TOXICANT TOXICITY TEST PROCEDURES

I. Standard Reference Toxicant (SRT)

- RMC uses a stock solution of potassium chloride at a concentration of 2,000 mg/L for Pimephales promelas, and a concentration of 1,000 mg/L for Ceriodaphnia dubia.
- Stock solutions are prepared by weighing out 2 g KCl to the nearest mg and dissolving in 750 ml of distilled water. Once the KCl has completely dissolved the solution is diluted to 1-liter with distilled water.
- The exact amount of KCl measured must be recorded on the SRT mixing log.
- 4-liters of SRT are usually prepared at one time.
- Each new batch of KCl must be subsampled and an aliquot analyzed by RMC Analytics to document the exact quantities of potassium and chloride ions in the solution.
- From the 2,000 mg/L KCl stock solution an aliquot of 750 ml is diluted with 750 mg/L of distilled water to prepare the 1,000 mg/L KCl SRT for C. dubia.

II. Dilutions

- The SRT stock solution is used in place of the effluent in a standard acute or chronic toxicity test.
- Dilutions for P. promelas

Concentration (mg/L)	ml 2,000 mg/L SRT	ml 20% DMW
2,000	1,500	0
1,000	750	750
500	375	1,125
250	187.5	1,312.5
125	93.75	1,406.25
0	0	1,500

- Dilutions for C. dubia

Concentration (mg/L)	ml 2,000 mg/L SRT	ml 20% DMW/B_{12}/se
1,000	200	200 (DI water)
500	100	300
250	50	450
125	25	475
62.5	12.5	487.5

III. Chronic Reference Toxicant Test

- Procedures followed are those presented in EPA/600/4-89/001 Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Water to Freshwater Organisms (i.e., the SRT is run as though it were a chronic toxicity test for a client).

IV. Acute Reference Toxicant Test

- A definitive test is performed using the procedures presented in EPA/600/4-90/027 Methods of Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (i.e., the SRT is run as though it were a definitive test for a client).

V. Frequency of SRT's

- Standard reference toxicant tests are performed monthly for organisms cultured in-house, and quarterly for organisms purchased from a supplier provided the supplier sends reference toxicant data. If the supplier has not conducted an SRT, then it must be performed in-house. Tests should be conducted more frequently should problems arise.

VI. Control Chart

- A control chart is generated from the SRT data and should be updated after each SRT completion.

Instructions for the Preparation of

Dilute Mineral Water (DMW)

A mixture of 20% diluted mineral water is prepared for use as culture media for fathead minnow and dilution water in chronic tests. The 20% DMW is prepared daily in the following manner:

1. 1 liter of deionized water from a reverse osmosis system.
2. Add 250 ml of Perrier to each liter of DI water.
3. Aerate the 20% DMW for 24 hours before use.
4. Maintain a log of p-chems on each batch of 20% DMW prepared. Record the following information: Temp, pH, D.O., conductivity, alkalinity, hardness, and total aeration time.

Instructions for the Preparation of

Dilute Mineral Water with Se/B_{12} (DMW)

A mixture of 20% diluted mineral water is prepared for use as culture media for C. dubia and dilution water in chronic tests. The 20% DMW is prepared daily as described above. However, the 20% DMW is "spiked" with a stock solution of vitamin B_{12} and Se, so as to produce a final concentration of 1 μg/L vit. B_{12} and 5 μg/L Se.

I. Preparation of stock solutions

A. 1 mg/L vit B_{12}

1. Weigh out 0.0010 g of vit. B_{12}.
2. Dissolve the B_{12} in 750 mL of deionized water.
3. Bring the final volume of 1 liter with deionized water.
4. Store the stock solution in the refrigerator.

B. 5 mg/L Se

1. The stock solution is prepared using a 1 mg/L Atomic Absorption Se with distilled water as the solvent. This may be purchased from Fisher Scientific.
2. Dilute 5 mL of the Atomic Absorption Se to 1 liter with deionized water.
3. Store the stock solution at room temperature.

II. Preparation of 1 μg/L vit. B_{12}, 5 μg/L Se 20% DMW.

1. Dilute 1 mL of each stock solution to 1 liter with 20% DMW.

STANDARD REFERENCE TOXICANT

MIXING VOLUMES

Concentration	2,000 mg/L KCl Stock Solution (ml)	DMW (ml)
For 1.5 liters		
2,000 mg/L	1,500	0
1,000 mg/L	750	750
500 mg/L	375	1,125
250 mg/L	187.5	1,312.5
125 mg/L	93.7	1,406.3
0 mg/L	0	1,500
For 1.0 liters		
2,000 mg/L	1,000	0
1,000 mg/L	500	500
500 mg/L	250	750
250 mg/L	125	875
125 mg/L	62.5	937.5
0 mg/L	0	1,000
For 500 ml (C. dubia)		
1,000 mg/L	250	250
500 mg/L	125	375
250 mg/L	62.5	437.5
125 mg/L	31.3	468.7
62.5 mg/L	15.6	484.4
0 mg/L	0	500

Concentration	2,000 mg/L KCl Stock Solution (ml)	DMW (ml)
For 1.25 liters		
2,000 mg/L	1,250	0
1,000 mg/L	625	625
500 mg/L	312.5	937.5
250 mg/L	156.3	1,093.7
125 mg/L	78.1	1,171.9
0 mg/L	0	
For 750 ml		
2,000 mg/L	750	0
1,000 mg/L	375	375
500 mg/L	187.5	562.5
250 mg/L	93.7	656.3
125 mg/L	46.9	703.1
62.5 mg/L	23.4	726.6
0 mg/L	0	750

*Mix fish dilutions in one gallon glass jars: 2,000 mg/L, 1,000, 500, 250, 125, 0

*Mix C. dubia dilutions in one liter plastic bottles: 1,000 mg/L, 500, 250, 125, 62.5, 0

Instructions for Preparing

Yeast/Cerophyll/Trout Chow Suspension (YCT)

1. Digest 5 g of #1 trout chow pellets in 2-liters of distilled water for one week.
2. Digestion takes place in a 1-gallon jug that is continually aerated with an air stone.
3. When digestion is complete place the trout chow suspension to settle in a refrigerator for 1-hour prior to mixing.
4. Weigh out 5 g of alfalfa tablets and mix in a blender with 1-liter of distilled water.
5. Place alfalfa mixture in the refrigerator for 24-hours to settle.
6. Prepare a yeast suspension by weighing out 5 g of Fleishmann's activated yeast, and mix in a blender with 1-liter of distilled water. Do not allow to settle before use.
7. Mix 300 ml each of digested trout chow, yeast suspension, and alfalfa.
8. Feed C. dubia 0.1 ml per 15 ml dilution water daily. Feeding is done using a digital pipetter which delivers an exact amount to each test chamber.

Drying and Weighing Instructions for Larval Fathead Minnows

At the conclusion of a 7-day chronic toxicity test. The larval fish need to be dried and weighed. Use the following procedure to prepare the fish for weighing:

1. Remove the fish from their test chambers by filter through a fine mesh net.
2. Rinse the fish with DI water to remove particulate matter.
3. Rinse the fish from the net into a 30 ml beaker with DI water. (One beaker for each replicate.)
4. Lower the water level to 10 to 15 ml in the beaker.
5. Prepare labeled weigh boats by placing them into a drying oven for one hour.
6. Place weigh boats into a desiccator until cool and weigh to the nearest 0.1 mg.
7. Place each replicate of fish into a weigh boat and dry fish at 100 C for a minimum of five hours.
8. Remove fish from oven and place in a desiccator until cool.
9. Weigh each replicate to the nearest 0.1 mg.

Instructions for Preparing

Brine Shrimp nauplii (Artemia sp.) Culture

1. Culture temperature - 25 C ± 2 C.
2. Measure out 2-liters distilled water into a one gallon jar.
3. Mix 6-teaspoons of Forty Fathoms sea salt, until dissolved.
4. Disperse 1-teaspoon of brine shrimp eggs in the surface of the solution.
5. Place two airstones into the jar and aerate with enough force to keep the eggs from settling.
6. Light conditions: Ambient laboratory lighting or place under a timed light: 16 hours light, 8 hours dark.
7. Brine shrimp nauplii will hatch in 18 to 24 hours.

Brine Shrimp (Artemia sp.) Preparation for
Feeding to Laboratory Organisms

1. Turn off aeration ten minutes prior to filtration.
2. Filter brine shrimp through Wattmann filter paper. Wet the paper with 20% DMW.
3. Pipette 150 to 200 ml of concentrated brine shrimp from the gallon jar.
4. When the saltwater has drained off wash the brine shrimp thoroughly with 20% DMW.
5. Rinse the brine shrimp off the filter paper with 20% DMW into a beaker.
6. Dilute the concentrate to approximately 40 ml with 20% DMW.
7. Each test vessel is fed 0.1 ml twice per day. One feeding is performed upon arrival in the morning, and the second feeding occurs at the end of the day prior to leaving. Feeding is performed with a digital pipetter which delivers the exact amount required to each test vessel.

RMC ENVIRONMENTAL SERVICES, Inc.

3450 Schuylkill Road
Spring City, PA 19475

215•948•4700
215•948•4752 FAX

Delivery of Samples for Acute and Chronic Bioassays

Twenty-four hour composite samples of effluent must be collected on three separate 24-hour collection schedules and shipped overnight to our laboratory. This is most easily accomplished as follows:

1. RMC Environmental Services, Inc. has shipped three coolers, two contain two 4 liter (one gallon) cubitainers for effluent and one contains four 4 liter (one gallon) cubitainers, icies, chain-of-custody seals, and chain-of-custody forms. Remove the icies and freeze.

2. Plant personnel should set an automatic sampler on Sunday, and ship two cubitainers (8 liters) of effluent via overnight delivery on Monday. We will initiate chronic testing on Tuesday, upon receipt of the sample (Day 1), and will use a portion of the sample for a Wednesday water change (Day 2).

3. Begin a second setting of the automatic sampler with a doubled volume on Tuesday, and ship four cubitainers (16 liters) on Wednesday. We will use portions of the sample for a Thursday water change (Day 3) and a Friday water change (Day 4), and for the acute toxicity tests.

4. A third and final setting of the automatic sampler on Thursday, with a two cubitainer (8 liter) shipment on Friday, will be used for water changes on Saturday, Sunday, and Monday (Days 5 through 7).

For freshwater chronic tests we require a minimum sample volume of eight liters (two gallons) for each of the three shipments. For freshwater acute tests we require a minimum sample volume of eight liters (two gallons) from the second sampling period.

It is important that the effluent be kept chilled during transportation. Please note that UPS does not deliver on Saturdays. Sample containers should be shipped to the following address:

RMC Environmental Services, Inc.
3450 Schuylkill Road
Spring City, PA 19475

If you have any questions regarding these instructions, please contact me at (215)948-4700, ext. 145.

Jennifer L. Koslo

Jennifer L. Koslo
Aquatic Toxicology Laboratory Manager

Printed on recycled paper

Delivery of Samples for Chronic Bioassays

Twenty-four hour composite samples of effluent must be collected on three separate 24-hour collection schedules and shipped overnight to our laboratory. This is most easily accomplished as follows:

1. RMC Environmental Services, Inc. has shipped three coolers, each containing two four liter (one gallon) cubitainers for effluent, icies, chain-of-custody seals, and chain-of-custody forms.

2. Plant personnel should set an automatic sampler on Sunday, and ship the effluent via overnight delivery on Monday. We will initiate testing on Tuesday, upon receipt of the sample (Day 1), and will use a portion of the sample for a Wednesday water change (Day 2).

3. Begin a second setting of the automatic sampler on Tuesday, with a shipment on Wednesday. We will use portions of the sample for a Thursday water change (Day 3) and a Friday water change (Day 4).

4. A third and final setting of the automatic sampler on Thursday, with a shipment of Friday, will be used for water changes on Saturday, Sunday, and Monday (Days 5 through 7).

For freshwater tests we require a minimum sample volume of eight liters (two gallons) for each of the three shipments.

It is important that the effluent be kept chilled during transportation. Please note that UPS does not deliver on Saturdays. Sample containers should be shipped to the following address:

RMC Environmental Services, Inc.
3450 Schuylkill Road
Spring City, PA 19475

If you have any questions regarding these instructions, please contact me at (215)948-4700, ext. 145.

Jennifer L. Koslo

Jennifer L. Koslo
Aquatic Toxicology Laboratory Manager

INSTRUCTIONS FOR PROGRAMMING THERMOSTAT

A. Set the program schedule and heat/cool temperatures.

1. Press DAY
   - M-F will appear in the display.
2. Press WAKE
3. Press SET HEAT/COOL
   - If the display reads cool, program the temperature using the "WARMER" and "COOLER" keys.
4. Press SET HEAT/COOL
   - If the display reads heat, program the temperature using the "WARMER" and "COOLER" keys.

* The heating temperature must be at least three degrees below the air conditioning temperature.

5. Press and release LEAVE
6. Press and release SLEEP
7. Press and release RETURN
8. Press DAY
   - SAT will appear on display.
   - Repeat above temperature setting procedure.
9. Press DAY
   - SUN will appear on the display.
   - Repeat above temperature setting procedure.
10. Press RUN PROGRAM

    To check the current setting, press PRESENT SETTING. For other options, i.e., temporarily changing the program and operating the thermostat manually, consult the instruction manual.

Temperature Settings for Toxicity Tests

ACUTE (20°)	CHRONIC (25°)
Set heat - 70	Set heat - 78
Set cool - 73	Set cool - 81

US EPA GLASSWARE CLEANING PROCEDURE

1. Soak 15 minutes in tap water, and scrub with detergent, or clean in an automatic dishwasher.

2. Rinse twice with tap water.

3. Carefully rinse once with fresh, dilute (10%, V:V) hydrochloric or nitric acid to remove scale, metals, and bases. To prepare a 10% solution of acid, add 10 ml of concentrated acid to 90 ml of deionized water.

4. Rinse twice with deionized water.

5. Rinse once with full-strength, pesticide-grade acetone to remove organic compounds (use a fume hood or canopy).

6. Rinse three times with deionized water.

Chemically cleaned items are inverted and stored on shelves in the laboratory until needed. After use in a toxicity test, a contaminated item is emptied of its contents (if applicable), rinsed with tap water, and placed in a designated area until the item may be chemically cleaned. Contaminated items are chemically cleaned as soon as possible after a toxicity test is terminated.

All test chambers and equipment should be thoroughly rinsed with the dilution water immediately prior to use in each test. Sample collection containers are rinsed with sample before filling.

Effluent Sampling and
Sample Handling and Shipping Instructions

1. Effluent Sampling

- The effluent sampling point is ordinarily a point between the final treatment and the discharge outfall. The sampling point should be easily accessible and should have sufficient area in which to place an automatic sampler.

- The effluent sample should be a 24-h composite sample collected over one 24-h period. An automatic wastewater sampler should be used to collect the sample. At a minimum, the sampler should be programmed to collect one sample every hour. A minimum hourly sample volume of 160 ml would be necessary to obtain a total sample volume of 3,840 ml (approximately one U.S. gallon) at the end of the 24-h period.

- The sample should be collected into a lead free glass container, and should be chilled as it is collected.

2. Effluent Sample Handling and Shipping

- Sample "holding time," defined here, begins when the composite sampling period is completed. If the data from the samples are to be considered representative of the true effluent characteristics, the lapsed time from collection to first use of the sample in test initiation should not exceed 36-h. The results of tests using samples held more than 36-h may not reflect the true toxicity of the effluent at the time of collection. The sampling schedule should be adjusted so that the lapsed time from sample collection to shipment is held to a minimum.

- To minimize the loss of toxicity due to volatilization of toxic constituents, all sample containers should be "completely" filled, leaving no air space between the contents and the lid.

- Samples collected for off-site toxicity testing are to be chilled to 4 C during and immediately after collection, and shipped iced to RMC Environmental Services, Inc.'s laboratory.

- RMC will provide all locations with a cooler, several ice packs, a one gallon plastic "milk" jug, and Chain-of-Custody forms. All sample containers should be rinsed with source water before being filled with sample. The sample container and Chain-of-Custody form should be completed with the date and time of sample collection, the sample location, and whether the sample has been chlorinated.

- Several sample shipping options are available, including air express. The samples should be shipped overnight to our laboratory. Samples should be shipped to the following address:

  RMC Environmental Services, Inc.
  3450 Schuylkill Road
  Spring City, PA 19475

- Questions regarding the sampling protocol should be directed to Jennifer Koslo at (215)948-4700, ext. 145.

RMC
ENVIRONMENTAL SERVICES

RMC ENVIRONMENTAL SERVICES, Inc.

RMC
ENVIRONMENTAL
SERVICES

3450 Schuylkill Road
Spring City, PA 19475

215•948•4700
215•948•4752 FAX

Delivery of Samples for Acute Screening Bioassays

Twenty-four hour composite samples of effluent must be collected on three separate 24-hour collection schedules and shipped overnight to our laboratory. This is most easily accomplished as follows:

1. RMC Environmental Services, Inc. has shipped three coolers, each containing a one gallon container for effluent, icies, and chain-of-custody forms.

2. Plant personnel should set an automatic sampler on Sunday, and ship the effluent via Overnight Delivery on Monday. We will initiate the first two tests on Tuesday, on receipt of the sample (Test 1).

3. Begin a second setting of the automatic sampler on Tuesday, with a shipment on Wednesday. We will initiate testing on Thursday, on receipt of the sample (Test 2).

4. A third and final setting of the automatic sampler on Thursday, with a shipment of Friday, will be used to initiate the third and final test on Saturday.

For freshwater tests we require a minimum sample volume of one gallon for each of the three shipments.

It is important that the effluent be kept chilled during transportation. Please note that UPS does not deliver on Saturdays. Sample containers should be shipped to the following address:

RMC Environmental Services, Inc.
3450 Schuylkill Road
Spring City, PA 19475

If you have any questions regarding these instructions, please contact me at (215)948-4700, ext. 145.

Jennifer L. Koslo
Aquatic Toxicology Laboratory Manager

Printed on recycled paper

APPENDIX E

(REV. 7/93)

BIOASSAY EQUIPMENT AND INSTRUCTION MANUALS

1. Masterflex pump heads and drives
2. Forma Scientific bath and circulator Model 2045-Masterline
3. Forma Scientific bath and circulator Model 2302
4. ACE Glass Dilutor System with Model 14058 control box
5. Aquafine electronic liquid sterilizer Model SL-10-A
6. YSI 71-A thermistemp temperature controller
7. YSI tele-thermometer Model 425C
8. YSI Model 51B oxygen meter
9. YSI Model 33 S-C-T meter (salinity-conductivity-temp)
10. Lab Line Model N9620-10 TampCon oven
11. Fisher ThermX stirrer Model 120M
12. Corning stirrer PC-353
13. Nikon dissecting scope
14. Corning checkmate modular testing system - temperature, dissolved oxygen, pH, conductivity, Redox Potential
15. American Scientific Products Electronic Analytical Balance Model S/P120
16. USA Standard Testing Sieves (ASTM E-11 Specification)
17. Soil Hydrometer (ASTM 152H)
18. ASTM Soil Hydrometer Cylinder

APPENDIX F

(REV. 7/93)

WATER ANALYSIS PARAMETER LIST

	Type of Water	Test Frequency	Parameters
1.	filtered tap water	monthly	temperature, pH, dissolved oxygen, conductivity total residual chlorine
2.	distilled water	each new batch from RMC Analytics	pH, conductivity
3.	distilled water	monthly	temperature, pH, dissolved oxygen, conductivity total residual chlorine
4.	distilled water	biannual	alkalinity, total as $CaCo_3$ ammonia, nitrogen chemical oxygen demand hardness as $CaCo_3$ organochlorine pesticides polychlorinated biphenyls (liquid) organophosphate pesticides carbon, total organic chlorine, total residual total suspended solids

APPENDIX G

(REV. 7/93)

RMC PROTOCOL SUMMARY FOR
ACUTE TOXICITY TESTS

Date: _____

Prepared by: _____

Facility

Name: _____

Location: _____

_____

NPDES Number: _____

Contact Person: _____

Telephone Number: _____

Permit Requirements

Test Species: _____

_____

Toxicity Testing Frequency in Permit: _____

Test Method Specified in Permit: _____

Test Endpoints: _____

Design Summary: _____

_____

_____

PART I: GENERAL CONDITIONS

Effluent Sampling

Treatment Plant: Retention Time (hrs) _____
Discharge (continuous/intermittent/batch) _____

Collection Point: _____

Effluent Type: _____

RMC PROTOCOL SUMMARY FOR
CHRONIC TOXICITY TESTS

Date: _____

Prepared by: _____

Facility

Name: _____

Location: _____

_____

NPDES Number: _____

Contact Person: _____

Telephone Number: _____

Permit Requirements

Test Species: _____

_____

Toxicity Testing Frequency in Permit: _____

Test Method Specified in Permit: _____

Test Endpoints: _____

Design Summary: _____

_____

_____

PART I: GENERAL CONDITIONS

Effluent Sampling

Treatment Plant: Retention Time (hrs) _____
Discharge (continuous/intermittent/batch) _____

Collection Point: _____

Effluent Type: _____

APPENDIX H

WATER COLUMN SEDIMENT TOXICITY TEST
Organism Survival

Client: _____ Job No: _____

rt Date: _____ Species: _____

Concentration	Replicate	# Organisms at Test Initiation	Hours						Comments
	1								
	2								
Control	3								
	4								
	5								
	1								
	2								
	3								
	4								
	5								
	1								
	2								
	3								
	4								
	5								
	1								
	2								
	3								
	4								
	5								
	1								
	2								
	3								
	4								
	5								
	1								
	2								
	3								
	4								
	5								
Date									
Time									
Analyst									

SEDIMENT P-CHEM SHEET

Client: _____ Job # _____ Start Date _____ End Date _____

Sample/ Concentration	P-Chem Parameter	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8	Day 9	Day 10	Sediment Characteristics
	Analyst											
	Sediment Temp											
	Sediment Redox Pot											% Moisture
	Water Column Temp											
CONTROL	Water Column pH											Particle Size Analysis
	Water Column D.O.											% Sand
	Conductivity/Salinity											% Silt
	Alkalinity											% Clay
	Hardness											
	Sediment Temp											
	Sediment Redox Pot											% Moisture
	Water Column Temp											
_____	Water Column pH											Particle Size Analysis
	Water Column D.O.											% Sand
	Conductivity/Salinity											% Silt
	Alkalinity											% Clay
	Hardness											
	Sediment Temp											
	Sediment Redox Pot											% Moisture
	Water Column Temp											
_____	Water Column pH											Particle Size Analysis
	Water Column D.O.											% Sand
	Conductivity/Salinity											% Silt
	Alkalinity											% Clay
	Hardness											
	Sediment Temp											
	Sediment Redox Pot											% Moisture
	Water Column Temp											
_____	Water Column pH											Particle Size Analysis
	Water Column D.O.											% Sand
	Conductivity/Salinity											% Silt
	Alkalinity											% Clay
	Hardness											
	Sediment Temp											
	Sediment Redox Pot											% Moisture
	Water Column Temp											
_____	Water Column pH											Particle Size Analysis
	Water Column D.O.											% Sand
	Conductivity/Salinity											% Silt
	Alkalinity											% Clay
	Hardness											

APPENDIX I

(REV. 7/93)

DATA ANALYSIS

Test Organism _____ RMC No. _____

Response _____ Test No. _____

Was any non-concentration-related mortality observed? Y _____ N _____

Were any stimulatory effects observed? Y _____ N _____

Were concentrations excluded due to survival LOEC:_____ Y _____ N _____

Data Transformation: _____ Arcsine-Square root (adjusted at 0.0 and 1.0)
_____ Log (base 10)
_____ Other _____

Endpoint Determinations:

Were there equal number of replicates amoung concentrations? Y _____ N _____
_____ Shapiro-Wilks normal distribution test: p=_____ Normal Y _____ N _____
_____ Bartletts variance homogeneity test: p=_____ Homog. Y _____ N _____
_____ ANOVA / Dunnetts test
_____ ANOVA / Bonferroni t-test
_____ Steels Many-One Rank Sum test
_____ Wilcoxon Rank Sum test
_____ Fishers Exact test
_____ Other_____

Minimum detectable difference = _____ *percent of control*

NOEC _____ LOEC _____

LCp / ICp estimation

_____ Probit Analysis
_____ Linear Interpolation (_____using smoothed/pooled response means)
_____ Other_____

LC / IC (10) _____
LC / IC (25) _____
LC / IC (50) _____
LC / IC ____ _____

Comments: _____

_____

_____

_____ _____
Signature Date

Collection Method: Grab _____
Composite sampler _____

If composite, compositing period (hrs) _____
number of grab samples per composite _____
interval between grab samples (minutes) _____

Sample Collection: lab personnel _____ permittee _____

Number of samples to be collected during test duration:_____

Sample Filtered (Y/N): _____ If yes, screen mesh size (mm) _____

Maximum Holding Time (hrs): _____

Sample Storage Container Type:_____

Sample Storage Temperature (C): _____

Aeration of sample anticipated (Y/N): _____

Sample Records: Logged on Chain-of-Custody Forms: Yes ____ No ____
Total Residual Chlorine levels determined: Yes ____ No ____

Dilution Water Source and Sampling

Receiving Stream: _____

Dilution Water Source: _____

Collection location relative to effluent discharge: _____

_____

Storage Container Type: _____

Storage Temperature (C): _____ Maximum Holding Time (hrs): _____

Alternate Dilution Water:

Briefly describe methods for preparation of Diluted Mineral Water (DMW) or Reconstituted Water:

_____
_____
_____

Sample filtered (Y/N): ____ If yes, screen mesh size (μm): _____

Maximum percent of diluted mineral water in a sample: _____

Maximum holding time (hrs): _____ Storage temperature (C): _____

Aeration of sample anticipated: Yes ____ No ____

Please specify type and amount of any micronutrients to be added to the dilution water:

_____

_____

_____

PART II: SPECIES RELATED CONDITIONS

Test Organism

Organism Supply

Species: 1. _____ _____
(common name) (scientific name)

2. _____ _____
(common name) (scientific name)

Source (check one): 1. _____ cultured _____ purchased from supplier

2. _____ cultured _____ purchased from supplier

Culture Stock Source: 1. _____

2. _____

Commercial Supplier: 1. _____

2. _____

Light/Culturing Conditions

Culture Method: _____

Tank Type: _____

Tank Liquid Volume (l): _____ Water Source: _____

Mean Water Temperature (C): _____ Maximum Range: _____

Food: _____ Feeding Regime: _____

Lighting: 16 hours light/8 hours dark cycle: Yes _____ No _____

Mean Salinity (ppt): _____ Maximum Salinity Range: _____

Minimum Dissolved Oxygen Concentration (% Sat.): _____

Dilution Water:

Effluent receiving water: _____

Dilution water source: _____

If a substitute dilution water was used, had its use been approved by PA DER? Yes _____ No _____

Collection location: _____

Collection date(s): _____

Bioassay Results:

	24 hour	48 hour	72 hour	96 hour
LC_{50}/EC_{50} (% effluent):	_____	_____	_____	_____

Calculation Method: _____

Does the data satisfy the statistical assumptions of the specified calculation method? Yes _____ No _____

Is the calculated LC_{50}/EC_{50} valid according to the specifications of the method used? Yes _____ No _____

Miscellaneous:

Was test organism stress observed during the test? Yes _____ No _____

If yes, specify concentrations and abnormalities _____

_____

Were any exposure chambers aerated during the test? Yes _____ No _____

If yes, specify concentrations and duration _____

_____

Printed on recycled paper

RMC ENVIRONMENTAL SERVICES

Test Organism Data:

Test organism source:

RMC culture _____ Commercial Hatchery (specify) _____

Test organism acclimation to dilution water:

Initial number of organisms	_____	
Total acclimation period	_____ day(s)	_____ hour(s)
Acclimation period to 100 percent dilution water at the specified test temperature:	_____ hour(s)	
Number of mortalities 48 hours prior to test:	_____	
Test organism age at start of test:	_____ day(s)	_____ hour(s)

Test Design:

Number of effluent test concentrations (minimum of 5) _____

Number of replicates/test concentration: _____

Number of test organisms/replicate: _____

Volume of liquid in test chambers: _____ ml

Flow-through bioassay exchange rate: _____ (cycles/day)

Effluent Sampling:

Plant sampling location: _____

Treatment plant retention time (hours): _____

Type of sample: Grab ____ 6 hr composite ____ 24 hr composite ____ Continuous feed ____

Sample Collection:

Beginning date: _____ Beginning time: _____
Ending date: _____ Ending time: _____

If composite sample, number of grab samples in composite: _____

Intervals between grab samples (minutes): _____

Maximum sample holding time (hours): _____

Testing location: On site _____ Remote Laboratory _____

Printed on recycled paper

AQUATIC TOXICITY LABORATORY
DAILY WORK CHECKLIST

Discharger: _____ Job Number: _____

Start Date: _____ Test Number: _____

The Eight Days of Chronic Toxicity

	1	2	3	4	5	6	7	8
Feed Fish (AM)								
Mix Brine Shrimp								
Sample Log In								
Sample Description- Odor								
Color								
Filter								
Dechlorinate								
Mix Dilutions								
Initial P-chems								
Final P-chems (fish)								
Renewals - Fish								
Renewals - C. dubia								
Final P-chems (C. dubia)								
Feeding (PM) C. dubia								
Fish								
Mix DMW								
Titrations								
Adjust Room Temp (PM)								
QA Data Entries								
Final C. dubia Counts								
Dry/Weigh Pans								
Dry/Weigh Fish								
_____								
_____								
_____								
_____								
_____								

Maximum test organism age at start of test (days):_____

Name and source of Standard Reference Toxicant to be used?

1. NOEC/LOEC: _____ / _____ Acceptable range: _____

2. NOEC/LOEC: _____ / _____ Acceptable range: _____

Test Duration (days): _____

Test Type (check one): **Static** _____ **Renewal (24 hr)** _____

Test Vessels (material and size):_____

Test Liquid Volume (ml): _____ **Test Temperature (C):** _____

Aeration procedures to be followed, if required: _____

_____

_____

Photoperiod/illumination: _____ / _____

Physical/chemical parameters monitored and monitoring frequency: _____

_____

_____

Dilution Factor: 0.3 _____ 0.5 _____ Other (specify): _____

Is the permit limitation one of the test concentrations: Yes _____ No _____

Number of test concentration: _____

Number of organisms per concentration: _____

Number of replicates/concentration: _____

Will the test chambers be covered: Yes _____ No _____

Please describe weighing/drying procedures: _____

_____

_____

_____

Calculation methods to be used:_____

_____

_____

_____

Please list any anticipated deviations from the specified methods:_____

_____

_____

_____

RMC ENVIRONMENTAL SERVICES, Inc.

RMC ENVIRONMENTAL SERVICES

3450 Schuylkill Road
Spring City, PA 19475

215•948•4700
215•948•4752 FAX

Temperature / Dissolved Oxygen Data

Client : _____ Job No. _____

Test Date : _____

D.O. Meter # _____ D.O. Probe # _____

Percent Dilution	Temperature (degrees C)					Dissolved Oxygen (ppm)				
	0	24	48	72	96	0	24	48	72	96
0 A										
0 B										
A										
B										
A										
B										
A										
B										
A										
B										
A										
B										
Date										
Time										
Initials										

Notes : _____

Printed on recycled paper

Number of replicates/concentration: _____

Will the test chambers be covered: Yes ____ No ____

Length and weight restrictions for test organisms: _____

_____

Maximum loading factors: _____

_____

Please describe weighing/drying procedures: _____

_____

Calculation methods to be used: _____

_____

Please list any anticipated deviations from the specified methods: _____

_____

_____

RMC ENVIRONMENTAL SERVICES, Inc.

RMC ENVIRONMENTAL SERVICES

3450 Schuylkill Road
Spring City, PA 19475

215-948-4700
215-948-4752 Fax

Biomonitoring Report Form - Acute Bioassays

Permit No.: _____

Facility Name: _____

RMC Project No.: _____

Facility Location: _____

Laboratory/Investigator: _____

_____

Bioassay Specifications:

Effluent Type (e.g., final, pre-chlorination): _____

Test Type: Static _____ Renewal (6 hr) _____ Renewal (24 hr) _____ Flow through _____

Test Duration (hours): 24 _____ 48 _____ 96 _____ Other (specify) _____

Test Organism: _____ _____
(common name) (scientific name)

Test end point: LC_{50} _____ EC_{50} _____ Other (specify) _____

Summary of Final Results:

Test Starting Date: _____ Completion Date: _____

LC_{50}/EC_{50} (% effluent): _____ 95% Confidence Interval: _____ to _____

Percent mortality in 100% effluent: _____

Quality Control Summary:

Control Mortality: _____ percent

Temperature maintained with +/-2 deg. C of test temperature? Yes _____ No _____

Dissolved Oxygen levels always greater than 40% saturation? Yes _____ No _____

Loading factor for all exposure chambers less than or equal to maximum allowed for the test type and temperature? Yes _____ No _____

Two or more concentrations exhibit a trend deviation? Yes _____ No _____

Certification:

Accuracy of report certified by: _____ _____
Laboratory Manager Date

Printed on recycled paper

RMC ENVIRONMENTAL SERVICES, Inc.

3450 Schuylkill Road
Spring City, PA 19475

215-948-4700
215-948-4752 FAX

Test Organism Length / Weight Data

Client: _____ Job No: _____

Test Dates: _____ Test Species: _____

_____ Source: _____

_____ Age: _____

	Length mm	Weight grams
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

Test Results

Mean Length (mm) _____

Stand. Dev. of Length +/- _____

Longest Organism _____

Shortest Organism _____

Mean Weight (mm) _____

Stand. Dev. of Weight +/- _____

Initials _____

Date _____

Notes: _____

_____

_____

_____

_____

_____

_____

_____

Printed on recycled paper

DILUTOR CALIBRATION LOG

Client:_____ Job No:_____

Start Date:_____ End Date:_____

Time:_____ Time:_____

Elapsed Cycles:_____

Elapsed Time:_____

Proportioning Chambers:

Date/ Time	0 Eff	____ Dil	____ Eff	____ Dil	____ Eff	____ Dil	____ Eff	____ Dil	____ Eff	100 Eff	Initials
Correction											
Correction											
Correction											
Correction											
Correction											

Splitters:

Date/ Time	0 A	0 B	____ A	____ B	____ A	____ B	____ A	____ B	____ A	____ B	100 A	100 B	Initials
Correction													
Correction													
Correction													
Correction													
Correction													

QA/QC_____

RMC ENVIRONMENTAL SERVICES, Inc.

RMC ENVIRONMENTAL SERVICES

3450 Schuylkill Road
Spring City, PA 19475

215-948-4700
215-948-4752 FAX

Mortality / Observation Data

Client : _____ Job No. _____

QA/QC _____ Test Date : _____

Percent Dilution	Live Count 0	24	48	72	96	Notes and Observations
0 A						
0 B						
A						
B						
A						
B						
A						
B						
A						
B						
A						
B						
Date						
Time						
Initials						

Aeration Procedure	Hours	Initials
Cycles Increased (where possible)	_____	_____
Exposure Chamber Aeration Started	_____	_____

RMC ENVIRONMENTAL SERVICES, Inc.

3450 Schuylkill Road
Spring City, PA 19475

215-948-4700
215-948-4752 FAX

Alkalinity / Hardness Data

Client : _____ Job No. _____

Test Date : _____

Method _____

Percent Dilution	Alkalinity as CaCO3					Hardness as CaCO3				
	0	24	48	72	96	0	24	48	72	96
0										
Date										
Time										
Initials										

Notes : _____

Printed on recycled paper

RMC ENVIRONMENTAL SERVICES, Inc.

RMC ENVIRONMENTAL SERVICES

3450 Schuylkill Road
Spring City, PA 19475

215•948•4700
215•948•4752 FAX

Conductivity / pH Data

Client : _____ Job No: _____

Test Date : _____

pH Meter # _____ Conductivity Meter # _____

Percent Dilution	pH					Conductivity				
	0	24	48	72	96	0	24	48	72	96
0 A										
0 B										
A										
B										
A										
B										
A										
B										
A										
B										
A										
B										
Date										
Time										
Initials										

Notes : _____

Printed on recycled paper

Please specify type and amount of any micronutrients to be added to the dilution water:

_____

_____

_____

PART II: SPECIES RELATED CONDITIONS

Test Organism

Organism Supply

Species: 1. _____ _____
(common name) (scientific name)

2. _____ _____
(common name) (scientific name)

Source (check one): 1. _____ cultured _____ purchased from supplier

2. _____ cultured _____ purchased from supplier

Culture Stock Source: 1. _____

2. _____

Commercial Supplier: 1. _____

2. _____

Maximum test organism age at start of test (days - hours): 1. _____ - _____

2. _____ - _____

Lighting: 16 hours light/8 hours dark cycle: Yes _____ No _____

Light/Culturing Conditions

Culture Method: _____

_____

Tank Type: _____

Tank Liquid Volume (l): _____ Water Source: _____

Mean Water Temperature (C): _____ Maximum Range: _____

Food: _____ Feeding Regime: _____

Collection Method: Grab _____
Composite sampler _____

If composite, compositing period (hrs) _____
number of grab samples per composite _____
interval between grab samples (minutes) _____

Sample Collection: lab personnel _____ permittee _____

*Number of samples to be collected during test duration:*_____

Sample Filtered (Y/N): _____ If yes, screen mesh size (mm) _____

Maximum Holding Time (hrs): _____

Sample Storage Container Type:_____

Sample Storage Temperature (C): _____

Aeration of sample anticipated (Y/N): _____

Sample Records: Logged on Chain-of-Custody Forms: Yes ____ No ____
Total Residual Chlorine levels determined: Yes ____ No ____

Dilution Water Source and Sampling

Receiving Stream: _____

Dilution Water Source: _____

Collection location relative to effluent discharge: _____

_____

Storage Container Type: _____

Storage Temperature (C): _____ Maximum Holding Time (hrs): _____

Alternate Dilution Water:

Briefly describe methods for preparation of Diluted Mineral Water (DMW) or Reconstituted Water:

_____
_____
_____

Sample filtered (Y/N): ____ If yes, screen mesh size (μm): _____

Maximum percent of diluted mineral water in a sample: _____

Maximum holding time (hrs): _____ Storage temperature (C): _____

Aeration of sample anticipated: Yes ____ No ____

Acclimation Procedures

Minimum total acclimation period (days): _____ (hrs): _____

Minimum changeover period from holding water to diluent (hrs): _____

Minimum acclimation period in 100% diluent at test temperature (hrs): _____

Maximum mortality acceptable during acclimation: _____

Maximum test organism age at start of test (days): _____

Name and source of Standard Reference Toxicant to be used?

1. LC_{50}: _____ Acceptable range: _____

2. LC_{50}: _____ Acceptable range: _____

PART III: TEST CONDITIONS

Test Duration (days): _____

Test Type (check one): Static _____ Renewal (16 hr) _____
Renewal (24 hr) _____ Flow through _____

Test Vessels (material and size): _____

Test Liquid Volume (ml): _____ Test Temperature (C): _____

Are salinity adjustments anticipated (Y/N)? _____

If yes, type of salts or brine to be used: _____

Aeration procedures to be followed, if required: _____

_____

Photoperiod/illumination: _____ / _____

Physical/chemical parameters monitored and monitoring frequency: _____

_____

_____

Dilution Factor: 0.3 _____ 0.5 _____ Other (specify): _____

Is the permit limitation one of the test concentrations: Yes _____ No _____

Number of test concentration: _____

Number of organisms per concentration: _____

RMC ENVIRONMENTAL SERVICES, INC.

RMC #_____

TEST DESIGN:

Number of effluent concentrations (minimum of 5) _____
Number of Replicates per Test Concentration _____
Number of Test Organisms per Replicate _____
Exposure Volume (milliliters) _____
Test Chamber Type _____
Test Chamber Size _____
Deviations from the specified testing methodology _____
_____
_____
_____

EFFLUENT SAMPLING

Plant Sampling Location: _____

Type of Sample: Grab_____ 24-hr Composite_____ Other_____

Testing Location: On-Site_____ Remote Laboratory_____

Sample Collection:

Beginning Date: _____ Beginning Time: _____
Ending Date: _____ Ending Time: _____

If composite sample, number of grab samples in composite: _____
Interval between grab samples (minutes): _____

Number of samples collected during test period: _____
Maximum sample holding time (hours): _____

Description of effluent (color, clarity, odor, etc.) _____
_____
_____

Any adjustments to the effluent sample (salinity, pH, etc.) _____
_____

DILUTION WATER

Effluent receiving water: _____
Dilution water source: _____
Collection location: _____
Preparation dates: _____

RMC ENVIRONMENTAL SERVICES, INC.

RMC #_____

BIOMONITORING REPORT FORM - Chronic Toxicity Tests

DSN:_____

Facility Name: _____

Facility Location:_____

_____

Date of last SRT Test:_____ NOEC:_____

TEST START DATE:_____ TEST END DATE:_____

TEST TYPE AND RESULTS: (Check applicable test and fill in NOEC and LOEC):

	Test	NOEC	LOEC
_____Fathead minnow (Pimephales promelas)			
7 Day Larval Survival and Growth Test	Survival	_____	_____
	Growth	_____	_____
_____Cladoceran (Ceriodaphnia dubia)	Test	NOEC	LOEC
7 Day Survival and Reproduction Test	Survival	_____	_____
	Reproduction	_____	_____

QUALITY CONTROL SUMMARY:

Fathead Minnow Test Acceptability:

1) Control Mortality (%): _____
2) Average Dry Weight in the Controls: _____

Cladoceran Test Acceptability:

1) Control Mortality (%): _____
2) An average of 15 or more young per surviving female? _____
3) At least 60% of surviving females produce third brood in 7 days? _____

CERTIFICATION:

Accuracy of report certified by: _____

Bioassay Laboratory Manager Date

Figure 1. Data form for the fathead minnow larval survival and growth test. Routine chemical and physical determinations. (Continued).

Discharger: _____ Test Dates: _____
Location: _____ Analyst: _____

	Day							
Conc:	1	2	3	4	5	6	7	Remarks
Temp.								
D.O. Initial								
Final								
pH Initial								
Final								
Alkalinity								
Hardness								
Conductivity								
Chlorine								

	Day							
Conc:	1	2	3	4	5	6	7	Remarks
Temp.								
D.O. Initial								
Final								
pH Initial								
Final								
Alkalinity								
Hardness								
Conductivity								
Chlorine								

	Day							
Conc:	1	2	3	4	5	6	7	Remarks
Temp.								
D.O. Initial								
Final								
pH Initial								
Final								
Alkalinity								
Hardness								
Conductivity								
Chlorine								

Figure 1. Data form for the fathead minnow larval survival and growth test. Routine chemical and physical determinations.

Discharger: _____ Test Dates: _____
Location: _____ Analyst: _____

Control:	Day 1	2	3	4	5	6	7	Remarks
Temp.								
D.O. Initial								
Final								
pH Initial								
Final								
Alkalinity								
Hardness								
Conductivity								
Chlorine								

Conc:	Day 1	2	3	4	5	6	7	Remarks
Temp.								
D.O. Initial								
Final								
pH Initial								
Final								
Alkalinity								
Hardness								
Conductivity								
Chlorine								

Conc:	Day 1	2	3	4	5	6	7	Remarks
Temp								
D.O. Initial								
Final								
pH Initial								
Final								
Alkalinity								
Hardness								
Conductivity								
Chlorine								

7 DAY CHRONIC BIOASSAY
FATHEAD MINNOW LARVAE - GROWTH DATA

Discharger: _____ Start Date: _____
Location: _____ Analyst: _____
Test Number: _____

Effluent Conc	Repl	Pan Numb	DRY WEIGHT IN MG'S Pan	Larvae and Pan	Number of Larvae	Remarks
	A					
	B					
Control	C					
	D					
	A					
	B					
____	C					
	D					
	A					
	B					
____	C					
	D					
	A					
	B					
____	C					
	D					
	A					
	B					
____	C					
	D					
	A					
	B					
____	C					
	D					

Comments: _____

7 DAY CHRONIC BIOASSAY
FATHEAD MINNOW LARVAE - SURVIVAL DATA

Discharger: _____ Start Date: _____
Location: _____ Analyst: _____
Test Number: _____

Effluent Conc	Repl	# at Test Start	NUMBER OF FISH SURVIVING AT THE END OF EACH 24-HOUR PERIOD 1	2	3	4	5	6	7	Remarks
	A									
	B									
Control	C									
	D									
	A									
	B									
____	C									
	D									
	A									
	B									
____	C									
	D									
	A									
	B									
____	C									
	D									
	A									
	B									
____	C									
	D									
	A									
	B									
____	C									
	D									

Comments: _____

7 DAY CHRONIC BIOASSAY

CERIODAPHNIA - SURVIVAL AND REPRODUCTION DATA

Discharger: _____ Job/Test Number: _____

Location: _____ Analyst: _____

Start Date: _____

Effluent Conc	Repl	NUMBER OF CERIODAPHNIA YOUNG PRESENT AT THE END OF EACH 24-HOUR PERIOD 1	2	3	4	5	6	7	8	Remarks
	1									
	2									
	3									
	4									
	5									
	6									
	7									
	8									
	9									
	10									
	1									
	2									
	3									
	4									
	5									
	6									
	7									
	8									
	9									
	10									
	1									
	2									
	3									
	4									
	5									
	6									
	7									
	8									
	9									
	10									

7 DAY CHRONIC BIOASSAY

CERIODAPHNIA - SURVIVAL AND REPRODUCTION DATA

Discharger: _____ Job/Test Number: _____

Location: _____ Analyst: _____

Start Date: _____

Effluent Conc	Repl	NUMBER OF CERIODAPHNIA YOUNG PRESENT AT THE END OF EACH 24-HOUR PERIOD 1	2	3	4	5	6	7	8	Remarks
Data Entry By										
	1									
	2									
	3									
	4									
	5									
Control	6									
	7									
	8									
	9									
	10									
	1									
	2									
	3									
	4									
	5									
_____	6									
	7									
	8									
	9									
	10									
	1									
	2									
	3									
	4									
	5									
_____	6									
	7									
	8									
	9									
	10									

MID-WEST RESEARCH INSTITUTE

STANDARD OPERATING PROCEDURES

Golder Associates

Analysis of Mirex, Photomirex, and Kepone in Aqueous Samples
Site: Ruetgers-Nease, Salem, Ohio site

1. Parameters

The parameters to be measured are mirex, photomirex, and kepone.

2. Range of Measurement (Working Linear Range)

Mirex: 0.2 ng to 4.0 ng injected
Photomirex: 0.2 ng to 4.0 ng injected
Kepone: 1.0 ng to 8.0 ng injected

3. Detection Limits, Reporting Limits, and Units

The following are the reporting limits for one liter extracted, final extract volume of 200 uL, 2-uL injected, with no further dilutions of the extract. These reporting limits are two times greater than the method detection limits obtained during the method validation study (see section 20).

- Mirex: 0.00544 ug/L
- Photomirex: 0.0474 ug/L
- Kepone: 0.132 ug/L

The concentration of mirex, photomirex and kepone in water samples will be reported in ug/L using three significant figures.

4. Sample Matrix

The sample matrix is water.

5. Principle, Scope, and Application

Mirex, photomirex, and kepone are extracted with methylene chloride from the aqueous matrix and analyzed by gas chromatography/electron capture negative ion mass spectrometry (GC/ECNIMS) after optional screening using gas chromatography with electron capture, flame ionization, or electron impact mass spectrometric detection. Quantitation of mirex, photomirex, and kepone is done by isotope dilution using $^{13}C_8$-mirex as the internal standard. GC/ECNIMS, in conjunction with the use of stable-isotope-labeled standards, represents the best available technology to analyze for residue levels of chlorinated compounds. Interferences from nonhalogenated compounds are virtually nonexistent and possible interferences from other chlorinated compounds are believed to be limited because measurements are done at high mass fragment ions that are highly compound specific.

The GC/ECNIMS methodology involves the acquisition of limited mass range scans covering the three most abundant fragment ion clusters from mirex, photomirex, and $^{13}C_8$-mirex, and covering the two most abundant fragment ion clusters from kepone. One ion cluster from the mass spectrum of each of the compounds is designated as the quantitation ion cluster. The remaining ion cluster(s) are designated as confirmation clusters. The mass spectra for mirex, photomirex, kepone, and $^{13}C_8$-mirex are presented in appendix A. The ions used for quantitation and confirmation are listed in table A.1 of this appendix.

Quantitation is done using the abundances of the two most abundant ions from the quantitation cluster of each compound. Criteria for acceptable calibration and quantitation are defined for the quantitation cluster ion and also for the confirmation cluster ions. In case of interferences at the primary quantitation cluster, the ions from the confirmation cluster can then be used for quantitation purposes.

Confirmation criteria for mirex, photomirex, and kepone are defined from ion abundance ratio parameters and relative retention time (RRT) parameters.

6. Interferences and Corrective Actions

Interferences will be shown by isotope ratios that do not meet the criteria listed in section 14.3 (sample analysis).

If interferences are present at the m/z values of the quantitation ions and the confirmation ion clusters are indicative of the presence of the target compound and do not show interferences, the confirmation ions will be used for quantitation purposes. In these instances, acceptable gas chromatography/mass spectrometry (GC/MS) calibration (i.e., relative response factor [RRF] criteria) for the confirmation cluster ions has to be demonstrated. These data will be flagged (see section 18.3).

If interferences are present at the m/z values of the ions from the confirmation cluster, but the quantitation cluster ions clearly indicate the presence of the target compound, the concentration of the compound will be reported, but the data will be flagged (see section 18.3).

If interferences are present at the m/z values of the ions from both the quantitation and the confirmation clusters, the data will be flagged and the sample reanalyzed by GC/MS using electron ionization/selected ion monitoring (i.e., m/z 272 and m/z 274), to confirm the presence of mirex, photomirex, or kepone (see section 18.3).

7. Safety Precautions

Mirex, photomirex, and kepone should only be handled in a hood. Eye shields should be worn at all times. Avoid contact with skin, eyes, and clothing. Keep containers tightly closed in a cool, dry place. Store containers only with compatible chemicals.

A material safety data sheet (MSDS) is available for mirex, kepone, all reagent chemicals, and all solvents. All laboratory personnel should be thoroughly familiar with the laboratory safety manual and Hazard Communication Standard before undertaking any laboratory work.

8. Sample Size, Collection, Preservation, Handling, and Holding Times

The sample size is one gallon and samples are collected in half-gallon, amber glass containers with Teflon-lined caps.

No preservatives are added, but the collected samples must be iced or refrigerated at 4DEGC from the time of collection until the time of extraction.

All aqueous samples must be extracted within 5 days of receipt and completely analyzed within 40 days of extraction. The 40 day analytical holding time is established for operational reasons and for consistency with other methods. Previous data indicates that the extracts are stable and the results are valid up to and beyond 100 days.

9. Apparatus and Materials

All glassware will be acid washed and subsequently rinsed with deionized (DI) water, methanol, and methylene chloride before use.

The following apparatus and materials are needed for this method.

- Separatory funnel - 2-liter with Teflon stopcock.
- Graduated cylinder - 1-liter.
- Erlenmeyer flask - 500 mL.
- Filtering funnel - 75-mm top diameter.
- Pyrex glass wool - Must be baked out.
- Disposable 1-mL pipet.
- Concentrator tube, Kuderna-Danish (KD) - 10-mL graduated (Kontes K-570050-1025 or equivalent).
- Evaporator flask, KD - 500-mL (Kontes K-570001-0500 or equivalent). Attached to a concentrator tube with springs or clips.

- Snyder columns, KD - Three ball macro (Kontes K-503000-0121 or equivalent) and three ball micro.

- Vortexer - Fisher/Genie 2 or equivalent.

- Vials - 2-mL capacity with Teflon-lined screw cap and crimp cap suitable for GC autosampler.

- Boiling chips - Approximately 10/40 mesh.

- Water bath - Heated, with concentric ring over cover. It should be capable of temperature control (+/-2DEGC). The bath should be used in a hood. A sand bath should also be available.

- Top-loading balance - Capable of accurately weighing 0.01 g.

- Analytical balance - Capable of accurately weighing 0.0001 g.

- Spatula - Stainless steel or Teflon.

- GC screening - A capillary chromatography system suitable for splitless injection and possessing all required accessories such as syringes, analytical columns, gases, electron capture detector (ECD) or flame ionization detector, and recording device. A data system is recommended for data acquisition, integration, and output. An electron impact GC/MS system may also be used.

- Nitrogen evaporation device - Equipped with a water bath that can be maintained at 35-40DEGC (e.g., N-Evap by Organomation or Turbo-Vap by Zymark).

- Capillary column - Use one of the following or an approved substitute.

  - J&W 30-m DB-5, 0.25-mm ID or 0.32-mm ID, 0.25-um film thickness
  - Quadrex 25-m methyl phenyl (5%) silicone, 0.25-mm ID, 0.5-um film thickness

- Syringes - 10-uL, 25-uL, 50-uL, 100-uL, 500-uL and 1-mL, as appropriate for making injections, adding internal standards, and preparing sample dilutions.

- GC/MS/data system

  - GC - Suitable for splitless injection; it must be temperature programmable.

  - MS - Equipped with pulsed positive negative ion chemical ionization (PPNICI) device.

  - GC/MS interface - Direct coupling of the capillary column from the GC to the MS must be possible.

- Data system - Must be capable of continuous acquisition and storage of all mass spectra obtained through the analytical run. The software should be capable of repetitive scanning of sections of the mass spectrum of the target compounds, and of producing extracted ion profiles and integrating ion abundances.

10. Routine Preventative Maintenance

10.1 Gas chromatograph (GC)

- Daily replacement of the injection port septum, injection port ferrule, and injection port liner.

- Daily clipping of the GC column (at least 8 cm).

10.2 Mass spectrometer (MS)

- Routine cleaning of the ion source (at least every three months).

- Monthly check of the following items: mechanical pump oil (routinely changed every 6 months); turbo pump oil (routinely changed every 6 months); diffusion pump oil (routinely changed every 6 months); and filters on pumps (routinely changed every 6 months).

10.3 Computer

- Weekly cleaning of computer air filters.

- Monthly cleaning of computer boards.

11. Reagents and Calibration Standards

11.1 The following are reagents and calibration standards needed for this method.

- Reagent water - Carbon-filtered, house-distilled water. No interferences should be observed at or above the reporting limit of the parameters of interest.

- Methanol, methylene chloride - Pesticide quality or equivalent.

- Sodium sulfate - Anhydrous. Granulated for drying extracts and baked out.

- Concentrated hydrochloric acid (HCl).

- Isopropanol, toluene - High purity solvent.

11.2 All standards are prepared in toluene containing 2% isopropanol. The preparation of all standard solutions is documented in a standards logbook. All standard solutions are traceable and cross-referenced by a number. They are stored in a freezer in glass containers with Teflon-lined caps with the appropriate identification marked on the outside of the container. The volumes in the containers are marked on the outside of the containers with a felt tip pen.

11.2.1 Stock solutions

Stock solution number 1: mirex, 2 mg/mL
Stock solution number 2: photomirex, 2 mg/mL
Stock solution number 3: kepone, 2 mg/mL
Stock solution number 4: $^{13}C_8$-mirex, 20 ug/mL

These stock solutions are prepared by weighing out the neat compounds and dissolving them in the appropriate volume of solvent. Volumetric glassware is used. All stock solutions are stored in the freezer in glass containers with Teflon-lined caps.

11.2.2 Matrix spike solution/waters

Working solution number 1: mirex, 1,000 ng/mL
photomirex, 1,000 ng/mL
kepone, 5,000 ng/mL

This solution is prepared by combining appropriate aliquots of stock solution numbers 1 through 3 and the solvent. For example, preparation of 100 mL of working solution number 1 would be done by transferring 50 uL of stock solution number 1, 50 uL of stock solution number 2, and 250 uL of stock solution number 3 to a 100-mL volumetric flask that contained some solvent. More solvent would then be added to the flask until it reaches the 100-mL mark. After appropriate mixing, the contents of the flask would be transferred to a container with a Teflon-lined cap.

Matrix spike solution/waters: mirex, 100 ng/mL
photomirex, 100 ng/mL
kepone, 500 ng/mL

This solution is prepared by a 1:10 dilution of working solution number 1. One mL of this matrix spike solution/waters is added to the aqueous samples designated as matrix spikes before extraction.

11.2.3 Internal standard spiking solution/waters

$^{13}C_8$-Mirex, 100 ng/mL

This solution is prepared by a 1:200 dilution of stock solution number 4. One mL (100 ng) of this internal standard spiking solution/waters is added to the aqueous samples before analysis.

Respiking of the extracts with $^{13}C_8$-mirex before GC/MS analysis (if necessary) will be done by adding 5 uL of the $^{13}C_8$-mirex stock solution number 4 (100 ng) to the extract.

11.2.4 GC/MS calibration standards

Working solution number 2: mirex, 8 ug/mL
photomirex, 8 ug/mL

This solution is prepared by combining appropriate aliquots of stock solutions numbers 1 and 2. For example, preparation of 25 mL of working solution number 2 would involve transferring 100 uL of stock solution number 1 and 100 uL of stock solution to a 25-mL volumetric flask that contained some solvent. More solvent would then be added to the flask until it reaches the 25-mL mark. After appropriate mixing, the contents of the flask would be transferred to a container with a Teflon-lined cap.

Working solution number 3: kepone, 40 ug/mL

This solution is prepared by a 1:50 dilution stock solution number 3.

GC/MS calibration standards at concentrations listed in section 12.2 are prepared according to the following chart.

Compound	GC/MS No. 1 (in uL)	GC/MS No. 2 (in uL)	GC/MS No. 3 (in uL)	GC/MS No. 4 (in uL)	GC/MS No. 5 (in uL)
Mirex/Photomirex Working Sol. No. 2	25	50	125	250	500
Kepone Working Sol. No. 3	25	50	100	150	200
Toluene with 2% isopropanol	900	850	725	550	250
$^{13}C_8$-Mirex Stock Solution No. 4	50	50	50	50	50
Total Volume	1,000	1,000	1,000	1,000	1,000

11.3 Mirex and kepone are purchased from Chemserve, P.O. Box 3108, West Chester, Pennsylvania, 19381, from Crescent Chemical Co., Inc., 1324 Motor Parkway, Hauppauge, New York, 11788, or from Supelco, Inc., Supelco Park, Bellefonte, Pennsylvania, 16823-0048. $^{13}C_8$-Mirex is purchased from Cambridge Isotope Laboratories, 20 Commerce Way, Woburn, Massachusetts, 01801. Photomirex is obtained from the Environmental Protection Agency (EPA) Research Triangle Institute.

12. Calibration Procedures

12.1 Instrument calibration

For perfluorotributylamine (PFTBA), the instrument calibration must be manually (hardware) verified and adjusted as necessary at the start of every 24 hours of operation. Ion abundance guidelines are noted in the following chart. Adherence to these guidelines may make instrument calibration easier.

m/z	Percent Base
283	>1
312	>1
414	>10
557	>15
595	>60
596	5-10 of 633
633	Base peak
634	8-13 of 633

12.2 Initial calibration

An acceptable initial GC/MS calibration will meet the following criteria.

- RRTs for mirex, photomirex, and kepone relative to $^{13}C_8$-mirex of +/-0.06 RRT units from the most recent GC/MS calibration standard.

- Relative ion abundance criteria for mirex, $^{13}C_8$-mirex, and photomirex (for the two most abundant ions in the quantitation cluster and for the two most abundant ions in the confirmation cluster) of +/-20% from the theoretical ratio.

- Relative ion abundance criteria for kepone for the two most abundant ions in the quantitation cluster of +/-20% from the theoretical ratio and for the two most abundant ions in the confirmation cluster of +/-35% from the theoretical ratio.

The following chart describes this in further detail.

Analyte	Quantitation Ions (m/z)	Control Limits (m/z Ratios)	Confirmation Ions (m/z)	Control Limits (m/z Ratios)
Mirex	368/370	0.83-1.25	402/404	0.71-1.07
$^{13}C_8$-Mirex	376/378	0.83-1.25	410/412	0.71-1.07
Photomirex	368/370	0.83-1.25	402/404	0.71-1.07
Kepone	383/385	0.83-1.25	418/420	0.58-1.20

- Mass spectral sensitivity for mirex, photomirex, and kepone as defined by the presence at signal-to-noise (S/N) levels of greater than 2.5 of the following ions for the standard that is used for continuing calibration (1 ng mirex, 1 ng photomirex, and 4 ng kepone). The specific ions are noted below.

  - Mirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 437, 439, 441

  - Photomirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 439

  - Kepone: m/z 381, 383, 385, 387; m/z 416, 418, 420, 422

- Mass spectral sensitivity for $^{13}C_8$-mirex as defined by the presence at S/N levels of greater than 5 for m/z 376, 378 and m/z 410, 412.

The response factor criteria are as follows:

- Acceptance criteria of average RRFs (RRF_{ave}) for mirex, photomirex, and kepone are 0.05 or larger.

- Acceptance criteria for the percent relative standard deviation (%RSD) of RRFs for mirex and photomirex relative to $^{13}C_8$-mirex are within 30% for the ions used for quantitation.

- Acceptance criteria for the %RSD of the RRF for kepone relative to $^{13}C_8$-mirex are within 40% for the ions used for quantitation.

The following chart describes the GC/MS calibration standards based on 1-mL aliquots with 1 uL injected.

Compound	Conc. No. 1	Conc. No. 2	Conc. No. 3	Conc. No. 4	Conc. No. 5
Mirex and Photomirex:					
ng/mL	200	400	1,000	2,000	4,000
ng/uL injected	0.2	0.4	1.0	2.0	4.0
Kepone:					
ng/mL	1,000	2,000	4,000	6,000	8,000
ng/uL injected	1.0	2.0	4.0	6.0	8.0
$^{13}C_8$-Mirex:					
ng/mL	1,000	1,000	1,000	1,000	1,000
ng/uL	1.0	1.0	1.0	1.0	1.0

12.3 Continuing calibration

The continuing calibration standard contains 1 ug/uL of mirex, 1 ug/uL of photomirex, 1 ug/uL of $^{13}C_8$-mirex, and 4 ug/uL of kepone. An acceptable GC/MS continuing calibration will meet the following criteria.

- RRTs for mirex, photomirex, and kepone relative to $^{13}C_8$-mirex of +/-0.06 RRT units from the most recent GC/MS calibration standard.

- Relative ion abundance criteria for mirex, $^{13}C_8$-mirex, and photomirex (for the two most abundant ions in the quantitation cluster and for the two most abundant ions in the confirmation cluster) of +/-20% from the theoretical ratio.

- Relative ion abundance criteria for kepone for the two most abundant ions in the quantitation cluster of +/-20% from the theoretical ratio and for the two most abundant ions in the confirmation cluster of +/-35% from the theoretical ratio.

  The following chart describes this in further detail.

Analyte	Quantitation Ions (m/z)	Control Limits (m/z Ratios)	Confirmation Ions (m/z)	Control Limits (m/z Ratios)
Mirex	368/370	0.83-1.25	402/404	0.71-1.07
$^{13}C_8$-Mirex	376/378	0.83-1.25	410/412	0.71-1.07
Photomirex	368/370	0.83-1.25	402/404	0.71-1.07
Kepone	383/385	0.83-1.25	418/420	0.58-1.20

- Mass spectral sensitivity for mirex, photomirex, and kepone as defined by the presence at S/N levels of greater than 2.5 of the following ions. The specific ions are noted in the following items.

  - Mirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 437, 439, 441

  - Photomirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 439

  - Kepone: m/z 381, 383, 385, 387; m/z 416, 418, 420, 422

- Mass spectral sensitivity for $^{13}C_8$-mirex as defined by the presence at S/N levels of greater than 5 for m/z 376, 378 and m/z 410, 412.

The response factor criteria are as follow.

- Acceptance criteria of RRFs for mirex, photomirex, and kepone are 0.05 or larger.

- Acceptance criteria for the percent difference (%D) of RRFs for mirex and photomirex relative to $^{13}C_8$-mirex are between -30% and +30% compared to the average RRF from the initial calibration.

- Acceptance criteria for the %D of the RRF for kepone relative to $^{13}C_8$-mirex are less than ±40% for the ions used for quantitation. After any continuing calibration where %D acceptance criteria for Kepone is not met, the laboratory must analyze the low level standard from the initial five level calibration. Kepone must be detected in the low level standard to ensure that adequate sensitivity for Kepone has been maintained over the entire calibration range. Mass spectral sensitivity for Kepone is defined by the presence of S/N ratios of greater than 2.5 for the following ions for Kepone: m/z 381, 383, 385, 387, 416, 418, 420, and 422.

- The absolute retention time of $^{13}C_8$-Mirex shall not change more than ± 3 seconds from the continuing calibration.

13. Sample Preparation

All samples are mixed by shaking the bottle.

13.1 Measure 1 liter of sample in a 1-liter graduated cylinder and add to a 2-liter separatory funnel.

13.2 For blanks, pour one liter of carbon-filtered water into the separatory funnel. For matrix-specific quality control (QC) samples, measure one liter of the appropriate sample required for each QC sample into a separatory funnel. The matrix spike and matrix spike duplicate (MS/MSD) will be done using 100 ng of mirex, 100 ng of photomirex, and 500 ng of kepone.

13.3 Add approximately 0.5 mL of concentrated HCl.

13.4 Add 100 ng of $^{13}C_8$-mirex (internal standard).

13.5 Add 60 mL of methylene chloride and shake for 2 minutes with frequent ventilation.

13.6 Allow the layers to separate for 10 minutes.

13.7 Drain the methylene chloride into an Erlenmeyer flask.

13.8 Repeat steps 13.5 through 13.7 two more times.

13.9 After the third extraction, pour the combined extract into a flask through a funnel containing anhydrous sodium sulfate. Rinse the sodium sulfate with an additional 50 mL of methylene chloride.

13.10 Add 200 uL of toluene to each flask.

13.11 Add a boiling chip to each flask and concentrate the extract to 5 mL in a water bath at 85-90DEGC using the KD concentrator equipped with a macro Snyder column. Replace the macro Snyder column with a micro Snyder column and concentrate further to 1 mL.

13.12 Remove the KD apparatus and blow down the extract to 200 uL under a stream of nitrogen.

13.13 Add 4 uL of isopropanol and mix well by vortexing. Store the extract in a crimp-top vial in the freezer.

13.14 Screen the extract by GC/ECD, GC/FID, or GC/MS as outlined in appendix B

13.15 If the extract is clean, inject 2 uL for GC/MS analysis.

13.16 If excess contamination is present in the extract, determine the dilution factor and dilute the extract using toluene containing 2% isopropanol. Always mix this well. From a 200-uL final extract volume, inject 2 uL for GC/MS analysis.

13.17 If $^{13}C_8$-mirex is diluted out and cannot be used as the internal standard anymore, respike the extract (200 uL) with 100 ng of $^{13}C_8$-mirex, mix well, and inject 2 uL for GC/MS analysis.

14. Analytical Measurement

14.1 Suggested instrument set up for GC/MS

- GC: HP 5890, equipped with HP 7673A autosampler or equivalent.

- Column: As specified in section 9.

- GC temperature program:
  Initial Temperature: 120 DEGC
  Initial Time: 3 Minutes
  First Ramp: 12 DEG C/ Minute
  Temperature 1: 250 DEG C
  Hold: 0.1 Minutes
  Second Ramp: 20 DEG C/ Minute
  Final Temperature: 310 DEG C
  Final Time: 2 Minutes

- Temperature GC injection port: 240DEGC.

- MS: Finnigan-MAT 4615B or Finnigan-MAT 4500 MS equipped with a PPNICI option and automated by a SuperIncos data system.

- Temperature ion source: 100DEGC (ion source block).

- Temperature transfer line: 280DEGC.

- Multiple ion detection (MID) descriptors: See appendix C.

14.2 File naming conventions

Initial calibration GC/MS analyses for mirex, photomirex, and kepone are specified in the following chart.

File Name*	GC/MS Calibration Standard
2SMPK01	No. 1 (0.2 ng/uL of mirex and photomirex, 1.0 ng/uL of kepone)
2SMPK02	No. 2 (0.4 ng/uL of mirex and photomirex, 2.0 ng/uL of kepone)
2SMPK03	No. 3 (1.0 ng/uL of mirex and photomirex, 4.0 ng/uL of kepone)
2SMPK04	No. 4 (2.0 ng/uL of mirex and photomirex, 6.0 ng/uL of kepone)
2SMPK05	No. 5 (4.0 ng/uL of mirex and photomirex, 8.0 ng/uL of kepone)

*The "2" prefix denotes the instrument number.

A continuing calibration GC/MS analysis is specified by the instrument number, followed by MPK (to denote a standard for mirex, photomirex, and kepone), and the date of the analysis. For example, 2MPK072589 is the file name for the standard on Finnigan MS number 2, analyzed on July 25, 1989.

The file names for samples are designated by the six-digit Erco sample number derived from the booking log number and the "CI" suffix. The file names can contain the following additional suffixes.

- DU for duplicates
- MS for matrix spikes
- SD for matrix spike duplicates
- R for reinjections
- X for reextracts
- D for dilutions

For example, 202701CI is the file name for sample number 01 pertaining to project 2027 that is logged for the analysis of mirex, photomirex, and kepone.

The nomenclature for blanks is determined using the date of extraction, with a suffix denoting the appropriate extraction technique. For example, 25JUL89F12CI is the file name for the first blank extracted by separatory funnel on July 25, 1989, and analyzed on Finnigan MS number 2.

14.3 Sample analysis

Confirmation of mirex, photomirex, and kepone in the samples will be done according to the following criteria.

- RRT of mirex, photomirex and kepone relative to $^{13}C_8$-mirex within +/-0.06 RRT units from the most recent GC/MS calibration standard.

- Relative ion abundance criteria for mirex, $^{13}C_8$-mirex, and photomirex (for the two most abundant ions in the quantitation cluster and for the two most abundant ions in the confirmation cluster) of +/-20% from the theoretical ratio.

- Relative ion abundance criteria for kepone for the two most abundant ions in the quantitation cluster of +/-20% from the theoretical ratio and for the two most abundant ions in the confirmation cluster of +/-35% from the theoretical ratio.

  Further details are listed in the section 12 chart.

- The presence at S/N levels greater than 2.5 of the following ions.

  - Mirex: m/z 366, 368, 370, 372 (quantitation cluster); m/z 400, 402, 404, 406 (confirmation cluster); and m/z 437, 439, 441 (confirmation cluster).

  - Photomirex: m/z 366, 368, 370, 372 (quantitation cluster) and m/z 400, 402, 404, 406 (confirmation cluster).

  - Kepone: m/z 381, 383, 385, 387 (quantitation cluster) and m/z 418, 420 (confirmation cluster).

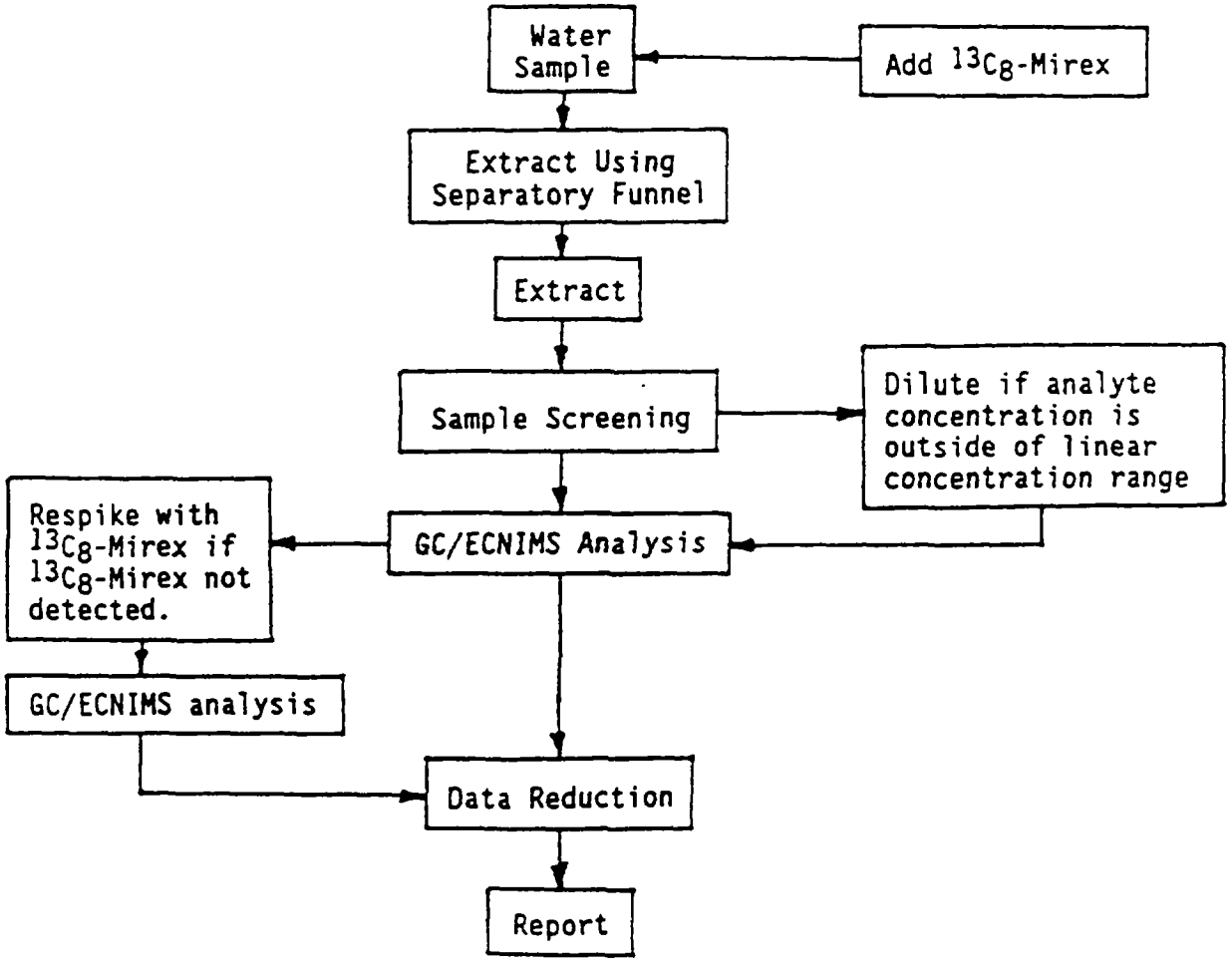
For data in which ion abundance criteria are not met (in particular, for the confirmation clusters) or data in which not all specified ions in the clusters are present, but, in the judgement of the analyst, the compound is present, the reporting of the compound will be qualified (in addition, see section 6 [interferences] and section 18.3).

14.4 Quantitation of the compounds

The quantitation methods used are in accordance with EPA Contract Laboratory Program (CLP) protocols for semivolatile organics with several modifications appropriate for the analytes and matrix. The summed abundances of two ions are used for quantitation. The response factors from the continuing calibrations are used. The generalized equation is presented in section 16.

The required criteria for the use of $^{13}C_8$-mirex as an internal standard are the the presence of the two major ions used for quantitation at S/N greater than 5.

15. Flow Chart



16. Data Treatment

16.1 The equation for quantitation of the compounds is as follows.

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

where:

C_x = concentration of the compound to be measured (ng/uL);
C_{is} = concentration of the internal standard (ng/uL);
A_x = summed areas of the quantitation ions of the compound to be measured;
A_{is} = summed areas of the quantitation ions of the internal standard; and
RRF = relative response factor from the continuing calibration standard.

16.2 The correction for contribution of mirex at m/z values measured for $^{13}C_8$-mirex will be done as follows.

16.2.1 Quantitation cluster

- Calculate 3.22% from m/z 368, subtract from m/z 376
- Calculate 0.351% from m/z 368 subtract from m/z 378

16.2.2 Confirmation cluster

- Calculate 6.65% from m/z 404, subtract from m/z 410
- Calculate 1.08% from m/z 404, subtract from m/z 412

16.3 Calculations required to verify automated data reduction are as follow.

16.3.1 To determine the RRF, use the following equation.

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

where:

A_x = summed areas of the quantitation ions for the compound to be measured;
A_{is} = summed areas of the quantitation ions for the specific internal standard;
C_{is} = concentration of the internal standard (ng/uL); and
C_x = concentration of the compound to be measured (ng/uL).

16.3.2 *The average RRF is determined by following equation.*

$$- RRF_{ave} = \frac{RRF_{conc1}+RRF_{conc2}+RRF_{conc3}+RRF_{conc4}+RRF_{conc5}}{5}$$

where:

RRF_x = RRF of a compound in each of the calibration solutions.

16.3.3 Percent relative standard deviation is determined by the following equation.

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

where:

RSD = relative standard deviation;
SD = standard deviation of initial RRFs (per compound); and
RRF_{ave} = mean of initial RRFs (per compound).

SD is determined with the following equation.

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

where:

$\bar{X}$ = mean of initial RRF (per compound); and
N = number of measurements.

16.3.4 Percent difference (%D) from average RRF is determined with the following equation.

$$- \%D = \frac{RRF_I - RRF_C}{RRF_I} \times 100$$

where:

RRF_I = average RRF from initial calibration; and
RRF_C = RRF from current calibration check standard.

16.3.5 Percent recovery is determined with the following equation.

$$- MSPR = \frac{SSR - SR}{SA} \times 100$$

where:

MSPR = matrix spike percent recovery;
SSR = spike sample results;
SR = sample result; and
SA = spike added from spiking mix.

16.3.6 Relative percent difference (RPD) is determined with the following equation.

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

where:

RPD = relative percent difference;
D_1 = first sample value; and
D_2 = second sample value (duplicate).

16.3.7 To determine the analyte concentration in an aqueous sample, use the following equation.

$$\text{- Concentration (ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_o)(V_i)}$$

where:

A_x = summed areas of the quantitation ions for the compound to be measured;
A_{is} = summed areas of the quantitation ions for the internal standard;
I_s = amount of internal standard injected in nanograms (ng);
RRF = RRF for the compound being measured, determined from the continuing calibration standard;
V_o = volume of water extracted in milliliters (mL);
V_i = volume of extract injected in microliters (uL); and
V_t = volume of total extract in microliters, taking dilutions into account. For example, a 1:10 dilution of a 1-mL extract will mean V_t = 10,000 uL.

17. Data Deliverables

The following items are the data deliverables that are applicable to this method.

- Case narrative.

- Perfluorotributylamine (PFTBA, FC-43) summary.

- Summary of initial calibration and continuing calibration check results.

- Summary of QC sample analyses.

- Laboratory blank compliance form.

- Summarized analytical results page.

- Summary sheets for the isotope ratios of the two most abundant ions in the quantitation cluster and the two most abundant ions in the in the major confirmation cluster for mirex, photomirex, $^{13}C_8$-mirex, and kepone.

- Logbook pages for extraction and GC/MS analysis.

- Raw data including the following items.

  - Work sheets, including sample calculations

  - Screening chromatograms using GC/FID, GC/ECD, GC/EI/MS, or GC/ECNIMS used to choose appropriate dilutions for analysis

  - Reconstructed ion chromatograms (RIC) at appropriate retention time intervals with mass chromatograms of the ions used for quantitation and confirmation. The following list describes the specific ions.

    - Mirex and photomirex: m/z 368, m/z 370, m/z 402, m/z 404
    - Kepone: m/z 383, m/z 385, m/z 418, m/z 420
    - $^{13}C_8$-Mirex: m/z 376, m/z 378, m/z 410, m/z 412

  - Listing of areas at appropriate m/z values used for quantitation; areas can be included on main chromatograms. This includes corrected areas for $^{13}C_8$-mirex.

  - Mass spectrum for each analyte (top of RIC peak).

18. Quality Control Requirements

18.1 Laboratory method blanks

No mirex, photomirex, or kepone will be present in the method blank above the reporting limit.

If a laboratory method blank exceeds these criteria, the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) must be reextracted and reanalyzed.

18.2 Precision and accuracy

The following recovery ranges and RPDs are calculated from method validation data (section 20). Four replicate analyses using spiking levels of 100 ng of mirex, 100 ng of photomirex, and 200 ng of kepone were done. The recovery range was defined by the mean +/-3 standard deviations; the RPD was defined as 3 times the standard deviation. RPDs that were calculated to be less than 30 were set at 30.

- Mirex - The recovery range is 87-100% and the RPD is less than 30%.

- Photomirex - The recovery range is 80-101% and the RPD is less than 30%.

- Kepone - The recovery range is 19-83% and the RPD is less than 32%.

18.3 Qualifier codes

One or more of the following qualifiers can be used.

- J = Result is detected below the reporting limit and is an estimated concentration.

- z = Presence of the compound is strongly indicated but the ion abundance ratio criteria are not met for the quantitation cluster ions.

- x = Presence of the compound is strongly indicated but the ion abundance ratio criteria are not met for the confirmation cluster ions.

- y = Presence of the compound is strongly indicated but not all specified ions in the clusters are present.

- k = Quantitation done using confirmation cluster ions.

- D = Compound identified at secondary dilution.

- E = Concentration exceeds calibration range.

- F = Reported value estimated due to an interference.

19. References

The following reference was used in developing this SOP.

- DeLeon, I.R., Remele, P.C., Miles, S.K., and Laseter, J.L. 1980. "Glass capillary columns in the gas chromatographic and gas chromatographic-mass spectrometric determination of trace levels of kepone in natural waters," Anal. Lett. 13(A6):503-515.

20. Method Validation Data

The method validation report was submitted in December 1989, by Enseco - Erco Laboratory.

Appendix B

A.1 Screening of Extracts

The screening of extracts will be done by capillary column chromatography using splitless injection on a DB-5 or equivalent column. The detector can be an electron capture detector, a flame ionization detector, or a mass spectrometer depending on the judgement of the analyst. The suggested temperature program is the same as the program for final analysis.

A.1.1 Inject one microliter of a quantitative calibration standard containing mirex, photomirex, and kepone. If possible this calibration standard should be identical to the highest concentration standard for the GC/ECNIMS analysis.

A.1.2 Inject one microliter of each extract to be screened and compare the response to that of the calibration standard. Estimate the concentration of mirex, photomirex, and kepone in the extract using a single point response factor obtained from the calibration standard.

A.1.3 Samples will be analyzed using selected ion monitoring. The 272 m/z ion will be the primary ion for quantitation.

Using the estimated concentration, make a dilution of the extract that brings the concentrations of mirex, photomirex, and kepone into the GC/ECNIMS calibration range. Dilutions should be made such that the estimated concentration is toward the upper end of the calibration range. Peaks in the samples at retention times that do not correspond to mirex, photomirex, and kepone will be evaluated on a case by case basis.

If necessary in the judgement of the analyst, an additional screen of the diluted extract may be made.

Analysis of Mirex, Photomirex, and Kepone in Soil Samples
Ruetgers-Nease, Salem, Ohio site

1. Parameters

The parameters to be measured are mirex, photomirex, and kepone.

2. Range of Measurement (Working Linear Range)

Mirex: 0.2 ng to 4.0 ng injected
Photomirex: 0.2 ng to 4.0 ng injected
Kepone: 1.0 ng to 8.0 ng injected

3. Detection Limits, Reporting Limits, and Units

The following are the reporting limits for 30 g (wet weight) of sample extracted, a final extract volume of 1 mL, 1-uL injected, with no further dilutions of the extract. These reporting limits are two times greater than the method detection limits obtained during the method validation study (see section 20).

- Mirex: 18.5 ug/Kg
- Photomirex: 20.4 ug/Kg
- Kepone: 68.0 ug/Kg

The concentration of mirex, photomirex and kepone in soil/sediment samples will be reported in ug/Kg (dry weight) using three significant figures.

4. Sample Matrix

The sample matrix is soil/sediment.

5. Principle, Scope, and Application

Mirex, photomirex, and kepone are extracted with methylene chloride/acetone from the solid matrix and analyzed by gas chromatography/electron capture negative ion mass spectrometry (GC/ECNIMS) after optional screening using gas chromatography with flame ionization, electron capture, or electron impact mass spectrometric detection. Quantitation of mirex, photomirex, and kepone is done by isotope dilution using $^{13}C_8$-mirex as the internal standard. GC/ECNIMS, in conjunction with the use of stable-isotope-labeled standards,

represents the best available technology to analyze for residue levels of chlorinated compounds. Interferences from nonhalogenated compounds are virtually nonexistent and possible interferences from other chlorinated compounds are believed to be limited because measurements are done at high mass fragment ions that are highly compound specific. The GC/ECNIMS methodology involves the acquisition of limited mass range scans covering the three most abundant fragment ion clusters from mirex, photomirex, and $^{13}C_8$-mirex, and covering the two most abundant fragment ion clusters from kepone. One ion cluster from the mass spectrum of each of the compounds is designated as the quantitation ion cluster. The remaining ion cluster(s) are designated as confirmation clusters. The mass spectra for mirex, photomirex, kepone, and $^{13}C_8$-mirex are presented in appendix A. The ions used for quantitation and confirmation are listed in table A.1 of this appendix.

Quantitation is done using the abundances of the two most abundant ions from the quantitation cluster of each compound. Criteria for acceptable calibration and quantitation are defined for the quantitation cluster ion and also for the confirmation cluster ions. In case of interferences at the primary quantitation cluster, the ions from the confirmation cluster can then be used for quantitation purposes.

Confirmation criteria for mirex, photomirex, and kepone are defined from ion abundance ratio parameters and relative retention time (RRT) parameters.

6. Interferences and Corrective Actions

Interferences will be shown by isotope ratios that do not meet the criteria listed in section 14.3 (sample analysis).

If interferences are present at the m/z values of the quantitation ions and the confirmation ion clusters are indicative of the presence of the target compound and do not show interferences, the confirmation ions will be used for quantitation purposes. In these instances, acceptable gas chromatography/mass spectrometry (GC/MS) calibration (i.e., relative response factor [RRF] criteria) for the confirmation cluster ions has to be demonstrated. These data will be flagged (see section 18.3).

If interferences are present at the m/z values of the ions from the confirmation cluster, but the quantitation cluster ions clearly indicate the presence of the target compound, the concentration of the compound will be reported, but the data will be flagged (see section 18.3).

If interferences are present at the m/z values of the ions from both the quantitation and the confirmation clusters, the data will be flagged and the sample reanalyzed by GC/MS using electron ionization/selected ion monitoring (i.e., m/z 272 and m/z 274), to confirm the presence of mirex, photomirex, or kepone (see section 18.3).

7. Safety Precautions

Mirex, photomirex, and kepone should only be handled in a hood. Eye shields should be worn at all times. Avoid contact with skin, eyes, and clothing. Keep containers tightly closed in a cool, dry place. Store containers only with compatible chemicals.

A material safety data sheet (MSDS) is available for mirex, kepone, all reagent chemicals, and all solvents. All laboratory personnel should be thoroughly familiar with the laboratory safety manual and Hazard Communication Standard before undertaking any laboratory work.

8. Sample Size, Collection, Preservation, Handling, and Holding Times

The sample size is 100 g and samples are collected in wide-mouth, amber glass jars with Teflon liner. Samples are stored at 4 C until extracted. Samples must be extracted within 10 days of receipt and analyzed within 40 days of extraction. The 40 day analytical holding time is established for operational reasons and for consistency with other methods. Previous results indicate that the extracts are stable and the results are valid up to and beyond 100 days after extraction.

No preservatives are added.

To determine the percent moisture, weight 5 to 10 g of sample into a tared weighing dish. Record the wet weight of the sample and allow it to dry overnight at 105°C. Reweigh the sample and record the dry weight of the sample. Calculate the percent moisture using the equation listed in section 16.3.7.

9. Apparatus and Materials

All glassware will be acid washed and subsequently rinsed with deionized (DI) water, methanol, and methylene chloride before use.

The following apparatus and materials are needed for this method.

- Disposable glass jars - 250 mL.
- Erlenmeyer flask - 500 mL.
- Filtering funnel - 75-mm top diameter.
- Pyrex glass wool - Must be baked out.
- Disposable 1-mL pipet.
- Concentrator tube, Kuderna-Danish (KD) - 10-mL graduated (Kontes K-570050-1025 or equivalent).
- Evaporator flask, KD - 500-mL (Kontes K-570001-0500 or equivalent). Attached to a concentrator tube with springs or clips.
- Snyder columns, KD - Three ball macro (Kontes K-503000-0121 or equivalent) and three ball micro.
- Vortexer - Fisher/Genie 2 or equivalent.
- Vials - 2-mL capacity with Teflon-lined screw cap and crimp cap suitable for GC autosampler.
- Boiling chips - Approximately 10/40 mesh.
- Water bath - Heated, with concentric ring over cover. It should be capable of temperature control (±2DEGC). The bath should be used in a hood. A sand bath should also be available.
- Top-loading balance - Capable of accurately weighing 0.01 g.
- Analytical balance - Capable of accurately weighing 0.0001 g.
- Spatula - Stainless steel or Teflon.
- Shaker - Lab-Line orbit shaker or equivalent.
- GC - A capillary chromatography system for sample screening, suitable for splitless injection and possessing all required accessories such as syringes, analytical columns, gases, electron capture detector (ECD) or flame ionization detector (FID), and recording device. A data system is recommended for data acquisition, integration, and output. An electron impact GC/MS system may also be used.

- Nitrogen evaporation device - Equipped with a water bath that can be maintained at 35-40DEGC (e.g., N-Evap by Organomation, or Turbo-Vap by Zymark).

- Capillary column - Use one of the following or an approved substitute.

  - J&W 30-m DB-5, 0.25-mm ID or 0.32-mm ID, 0.25-um film thickness
  - Quadrex 25-m methyl phenyl (5%) silicone, 0.25-mm ID, 0.5-um film thickness

- Syringes - 10-uL, 25-uL, 50-uL, 100-uL, 500-uL and 1-mL, as appropriate for making injections, adding internal standards, and preparing sample dilutions.

- GC/MS/data system

  - GC - Suitable for splitless injection; it must be temperature programmable.

  - MS - Equipped with pulsed positive negative ion chemical ionization (PPNICI) device.

  - GC/MS interface - Direct coupling of the capillary column from the GC to the MS must be possible.

  - Data system - Must be capable of continuous acquisition and storage of all mass spectra obtained through the analytical run. The software should be capable of repetitive scanning of sections of the mass spectrum of the target compounds, and of producing extracted ion profiles and integrating ion abundances.

10. Routine Preventative Maintenance

10.1 Gas chromatograph (GC)

- Daily replacement of the injection port septum, injection port ferrule, and injection port liner.

- Daily clipping of the GC column (at least 8 cm).

10.2 Mass spectrometer (MS)

- Routine cleaning of the ion source (at least every three months).

- Monthly check of the following items: mechanical pump oil (routinely changed every 6 months); turbo pump oil (routinely changed every 6 months); diffusion pump oil (routinely changed every 6 months); and filters on pumps (routinely changed every 6 months).

10.3 Computer

- Weekly cleaning of computer air filters.

- Monthly cleaning of computer boards.

11. Reagents and Calibration Standards

11.1 The following are the reagents and calibration standards needed for this method.

- Reagent water - Carbon-filtered, house-distilled water. No interferences should be observed at or above the reporting limit of the parameters of interest.

- Acetone, methylene chloride - Pesticide quality or equivalent.

- Toluene, isopropanol - High purity solvent.

- Sodium sulfate - Anhydrous. Granulated for drying extracts (powdered for extracting soil samples) and baked out.

11.2 All standards are prepared in toluene containing 2% isopropanol. The preparation of all standard solutions is documented in a standards logbook. All standard solutions are traceable and cross-referenced by a number. They are stored in a freezer in glass containers with Teflon-lined caps with the appropriate identification marked on the outside of the container. The volumes in the containers are marked on the outside of the containers with a felt tip pen.

11.2.1 <u>Stock solutions</u>

Stock solution number 1: mirex, 2 mg/mL
Stock solution number 2: photomirex, 2 mg/mL
Stock solution number 3: kepone, 2 mg/mL
Stock solution number 4: $^{13}C_8$-mirex, 20 ug/mL

These stock solutions are prepared by weighing out the neat compounds and dissolving them in the appropriate volume of solvent. Volumetric glassware is used. All stock solutions are stored in the freezer in glass containers with Teflon-lined caps.

11.2.2 Matrix spike solution/soils

Mirex, 1,000 ng/mL
Photomirex, 1,000 ng/mL
Kepone, 5,000 ng/mL

This solution is prepared by combining appropriate aliquots of stock solution numbers 1 through 3 and the solvent. For example, preparation of 100 mL of the matrix spike solution/soils would be done by transferring 50 uL of stock solution number 1, 50 uL of stock solution number 2, and 250 uL of stock solution number 3 to a 100-mL volumetric flask that contained some solvent. More solvent would then be added to the flask until it reaches the 100-mL mark. After appropriate mixing, the contents of the flask would be transferred to a container with a Teflon-lined cap.

One mL of this matrix spike solution/soils is added to the soil samples designated as matrix spikes before extraction.

11.2.3 Internal standard spiking solution/soils

$^{13}C_8$-Mirex, 1,000 ng/mL

This solution is prepared by a 1:20 dilution of stock solution number 4. One mL (1,000 ng) of this internal standard spiking solution/soils is added to the soil samples before analysis.

Respiking of the extracts with $^{13}C_8$-mirex before GC/MS analysis (if necessary) will be done by adding 50 uL of the $^{13}C_8$-mirex stock solution number 4 (1,000 ng) to the extract.

11.2.4 GC/MS calibration standards

Working solution number 2: mirex, 8 ug/mL
photomirex, 8 ug/mL

This solution is prepared by combining appropriate aliquots of stock solutions numbers 1 and 2. For example, preparation of 25 mL of working solution number 2 would involve transferring 100 uL of stock solution number 1 and 100 uL of stock solution to a 25-mL volumetric flask that contained some solvent. More solvent would then be added to the flask until it reaches the 25-mL mark. After appropriate mixing, the contents of the flask would be transferred to a container with a Teflon-lined cap.

Working solution number 3: kepone, 40 ug/mL

This solution is prepared by a 1:50 dilution stock solution number 3.

GC/MS calibration standards at concentrations listed in section 12.2 are prepared according to the following chart.

Compound	GC/MS No. 1 (in uL)	GC/MS No. 2 (in uL)	GC/MS No. 3 (in uL)	GC/MS No. 4 (in uL)	GC/MS No. 5 (in uL)
Mirex/Photomirex Working Sol. No. 2	25	50	125	250	500
Kepone Working Sol. No. 3	25	50	100	150	200
Toluene with 2% isopropanol	900	850	725	550	250
$^{13}C_8$-Mirex Stock Solution No. 4	50	50	50	50	50
Total Volume	1,000	1,000	1,000	1,000	1,000

11.3 Mirex and kepone are purchased from Chemserve, P.O. Box 3108, West Chester, Pennsylvania, 19381, from Crescent Chemical Co., Inc., 1324 Motor Parkway, Hauppauge, New York, 11788, or from Supelco, Inc., Supelco Park, Bellefonte, Pennsylvania, 16823-0048. ^{13}C-Mirex is purchased from Cambridge Isotope Laboratories, 20 Commerce Way, Woburn, Massachusetts, 01801. Photomirex is obtained from the Environmental Protection Agency (EPA) Research Triangle Institute.

12. Calibration Procedures

12.1 Instrument calibration

For perfluorotributylamine (PFTBA), the instrument calibration must be manually (hardware) verified and adjusted as necessary at the start of every 24 hours of operation. Ion abundance guidelines criteria are noted in the following chart. Adherence to these guidelines may make calibration of the instrument easier.

m/z	Percent Base
283	>1
312	>1
414	>10
557	>15
595	>60
596	5-10 of 633
633	Base peak
634	8-13 of 633

12.2 Initial calibration

An acceptable initial GC/MS calibration will meet the following criteria.

- RRTs for mirex, photomirex, and kepone relative to $^{13}C_8$-mirex of ±0.06 RRT units from the most recent GC/MS calibration standard.

- Relative ion abundance criteria for mirex, $^{13}C_8$-mirex, and photomirex (for the two most abundant ions in the quantitation cluster and for the two most abundant ions in the confirmation cluster) of ±20% from the theoretical ratio.

- Relative ion abundance criteria for kepone for the two most abundant ions in the quantitation cluster of ±20% from the theoretical ratio and for the two most abundant ions in the confirmation cluster of ±35% from the theoretical ratio.

The following chart describes this in further detail.

Analyte	Quantitation Ions (m/z)	Control Limits (m/z Ratios)	Confirmation Ions (m/z)	Control Limits (m/z Ratios)
Mirex	368/370	0.83-1.25	402/404	0.71-1.07
$^{13}C_8$-Mirex	376/378	0.83-1.25	410/412	0.71-1.07
Photomirex	368/370	0.83-1.25	402/404	0.71-1.07
Kepone	383/385	0.83-1.25	418/420	0.58-1.20

- Mass spectral sensitivity for mirex, photomirex, and kepone as defined by the presence at signal-to-noise (S/N) levels of greater than 2.5 of the following ions for the standard that is used for continuing calibration (1 ng mirex, 1 ng photomirex, and 4 ng kepone). The specific ions are noted below.

  - Mirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 437, 439, 441

  - Photomirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 439

  - Kepone: m/z 381, 383, 385, 387; m/z 416, 418, 420, 422

  - Mass spectral sensitivity for $^{13}C_8$-mirex as defined by the presence at S/N levels of greater than 5 for m/z 376, 378 and m/z 410, 412.

The response factor criteria are as follow:

- Acceptance criteria of average RRFs (RRF_{ave}) for mirex, photomirex, and kepone are 0.05 or larger.

- Acceptance criteria for the percent relative standard deviation (%RSD) of RRFs for mirex and photomirex relative to $^{13}C_8$-mirex are within 30% for the ions used for quantitation.

- Acceptance criteria for the %RSD of the RRF for kepone relative to $^{13}C_8$-mirex are within 40% for the ions used for quantitation.

The following chart describes the GC/MS calibration standards based on 1-mL aliquots with 1 uL injected.

Compound	Conc. No. 1	Conc. No. 2	Conc. No. 3	Conc. No. 4	Conc. No. 5
Mirex and Photomirex:					
ng/mL	200	400	1,000	2,000	4,000
ng/uL injected	0.2	0.4	1.0	2.0	4.0
Kepone:					
ng/mL	1,000	2,000	4,000	6,000	8,000
ng/uL injected	1.0	2.0	4.0	6.0	8.0
$^{13}C_8$-Mirex:					
ng/mL	1,000	1,000	1,000	1,000	1,000
ng/uL	1.0	1.0	1.0	1.0	1.0

12.3 Continuing calibration

The continuing calibration standard contains 1 ug/uL of mirex, 1 ug/uL of photomirex, 1 ug/uL of $^{13}C_8$-mirex, and 4 ug/uL of kepone. An acceptable GC/MS continuing calibration will meet the following criteria.

- RRTs for mirex, photomirex, and kepone relative to $^{13}C_8$-mirex of ±0.06 RRT units from the most recent GC/MS calibration standard.

- Relative ion abundance criteria for mirex, $^{13}C_8$-mirex, and photomirex (for the two most abundant ions in the quantitation cluster and for the two most abundant ions in the confirmation cluster) of ±20% from the theoretical ratio.

- Relative ion abundance criteria for kepone for the two most abundant ions in the quantitation cluster of ±20% from the theoretical ratio and for the two most abundant ions in the confirmation cluster of ±35% from the theoretical ratio.

The following chart describes this in further detail.

Analyte	Quantitation Ions (m/z)	Control Limits (m/z Ratios)	Confirmation Ions (m/z)	Control Limits (m/z Ratios)
Mirex	368/370	0.83-1.25	402/404	0.71-1.07
$^{13}C_8$-Mirex	376/378	0.83-1.25	410/412	0.71-1.07
Photomirex	368/370	0.83-1.25	402/404	0.71-1.07
Kepone	383/385	0.83-1.25	418/420	0.58-1.20

- Mass spectral sensitivity for mirex, photomirex, and kepone as defined by the presence at S/N levels of greater than 2.5 of the following ions. The specific ions are noted in the following items.

  - Mirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 437, 439, 441

  - Photomirex: m/z 366, 368, 370, 372; m/z 400, 402, 404, 406; m/z 439

  - Kepone: m/z 381, 383, 385, 387; m/z 416, 418, 420, 422

- Mass spectral sensitivity for $^{13}C_8$-mirex as defined by the presence at S/N levels of greater than 5 for m/z 376, 378 and m/z 410, 412.

The response factor criteria are as follow.

- Acceptance criteria of RRFs for mirex, photomirex, and kepone are 0.05 or larger.

- Acceptance criteria for the % difference (%D) of RRFs for mirex and photomirex relative to $^{13}C_8$-mirex are between -30% and +30% compared to the average RRF from the initial calibration.

- Acceptance criteria for the %D of the RRF for kepone relative to $^{13}C_8$-mirex are less than ±40% for the ions used for quantitation. After any continuing calibration where %D acceptance criteria for Kepone is not met, the laboratory must

analyze the low level standard from the initial five level calibration. Kepone must be detected in the low level standard to ensure that adequate sensitivity for Kepone has been maintained over the entire calibration range. Mass spectral sensitivity for Kepone is defined by the presence of S/N ratios of greater than 2.5 for the following ions for Kepone: m/z 381, 383, 385, 387, 416, 418, 420, and 422.

- The absolute retention time of $^{13}C_8$-Mirex shall not change more than ± 3 seconds from the continuing calibration.

13. Sample Preparation

13.1 Decant and discard any water layer.

13.2 Add 30 g of homogenized soil to a disposable glass jar and combine it with 60 g of anhydrous sodium sulfate and mix well.

13.3 For blanks, substitute soil with sodium sulfate. For matrix-specific quality control (QC) samples, weigh out 30 g of the [5-ropriate sample required for each QC sample. The matrix spike and matrix spike duplicate (MS/MSD) will be done using 1,000 ng of mirex, 1,000 ng of photomirex, and 5,000 ng of kepone.

13.4 Add 1,000 ng of $^{13}C_8$-mirex (internal standard).

13.5 Add 100 mL of methylene chloride/acetone (70:30 mixture) and agitate on an automatic shaker for 30 minutes.

13.6 Centrifuge for 3 minutes.

13.7 Decant liquid into an Erlenmeyer flask.

13.8 Repeat steps 13.5 through 13.7 two more times.

13.9 Quantitatively transfer the combined extracts through sodium sulfate into the KD apparatus. Wash the remaining soil/sodium sulfate mixture with 60 mL of methylene chloride/acetone and transfer the liquid into the KD apparatus.

13.10 Add 1 mL of toluene.

13.13 Screen the extract by GC/ECD, GC/FID, or GC/MS as outlined in appendix B.

13.14 If the extract is clean, inject 1 uL for GC/MS analysis.

13.15 If excess contamination is present in the extract, determine the dilution factor and dilute the extract using toluene containing 2% isopropanol. Always mix this well. From a 1-mL final extract volume, inject 1 uL for GC/MS analysis.

13.16 If $^{13}C_8$-mirex is diluted out and cannot be used as the internal standard anymore, respike the extract (1 mL) with 1,000 ng of $^{13}C_8$-mirex, mix well, and inject 1 uL for GC/MS analysis.

14. Analytical Measurement

14.1 Suggested instrument set up for GC/MS

- GC: HP 5890, equipped with HP 7673A autosampler or equivalent.

- Column: As specified in section 9.

- GC temperature program:
  Initial Temperature: 120 DEG C
  Initial Time: 3 Minutes
  First Ramp: 12 DEG C/ Minute
  Temperature: 250 DEG C
  Hold: 0.1 Minutes
  Second Ramp: 20 DEG C/ Minute
  Final Temperature: 310 DEG C
  Final Time: 2 Minutes

- Temperature GC injection port: 240DEGC.

- MS: Finnigan-MAT 4615B or Finnigan-MAT 45000 MS equipped with a PPNICI option and automated by a SuperIncos data system.

- Temperature ion source: 100DEGC (ion source block).

- Temperature transfer line: 280DEGC.

- Multiple ion detection (MID) descriptors: See appendix C

14.2 File naming conventions

Initial calibration GC/MS analyses for mirex, photomirex, and kepone are specified in the following chart.

File Name*	GC/MS Calibration Standard
2SMPK01	No. 1 (0.2 ng/uL of mirex and photomirex, 1.0 ng/uL of kepone)
2SMPK02	No. 2 (0.4 ng/uL of mirex and photomirex, 2.0 ng/uL of kepone)
2SMPK03	No. 3 (1.0 ng/uL of mirex and photomirex, 4.0 ng/uL of kepone)
2SMPK04	No. 4 (2.0 ng/uL of mirex and photomirex, 6.0 ng/uL of kepone)
2SMPK05	No. 5 (4.0 ng/uL of mirex and photomirex, 8.0 ng/uL of kepone)

*The "2" prefix denotes the instrument number.

A continuing calibration GC/MS analysis is specified by the instrument number, followed by MPK (to denote a standard for mirex, photomirex, and kepone), and the date of the analysis. For example, 2MPK072589 is the file name for the standard on Finnigan MS number 2, analyzed on July 25, 1989.

The file names for samples are designated by the six-digit Erco sample number derived from the booking log number and the "CI" suffix. The file names can contain the following additional suffixes.

- DU for duplicates
- MS for matrix spikes
- SD for matrix spike duplicates
- R for reinjections
- X for reextracts
- D for dilutions

For example, 202701CI is the file name for sample number 01 pertaining to project 2027 that is logged for the analysis of mirex, photomirex, and kepone.

The nomenclature for blanks is determined using the date of extraction, with a suffix denoting the appropriate extraction technique. For example, 25JUL89K12CI is the file name for the first blank extracted by shaker on July 25, 1989, and analyzed on Finnigan MS number 2.

14.3 Sample analysis

Confirmation of mirex, photomirex, and kepone in the samples will be done according to the following criteria.

- RRT of mirex, photomirex and kepone relative to $^{13}C_8$-mirex within ±0.06 RRT units from the most recent GC/MS calibration standard.

- Relative ion abundance criteria for mirex, $^{13}C_8$-mirex, and photomirex (for the two most abundant ions in the quantitation cluster and for the two most abundant ions in the confirmation cluster) of ±20% from the theoretical ratio.

- Relative ion abundance criteria for kepone for the two most abundant ions in the quantitation cluster of ±20% from the theoretical ratio and for the two most abundant ions in the confirmation cluster of ±35% from the theoretical ratio.

  Further details are listed in the section 12 chart.

- The presence at S/N levels greater than 2.5 of the following ions.

  - Mirex: m/z 366, 368, 370, 372 (quantitation cluster); m/z 400, 402, 404, 406 (confirmation cluster); and m/z 437, 439, 441 (confirmation cluster).

  - Photomirex: m/z 366, 368, 370, 372 (quantitation cluster) and m/z 400, 402, 404, 406 (confirmation cluster).

  - Kepone: m/z 381, 383, 385, 387 (quantitation cluster) and m/z 418, 420 (confirmation cluster).

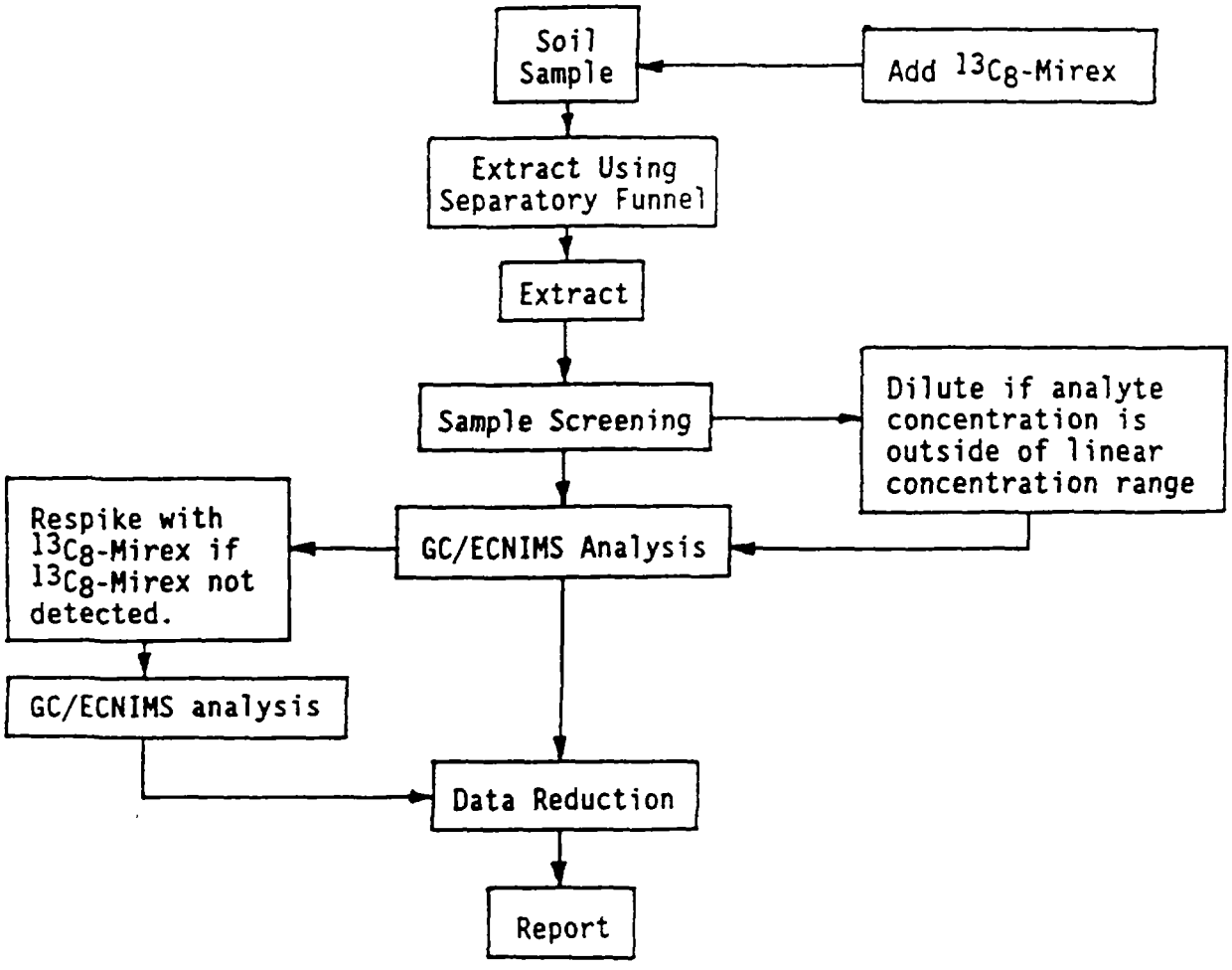
For data in which ion abundance criteria are not met (in particular, for the confirmation clusters) or data in which not all specified ions in the clusters are present, but, in the judgement of the analyst, the compound is present, the reporting of the compound will be qualified (in addition, see section 6 [interferences] and section 18.3).

14.4 Quantitation of the compounds

The quantitation methods used are in accordance with EPA Contract Laboratory Program (CLP) protocols for semivolatile organics with several modifications appropriate for the analytes and matrix. The summed abundances of two ions are used for quantitation. The response factors from the continuing calibrations are used. The generalized equation is presented in section 16.

The required criteria for the use of $^{13}C_8$-mirex as an internal standard are the the presence of the two major ions used for quantitation at S/N greater than 5.

15. Flow Chart



16. Data Treatment

16.1 The equation for quantitation of the compounds is as follows.

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

where:

C_x = *concentration of the compound to be measured (ng/uL);*
C_{is} = concentration of the internal standard (ng/uL);
A_x = area of the quantitation ions of the compound to be measured;
A_{is} = area of the quantitation ions of the internal standard; and
RRF = relative response factor from the continuing calibration standard.

16.2 The correction for contribution of mirex at m/z values measured for $^{13}C_8$-mirex will be done as follows.

16.2.1 Quantitation cluster

- Calculate 3.22% from m/z 368, subtract from m/z 376
- Calculate 0.351% from m/z 368 subtract from m/z 378

16.2.2 Confirmation cluster

- Calculate 6.65% from m/z 404, subtract from m/z 410
- Calculate 1.08% from m/z 404, subtract from m/z 412

16.3 Calculations required to verify automated data reduction are as follow.

16.3.1 To determine the RRF, use the following equation.

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

where:

A_x = summed areas of the quantitation ions for the compound to be measured;
A_{is} = summed areas of the quantitation ions for the specific internal standard;
C_{is} = concentration of the internal standard (ng/uL); and
C_x = concentration of the compound to be measured (ng/uL).

16.3.2 The average RRF is determined by following equation.

$$- RRF_{ave} = \frac{RRF_{conc1}+RRF_{conc2}+RRF_{conc3}+RRF_{conc4}+RRF_{conc5}}{5}$$

where:

RRF_X = RRF of a compound in each of the calibration solutions.

16.3.3 Percent relative standard deviation is determined by the following equation.

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

where:

RSD = relative standard deviation;
SD = standard deviation of initial RRFs (per compound); and
RRF_{ave} = mean of initial RRFs (per compound).

SD is determined with the following equation.

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

where:

$\bar{x}$ = mean of initial RRF (per compound); and
N = number of measurements.

16.3.4 Percent difference (%D) from average RRF is determined with the following equation.

$$- \%D = \frac{RRF_I - RRF_C}{RRF_I} \times 100$$

where:

RRF_I = average RRF from initial calibration; and
RRF_C = RRF from current calibration check standard.

16.3.5 Percent recovery is determined with the following equation.

$$- MSPR = \frac{SSR - SR}{SA} \times 100$$

where:

MSPR = matrix spike percent recovery;
SSR = spike sample results;
SR = sample result; and
SA = spike added from spiking mix.

16.3.6 Relative percent difference (RPD) is determined with the following equation.

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

where:

RPD = relative percent difference;
D_1 = first sample value; and
D_2 = second sample value (duplicate).

16.3.7 Percent moisture is determined with the following equation.

$$- \text{Percent moisture} = \frac{\text{wet weight} - \text{dry weight}}{\text{wet weight}} \times 100$$

16.3.8 To determine the analyte concentration in a soil sample (dry weight), use the following equation.

$$- \text{Concentration (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

where:

A_x = summed areas of the quantitation ions for the compound to be measured;
A_{is} = summed areas of the quantitation ions for the internal standard;
I_s = amount of internal standard injected in nanograms (ng);

RRF = RRF for the compound being measured, determined from the continuing calibration standard;
V_i = volume of extract injected in microliters (uL);
W_s = weight of the sample extracted (grams);
D = determined by the equation below; and
V_t = volume of total extract in microliters (uL), taking dilutions into account. For example, a 1:10 dilution of a 1-mL extract will mean V_t = 10,000 uL.

$$- D = \frac{100 - \%\ \text{moisture}}{100}$$

16.3.9 To determine the analyte concentration is a soil sample (wet weight), use the following equation.

$$- \text{Concentration (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_i)(W_s)}$$

where:

A_x = summed areas of the quantitation ions for the compound to be measured;
A_{is} = summed areas of the quantitation ions for the internal standard;
I_s = amount of internal standard injected in nanograms (ng);
RRF = RRF for the compound being measured, determined from the continuing calibration standard;
V_i = volume of extract injected in microliters (uL);
W_s = weight of the sample extracted (grams); and
V_t = volume of total extract in microliters (uL), taking dilutions into account. For example, a 1:10 dilution of a 1-mL extract will mean V_t = 10,000 uL.

17. Data Deliverables

The following items are the data deliverables that are applicable to this method.

- Case narrative.
- Perfluorotributylamine (PFTBA, FC-43) summary.
- Summary of initial calibration and continuing calibration check results.
- Summary of QC sample analyses.
- Laboratory blank compliance form.
- Summarized analytical results page.
- Summary sheets for the isotope ratios of the two most abundant ions in the quantitation cluster and the two most abundant ions in the in the major confirmation cluster for mirex, photomirex, $^{13}C_8$-mirex, and kepone.
- Logbook pages for extraction and GC/MS analysis.
- Raw data including the following items.
  - Work sheets (including sample calculations).
  - Screening chromatograms using GC/FID, GC/ECD, GC/EI/MS, or GC/ECNIMS used to choose appropriate dilutions for analysis.
  - Reconstructed ion chromatograms (RIC) at appropriate retention time intervals with mass chromatograms of the ions used for quantitation and confirmation. The following list describes the specific ions.
    - Mirex and photomirex: m/z 368, m/z 370, m/z 402, m/z 404
    - Kepone: m/z 383, m/z 385, m/z 418, m/z 420
    - $^{13}C_8$-Mirex: m/z 376, m/z 378, m/z 410, m/z 412
  - Listing of areas at appropriate m/z values used for quantitation; areas can be included on main chromatograms. This includes corrected areas for $^{13}C_8$-mirex.
  - Mass spectrum for each analyte (top of RIC peak).

18. Quality Control Requirements

18.1 Laboratory method blanks

No mirex, photomirex, or kepone will be present in the method blank above the reporting limit.

If a laboratory method blank exceeds these criteria, the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) must be reextracted and reanalyzed.

18.2 Precision and accuracy

The following recovery ranges and RPDs are calculated from method validation data (section 20). Four replicate analyses using spiking levels of 1,000 ng of mirex, 1,000 ng of photomirex, and 2,000 ng of kepone were done. The recovery range was defined the mean ±3 standard deviations; the RPD was defined as 3 times the standard deviation. RPDs that were calculated to be less than 30 were set at 30.

- Mirex - The recovery range is 57-124% and the RPD is less than 34%.
- Photomirex - The recovery range is 53-122% and the RPD is less than 35%.
- Kepone - The recovery range is 25-65% and the RPD is less than 30%.

18.3 Qualifier codes

One or more of the following qualifiers can be used.

- J = Result is detected below the reporting limit and is an estimated concentration.
- z = Presence of the compound is strongly indicated but the ion abundance ratio criteria are not met for the quantitation cluster ions.
- x = Presence of the compound is strongly indicated but the ion abundance ratio criteria are not met for the confirmation cluster ions.

- y = Presence of the compound is strongly indicated but not all specified ions in the clusters are present.

- k = Quantitation done using confirmation cluster ions.

- D = Compound identified at secondary dilution.

- E = Concentration exceeds calibration range.

- F = Reported value estimated due to an interference.

19. References

The following reference was used in developing this SOP.

- DeLeon, I.R., Remele, P.C., Miles, S.K., and Laseter, J.L. 1980. "Glass capillary columns in the gas chromatographic and gas chromatographic-mass spectrometric determination of trace levels of kepone in natural waters," Anal. Lett. 13(A6):503-515.

20. Method Validation Data

The method validation report was submitted in December 1989, by Enseco - Erco Laboratory.

CONFIDENTIAL

Appendix B

A.1 Screening of Extracts

The screening of extracts will be done by capillary column chromatography using splitless injection on a DB-5 or equivalent column. The detector can be an electron capture detector, a flame ionization detector, or a mass spectrometer depending on the judgement of the analyst. The suggested temperature program is the same as the program for final analysis.

A.1.1 Inject one microliter of a quantitative calibration standard containing mirex, photomirex, and kepone. If possible this calibration standard should be identical to the highest concentration standard for the GC/ECNIMS analysis.

A.1.2 Inject one microliter of each extract to be screened and compare the response to that of the calibration standard. Estimate the concentration of mirex, photomirex, and kepone in the extract using a single point response factor obtained from the calibration standard.

A.1.3 Samples will be analyzed using selected ion monitoring. The 272 m/z ion will be the primary ion for quantitation.

Using the estimated concentration, make a dilution of the extract that brings the concentrations of mirex, photomirex, and kepone into the GC/ECNIMS calibration range. Dilutions should be made such that the estimated concentration is toward the upper end of the calibration range. Peaks in the samples at retention times that do not correspond to mirex, photomirex, and kepone will be evaluated on a case by case basis.

If necessary in the judgement of the analyst, an additional screen of the diluted extract may be made.

ATTACHMENT B

SAMPLING AND FIELD TESTING PROCEDURES

See Appendix A, Field Sampling Plan
Section I - Volume 2

VOLUME 2

SECTION III - HEALTH AND SAFETY PLAN

REVISION #2

April 1994

Project No.: 933-6158

April 1994

HEALTH AND SAFETY PLAN
NEASE CHEMICAL SITE
SALEM, OHIO SITE

Prepared for:

Ruetgers-Nease Corporation to satisfy the requirements of the Administrative Order of Consent for the Salem Interim Remedial Measures, dated November 17, 1993.

ADDENDUM TO

HEALTH & SAFETY PLAN

Prepared by:

Golder Associates Inc.
April 1994

The approved RI Work Plan - Health and Safety Plan as amended by this addendum, constitutes the Health and Safety Plan for this project.

1. Figure HSP 3-1 Delete ERM personnel and replace with appropriate GAI personnel:

U.S. EPA Region 5 Remedial Project Manager:	Sheila Sullivan
OPEA Site Coordinator:	Joseph Trocchio
Project Director:	P. Stephen Finn
Health and Safety Officer:	William E. Harris, OSHT
Site Safety Manager:	Geoffrey R. Forrest
Field Operations Manager:	Joseph Cavanagh
Field Personnel:	Geoffrey R. Forrest and Joseph Cavanagh

2. Page HSP-24 through 28, replace personnel in Sections 3.1 through 3.8.

3.1	U.S. EPA Region 5 Remedial Project Manager:	Sheila Sullivan
	OEPA Site Coordinator:	Joseph Trocchio
3.3	Principal-In-Charge:	P. Stephen Finn
3.4	Project Manager:	Geoffrey R. Forrest
3.5	Field Operations Manager:	Joseph Cavanagh
3.6	GAI's Health and Safety Officer is William E. Harris, OSHT and is responsible for overall coordination of safety matters for GAI's Eastern Regional Offices. [no further changes]	
3.7	Site Safety Manager:	Geoffrey R. Forrest

3. Page HSP-35 Section 5.1 #1 Add: The Site Safety Manager will contact the appropriate off-site emergency assistance group(s) prior to the state of activities and will get approximate response times for the group(s).

4. Page HSP-36 #7 Delete: "Contact lenses will not be permitted at the Site." [Contact lenses in conjunction with safety glasses will be permitted in accordance with the latest ANSI standards regarding respiratory protection.]

5. Page HSP-41 Section 5.2.3.1 #1 Change: Full or half face air-purifying respirators with appropriate cartridges to provide protection from acids, volatile and non-volatile organic vapors. [This field activity involves liquid sampling and, hence, particulates will not be a problem. Exposure will be

limited and readily controlled so half face respirators should provide adequate protection.]

6. Page HSP-42 Section 5.2.3.1 #6 Delete [Given the limited and readily controlled potential exposures, 5-minute escape packs should not be required.]

7. Section 5.2.4 Add: It is anticipated that the activities to be undertaken can be conducted using Level D protection.

8. Page HSP-43 Section 5.2.4.1 #1 Delete [Level D is by definition used in non-respiratory hazard areas and activities.]

9. Page HSP-43 Section 5.2.4.2 #2 Change: Cotton Coveralls to Work Clothes

10. Page HSP-49 Section 6.3 Change: Verification of training will be submitted to the GAI Health and Safety Officer.

11. Page HSP-50 Section 7.0 Change: All instrument calibration records and data forms will be held on-site during the project and transferred to the project file at the end of the project.

12. Page HSP-50 Section 7.1 Change: To ensure that all monitoring instruments are operating properly, field calibrations will be performed and documented using the appropriate form. Calibrations will be conducted according to manufacturer specifications and will be performed at a minimum of once per day, at the beginning of the day.

13. Page HSP-51 Section 7.1.2 Delete [Current field activities do not require monitoring for these parameters.]

14. Page HSP-52 Section 7.3 #1 Change: Full face to Full or half face respirators

15. Page HSP-52 Section 7.3 #7 Change: Coveralls, (e.g. Tyvek, uncoated) to splash aprons.

16. Page HSP-53 #13 Delete

17. Page HSP-54 Section 8.1.1 #2,3,4 Delete [Current field activities do not require the monitoring of these parameters.]

18. Page HSP-54 Section 8.1.1 #2, 3, 4 Delete [Current field activities do not require the monitoring of these parameters.]

19. Page HSP-55 #1 Change: GAI will use the following action levels for air monitoring conducted in the breathing zone:

Background	Level D
up to 5 ppm for one hour OR	Level C
10 ppm for one minute OR	
any peak greater than 25 ppm	

Concentrations greater than 50 ppm for more than 1 hour will necessitate the use of Level B protection. It is not anticipated that this level of exposure will occur.

20. Page HSP-55 Section #2, 3, 4 Delete [Current field activities do not require monitoring for these parameters.]

21. Page HSP-66 Station 3 #2 Change: High pressure to Garden-type spray unit. (Note: this change is applicable to similar decontamination rinses throughout the plan.)

22. Page HSP-90 Section 10.1 Paragraph two Change: Copies of accident reports will be kept in the project file and by the home office's human relations director.

23. Page HSP-91 Paragraph two Delete: "OSHA_recordable" from first sentence and add Office Manager to the list of persons to be notified.

24. Page HSP-92 Emergency Equipment List Delete: fire blanket, stretcher, hand-held air horns, egress air packs. Change eyewash station to eye wash bottle.

25. Page HSP-98 Second bullet Delete: Certified CPR personnel will be on-site at all times during field activities.

26. Page HSP-102 Change: ERM phone number to GAI, Mt. Laurel, NJ (609) 273-1110.

27. Page HSP-103 Section 11.0 Delete #2, #6, #7 (combined with #9)

28. Page HSP-105 Section 12.1 Delete: first two sentences

29. Page HSP-105 Section 12.1 Change ERM-Midwest's to Golder Associate's

30. Page HSP-106 Physical Exam Delete: contents of exam [The contents of GAI's medical program is quite complete and may vary based on individuals, past exposure history and/or age and sex. The exam meets requirements set under 29 CFR 1910.120.]

31. Page HSP-108 Last paragraph Change: Reports are sent to individuals and/or your physician upon request. A medical clearance statement is kept on file in the GAI office. Original reports are kept by the attending physician.

32. Page HSP-109 Change: GAI conducts its own OSHA-mandated training courses relevant to 29 CFR 1910.120 as the standard pertains to GAI's work activities. Individuals who received their initial training outside of GAI may or may not have received training in all the "non-essential" (e.g. Sampling and Chain-of-Custody) items listed in this section. All GAI field employees working on hazardous waste sites have received training as specified under 29 CFR 1910.120.

33. Appendix A Page B-2 Delete: [Physician's Medical Opinions are kept on file in the GAI offices.]

34. Appendix A Page B-6 Delete: [Individuals are issued respirator fit test cards following successful qualitative site testing and will have these cards available while on site.]

35. Appendix A Page B-7 Delete: [Hearing Protection is provided to employees at their request or as necessary by the project and is not accompanied by an Issuance Form.]

36. Appendix A Pages B-8 through 10 Change: Similar forms are provided to field personnel tasked with air monitoring activities.

Volume 4: Health and Safety Plan
Ruetgers-Nease Salem, Ohio Site

Revision 4: February 28, 1990
Revision 3: December 13, 1989
Revision 2: August 3, 1989
Revision 1: October 22, 1988

Submitted to

United States Environmental Protection Agency, Region 5
Hazardous Waste Enforcement Branch
230 S. Dearborn St.
Chicago, Illinois 60604

Ohio Environmental Protection Agency
Corrective Actions Section
Division of Solid and Hazardous Waste Management
1800 Watermark Dr.
Columbus, Ohio 43266

Northeast District Office
Division of Solid and Hazardous Waste Management
2110 Aurora Rd.
Twinsburg, Ohio 44087

Submitted by

Ruetgers-Nease
Chemical Company, Inc.
201 Struble Rd.
State College, Pennsylvania 16801

TABLE OF CONTENTS

Section	Title	Page
1.0	INTRODUCTION	HSP-1
	1.1 Plan Purpose.....	HSP-1
	1.2 Site Background and History.....	HSP-2
	1.2.1 Location.....	HSP-2
	1.2.2 History.....	HSP-6
	1.2.3 Existing Conditions.....	HSP-7
	1.3 Problem Statement.....	HSP-7
	1.3.1 Sources.....	HSP-8
	1.3.2 Affected Media.....	HSP-8
	1.3.3 Sampling Rationale.....	HSP-8
2.0	SCOPE AND LIMITATIONS.....	HSP-21
3.0	ORGANIZATION AND RESPONSIBILITIES.....	HSP-22
	3.1 U.S. EPA and OEPA Project RPM and Coordinators.....	HSP-22
	3.2 Project Coordinator.....	HSP-24
	3.3 Principal-in-Charge.....	HSP-24
	3.4 Project Manager.....	HSP-24
	3.5 Site Manager.....	HSP-24
	3.6 ERM-Midwest Project Health and Safety Officer.....	HSP-25
	3.7 Field Operations Manager.....	HSP-26
	3.8 Field Personnel.....	HSP-28
4.0	SITE HAZARD EVALUATION.....	HSP-29
	4.1 Health Effects.....	HSP-29
5.0	PERSONNEL PROTECTION AND HEALTH AND SAFETY GUIDANCE FOR SPECIFIC TASKS.....	HSP-35
	5.1 Standard Operating Procedures.....	HSP-35
	5.2 Protective Equipment Selection and Limitation.....	HSP-38
	5.2.1 Level A.....	HSP-38
	5.2.1.1 Protective Equipment.....	HSP-38
	5.2.1.2 Selection Criteria..	HSP-39
	5.2.2 Level B.....	HSP-39

TABLE OF CONTENTS (Continued)

Section	Title	Page
	5.2.2.1 Protective Equipment.....	HSP-40
	5.2.2.2 Selection Criteria.	HSP-40
	5.2.3 Level C.....	HSP-41
	5.2.3.1 Protective Equipment.....	HSP-41
	5.2.3.2 Selection Criteria.	HSP-42
	5.2.4 Level D.....	HSP-43
	5.2.4.1 Protective Equipment.....	HSP-43
	5.2.4.2 Selection Criteria.	HSP-43
	5.3 Special Procedures for Subsurface Activities.....	HSP-44
	5.4 Special Procedures for Test Pit Operations.....	HSP-44
6.0	SITE CONTROL.....	HSP-46
	6.1 Access-Personnel.....	HSP-46
	6.2 Security.....	HSP-48
	6.2.1 Communications.....	HSP-48
	6.2.2 Administrative Actions for Violations.....	HSP-48
	6.3 Training Verification.....	HSP-49
7.0	MONITORING EQUIPMENT.....	HSP-50
	7.1 Air Monitoring.....	HSP-50
	7.1.1 Organic Vapors.....	HSP-50
	7.1.2 Oxygen, Combustible Gases, and Toxic Gases.....	HSP-51
	7.2 Emergency Equipment.....	HSP-51
	7.2.1 Fire Extinguishers.....	HSP-51
	7.2.2 First Aid.....	HSP-51
	7.3 Personnel Protection Equipment.....	HSP-52
8.0	WORK LIMITATION LEVELS.....	HSP-54
	8.1 Work Limitation Criteria.....	HSP-54

TABLE OF CONTENTS (Continued)

Section	Title	Page
	8.1.1 Air Criteria.....	HSP-54
	8.1.2 Extreme Weather Conditions..	HSP-56
	8.2 Site Evacuation.....	HSP-57
	8.2.1 Upwind Withdrawal.....	HSP-57
	8.2.2 Evacuation of Site.....	HSP-58
9.0	DECONTAMINATION AND HAZARDOUS MATERIAL HANDLING PROCEDURES.....	HSP-59
	9.1 Decontamination Stations.....	HSP-59
	9.2 Equipment Decontamination Procedures.....	HSP-60
	9.2.1 Heavy Equipment.....	HSP-60
	9.2.2 Small Sampling Equipment....	HSP-61
	9.2.3 Well Construction Supplies..	HSP-64
	9.3 Personnel Decontamination Procedures.....	HSP-65
	9.3.1 Level A Decontamination Procedures.....	HSP-65
	9.3.2 Level B Decontamination Procedures.....	HSP-72
	9.3.3 Level C Decontamination.....	HSP-78
	9.3.4 Level D Decontamination.....	HSP-85
	9.4 Hazardous Materials Handling Procedures.....	HSP-88
	9.4.1 Off-Site.....	HSP-88
	9.4.2 On-Site.....	HSP-89
10.0	EMERGENCY PROCEDURES AND CONTACTS.....	HSP-90
	10.1 General Medical Procedures.....	HSP-90
	10.2 Emergency Horn.....	HSP-92
	10.3 Evacuation Plan.....	HSP-92
	10.4 Cold/Hot Weather Related Emergencies.....	HSP-93
	10.5 Chemical Exposure.....	HSP-96
	10.6 Fires.....	HSP-98
	10.7 Soils.....	HSP-99
	10.8 Unusual Objects or Events.....	HSP-99
	10.9 Emergency Personnel.....	HSP-100
	10.10 Emergency Contacts.....	HSP-100

TABLE OF CONTENTS (Continued)

Section	Title	Page
11.0	FIELD DOCUMENTATION OF HEALTH AND SAFETY PROCEDURES.....	HSP-103
12.0	MEDICAL SURVEILLANCE PROGRAM.....	HSP-105
	12.1 Background.....	HSP-105
	12.2 Physical Examination.....	HSP-105
13.0	PERSONNEL TRAINING.....	HSP-109
	13.1 Training Course.....	HSP-109
	13.2 Refresher Courses.....	HSP-112
	13.3 Supervisor Training Course.....	HSP-112
	13.4 Hearing Conservation.....	HSP-113
	13.5 Health and Safety Site Plan.....	HSP-114

LIST OF APPENDICES

Appendix	Title
APPENDIX A	THE HEALTH EFFECTS AND THE RESPONSE TO EXPOSURE TO SITE SPECIFIC COMPOUNDS
APPENDIX B	HEALTH AND SAFETY FORMS

LIST OF FIGURES

Figure	Title	Page
Figure 1-1	Site Location Map.....	HSP-3
Figure 1-2	Stratigraphic and Hydrogeological Cross Section.....	HSP-5
Figure 3-1	Health and Safety Project Organization.....	HSP-23
Figure 6-1	Exclusion Areas.....	HSP-47
Figure 10-1	Route to the Salem Community Hospital.....	HSP-101

TABLE OF CONTENTS (Continued)

LIST OF TABLES

Table	Title	Page
Table 1-1	Potential Contaminate Sources.....	HSP-9
Table 1-2	Media Affected or Potentially Affected.....	HSP-10
Table 1-3	Sampling Rationale	HSP-12
Table 1-4	RI Sampling Summary.....	HSP-18
Table 1-5	Critical Sample Summary.....	HSP-19
Table 4-1	Health and Safety Limits for Possible Hazardous Compounds	HSP-30

1.0 INTRODUCTION

This Health and Safety Plan (HSP) describes the procedures that will be followed in conducting field investigation activities at the Ruetgers-Nease Chemical Company (Ruetgers-Nease) Salem, Ohio Site. This section of the HSP describes the purpose of the plan and provides background information on Site conditions.

1.1 Plan Purpose

This HSP is designed to protect the health and safety of personnel involved in the investigation of hazardous substances at the Site and to develop a contingency plan for dealing with on-site emergencies, some of which may have the potential for an off-site impact. Topics addressed in the health and safety program described by this HSP include:

1. Site characteristics
2. Waste characteristics
3. Hazard evaluation
4. Site control
5. Engineering safeguards
6. Personnel training
7. Personnel protection
8. Monitoring equipment

9. Work limitations

10. Action levels

11. Decontamination

12. Medical surveillance

13. Emergency contacts

14. Incident reporting

15. Field documentation of the health and safety procedures

16. Handling of on and off-site generated materials

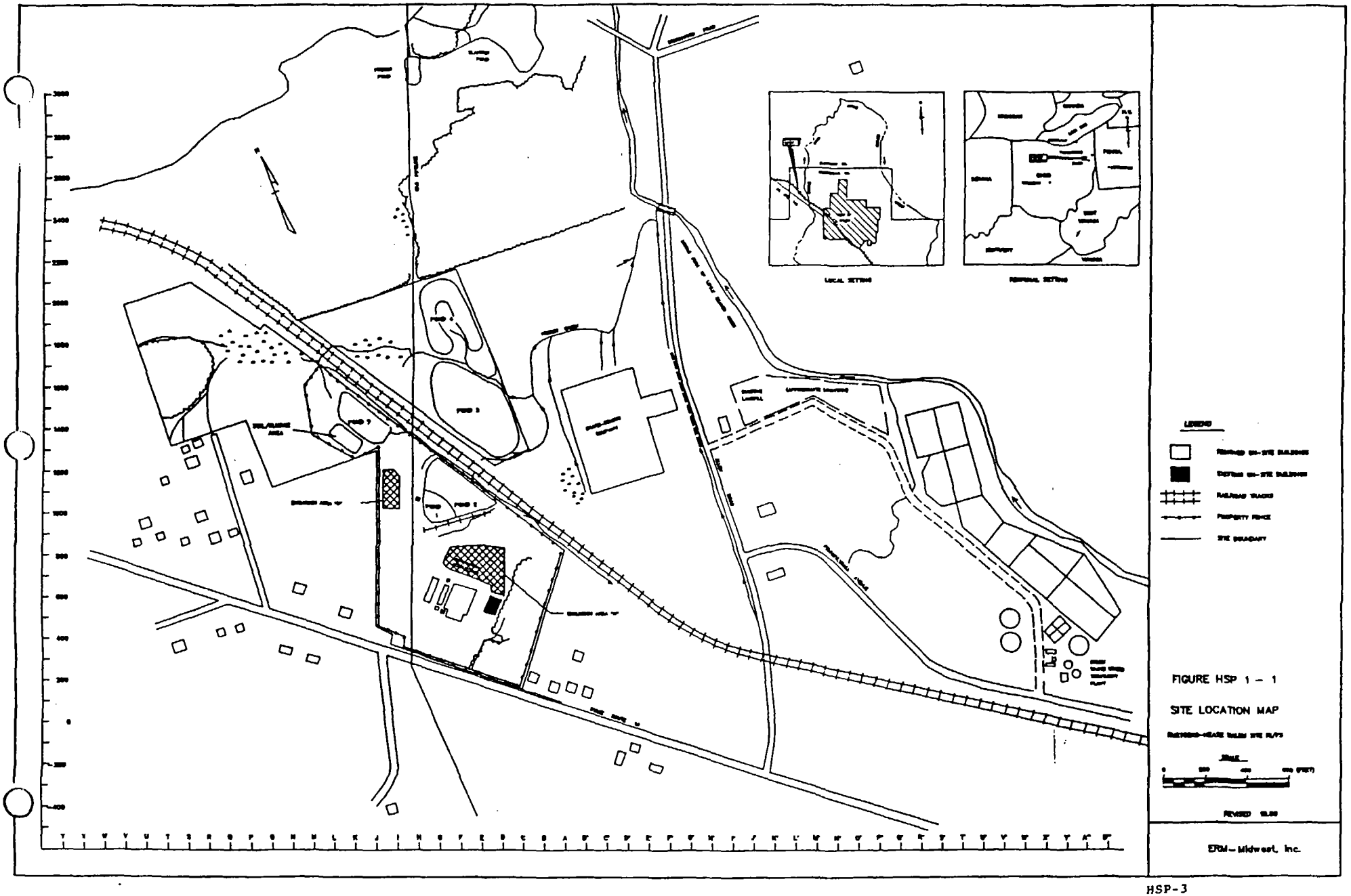
This document describes these program elements as they apply to the activities at the Salem Site.

1.2 Site Background and History

The purpose of providing background information is to educate RI/FS personnel regarding conditions at the Site; the potential risks due to physical, chemical, and toxicological properties of the materials present at the Site; and other types of hazards associated with the investigation such as working with water or heavy equipment.

1.2.1 Location

The Site is located approximately one mile northwest of the City of Salem (see Figure 1-1). Conrail railroad tracks separate the Site into two unequal sections that total


LOCAL SETTING
REGIONAL SETTING
LEGEND
RAILROAD TRACKS
PROPERTY FENCE
SITE BOUNDARY
FIGURE HSP 1 - 1
SITE LOCATION MAP
SCALE
REVISED
ERM-Midwest, Inc.

approximately 44 acres. The Site is bounded by small light-industrial operations along Allen Road to the east, residences to the immediate southwest, State Route 14A to the south, and wooded areas and pasture lands to the north. Site stormwater drains in a northeasterly direction to the main surface water body in the area, the Middle Fork Little Beaver Creek (MFLBC), which flows northward and then southward to Little Beaver Creek which eventually flows into the Ohio River.

There is an inactive landfill located approximately 1,200 feet east of the Site along the west bank of the MFLBC. This landfill was operated as a dump and extends from the MFLBC to the east side of Allen Road. The area is presently covered with vegetation and construction rubble.

The area's potable water supply is provided by both a public water system and private wells. The small businesses along Allen Road receive drinking water from the City of Salem, although Dunlap Disposal uses well water for non-potable purposes. Residents along State Route 14A and further north on Allen Road and Goshen Road use either public water or private wells. The City of Salem has a reservoir which draws water from Cold Run Creek, approximately seven miles south of the Site. Cold Run Creek is in a different watershed than is the Site.

The area is underlain by glacial deposits of the Kent Moraine, a five to fifteen mile wide belt of nonhomogeneous glacial drift. Approximately 10 to 25 feet of drift underlies the Site (Figure 1-2). This drift consists mostly of silty or sandy, gray-blue, plastic clay with some pebbles and boulders. Sand and gravel deposits within the drift have permeabilities higher than that of the silty or sandy clays. The drift is underlain by sedimentary rocks consisting mainly of interbedded sandstones, shales and coal seams.

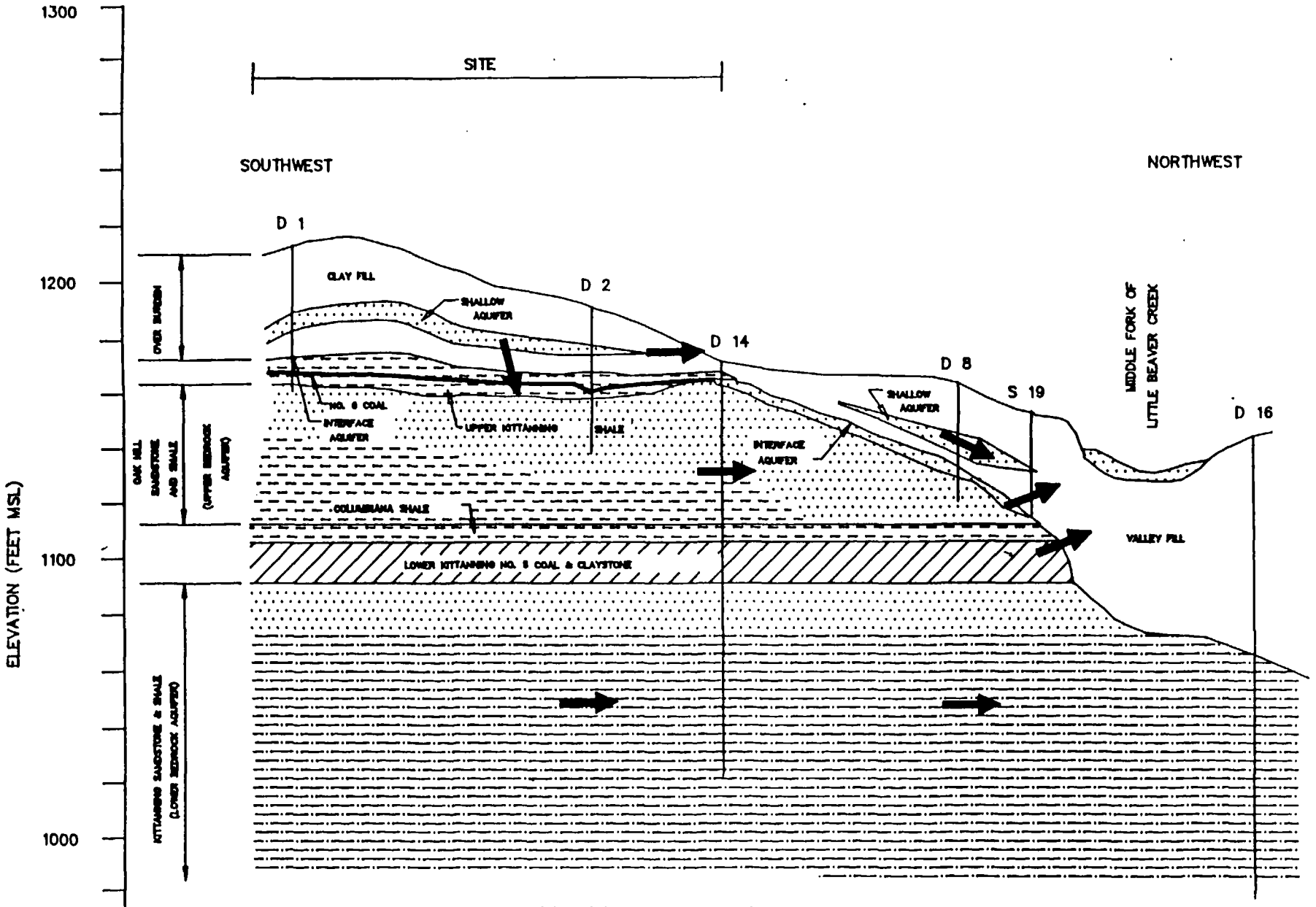


FIGURE HSP 1 – 2

STRATIGRAPHIC AND HYDROGEOLOGIC

CROSS SECTION

RUETGERS–NEASE SALEM SITE RI/FS

REVISED 10.88

DIRECTION OF GROUND WATER FLOW

ERM–Midwest, Inc.

Prior to drift deposition, glacial forces eroded a valley into the sedimentary rocks east of the Site. This eroded valley was filled as glacial drift was deposited.

The distinct aquifers within the study area have been named the Shallow, Interface, Upper Bedrock, Lower Bedrock and Valley Fill. Both confined and unconfined conditions are found in the area. Ground water beneath the Site is apparently moving toward the MFLBC. Hydrogeologic conditions near the MFLBC and within the Valley Fill have not been fully delineated.

1.2.2 History

From 1961 until 1973, Nease Chemical Company produced chemicals such as household cleaning compounds, pesticides, fire retardants, and chemical intermediates at the Site. Products and chemical intermediates were produced in batch processes.

Nease's waste handling facilities included air scrubbers and a multiple pond/settling tank system for neutralization and treatment of acidic waste. Five unlined lagoons (1, 2, 3, 4, and 7) were used for treatment and storage of either acidic waste or lime slurries from waste neutralization. In 1969, a pipeline was constructed to carry neutralized wastewater to the Salem Wastewater Treatment Plant. Some 55-gallon drums containing wastes were buried on-site in Exclusion Area A.

Following notification from OEPA of wastewater violations, Nease Chemical Company agreed in a Consent Judgment in 1973, to discontinue manufacturing operations at the Site until such time as a new wastewater permit was obtained. Instead, Nease decided to close the facility. Pond water was neutralized and removed to the Salem

Wastewater Treatment Plant. Nease also filled/graded several ponds, and removed all production facilities with the exception of a warehouse and two small block buildings. On December 30, 1977, Nease merged with Ruetgers Chemicals, Inc. to form Ruetgers-Nease Chemical Company, Inc.

1.2.3 Existing Conditions

Currently, the Site contains a single-story warehouse; two small concrete-block buildings; concrete pads, foundations, and tile floors remaining from the manufacturing facilities; concrete tank saddles; and the pond areas. The Site west of the Conrail tracks is surrounded by a fence with access from Route 14. Much of the Site has been revegetated by weeds and grasses.

The banks of Pond 1 slope steeply to the water surface, which is about eight to ten feet below grade. The surface of Pond 2 contains isolated patches of grasses and weeds surrounded by barren areas. The surfaces of Ponds 3, 4 and 7 consist of weeds, grasses, small shrubs and some small trees. A soil/sludge pile west of Pond 7 is covered by weeds, grasses, shrubs and small trees, and is approximately five to eight feet above grade. The surfaces of Ponds 3 and 7 may not support heavy equipment.

The remainder of the study area contains large fields, stands of trees, plowed fields, houses and buildings. The Crane-Deming swamp contains areas of bare soils, and wetland grasses. Trees and dense vegetation border the MFLBC.

1.3 Problem Statement

Previous studies have identified sources on-site and have indicated that contaminant migration off-site may be

occurring. The RI approach has been developed, in part, to identify all sources, characterize contaminants, and determine the limits of contaminant migration. A description of known sources, affected media, and the rationale for sampling are provided in the following sections.

1.3.1 Sources

Potential sources of contamination at the Site are listed in Table 1-1. The actual contribution of sources to affected media have not been qualified and quantified. The complete set of contaminants migrating from these sources has not been qualified or quantified by samples meeting USEPA QA/QC guidelines.

1.3.2 Affected Media

Previous studies have identified contaminants in various media. Media affected or potentially affected are listed on Table 1-2.

1.3.3 Sampling Rationale

Sample locations and analyses have been identified that will provide data necessary to meet the objectives of the RI/FS and to complete activities. Sample types, locations, collection methods, and sampling objectives are listed on Table 1-3. Scheduled analyses are listed on Table 1-4. Samples considered critical to achieving project objectives, and a summary of activities to ensure these samples meet QA/QC criteria are listed on Table 1-5

Target Compound List (TCL) analysis will be conducted using Contract Laboratory Program (CLP) procedures. Methods will be developed and validated for mirex, kepone, photomirex, DCNB, and DPS, which are not on the TCL. Methods

HSP TABLE 1-1

POTENTIAL CONTAMINANT SOURCES(1)
RUETGERS-NEASE SALEM SITE RI/FS

Source	Characterization
Exclusion Areas A and B	Some buried drums and contaminated soils (removed) contained volatile and non-volatile organics.
Pond 1, 2	Disposal of treated process waters containing volatile organic compounds in non-secure/unlined areas (some Pond 1 soil removed).
Ponds 3,4,7,	Contaminants, if present in soil/sludge stockpile neutralized calcium sulfate sludge, may migrate through unlined pond bottoms or in surface runoff.
Manufacturing areas, on-site surface and subsurface soils	Possible chemical spills may have contaminated soils.

(1) Based on sampling by Ruetgers-Nease performed to date. Confirming and characterizing sources is an objective of the RI.

HSP TABLE 1-2

AFFECTED OR POTENTIALLY AFFECTED MEDIA
RUETGERS-NEASE SALEM RI/FS

Media	Location	Potential Contaminants
Surface Water	Feeder Creek(1)	Volatile Organics Non-Volatile Organics Additional Organics
	MFLBC(1)	Volatile Organics Non-Volatile Organics Additional Organics
Sediments	Feeder Creek(1)	Volatile Organics Non-Volatile Organics Additional Organics
	MFLBC(1)	Volatile Organics Non-Volatile Organics Additional Organics
	Slanker Pond(1)	Non-Volatile Organics Additional Organics
	Crane-Deming(1) Swamp	Volatile Organics Non-Volatile Organics Additional Organics
Fish	MFLBC(2)	Non-Volatile Organics Additional Organics
Soils	On-Site(1)	Volatile Organics Non-Volatile Organics Additional Organics
Sludges	Salem Wastewater Treatment Plant Sludge Cells 4, 6 and 8 (2)	Volatile Organics Non-Volatile Organics Additional Organics

(1) Based on sampling by Ruetgers-Nease performed to date.
(2) Ruetgers-Nease has not sampled these locations.
(3) This aquifer may, or may not exist down gradient of the site.

*Non-Volatile Organics are defined here and throughout the document as TCL BNA +25, TCL pesticides/PCBs

HSP TABLE 1-2 (Cont'd)

AFFECTED OR POTENTIALLY AFFECTED MEDIA
RUETGERS-NEASE SALEM RI/FS

Media	Location	Potential Contaminants
Ground Water	Shallow(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Interface(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Upper Bedrock(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Lower Bedrock(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Valley Fill(2)(3) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics

(1) Based on sampling by Ruetgers-Nease performed to date.
(2) Ruetgers-Nease has not sampled these locations.
(3) This aquifer may, or may not exist down gradient of the site.

*Non-Volatile Organics are defined here and throughout the document as TCL BNA +25, TCL pesticides/PCBs

HSP TABLE 1-3

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
1. Characteristics of on-site surface and subsurface soils.	1. On site soil contaminant distribution - horizontal and vertical 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Test pits, analysis of samples from side walls of pits and backhoe bucket
2. Characteristics of off-site surface and subsurface soils in the Crane-Deming Swamp.	1. Contaminant distribution in the Crane-Deming Swamp - horizontal and vertical 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Test pits, analysis of samples from side walls of pits and backhoe bucket

HSP TABLE 1-3 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
3. Characteristics of on-site Non-native Pond materials.	1. Pond contaminant distribution - vertical and horizonal 2. Non-native material physical characterization 3. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 4. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Soil borings, sampling and analysis of split spoon and shelby tube samples
4. Characteristics of Native Pond materials (soils under the pond bottom).	1. Pond contaminant distribution - vertical horizonal 2. Native material physical characterization 3. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment	Soil borings, sampling and analysis of split spoon and shelby tube samples

HSP TABLE 1-3 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
	4. Support the identification, development, and evaluation of remedial alternatives/ technology, and detailed alternative evaluation completed during the Feasibility Study.	
5. Characteristics of off-site surface and subsurface soils.	1. Off-site soil contaminant distribution 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Soil borings, sampling and analysis of split spoon or auger samples
6. Characteristics of on and off-site sediments.	1. Sediment contaminant distribution - horizontal - within the drainage ways, Feeder Creek, Slanker Pond and MFLBC	Collection and analysis of location specific surface sediments

HSP TABLE 1-3 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
	2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	
7. Characteristics of on and off-site surface water bodies	1. Contaminant distribution - horizontal - within the drainage ways, Feeder Creek, Slanker Pond and MFLBC 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Collection and analysis of location specific surface water samples

HSP TABLE 1-3 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
8. Characteristics of on and off-site ground water	1. Contaminant distribution - horizontal and vertical aquifers 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Sampling and analysis of monitoring and residential wells
9. Air monitoring station upwind and downwind of the site	1. Define areal extent of contaminant concentrations 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Sampling and analysis of 6 stations

HSP TABLE 1-3 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed		Rationale	Data Gathering Methods
10. Mapping and surveying	1.	Locate existing structures and obstructions for alternatives evaluation, site features, and topography description	Site survey, site inspections, existing and updated facility maps

HSP Table 1-4

RI Sampling Summary[1]
Ruetgers-Nease Salem Site RI/FS

Media	Location	Sample Type	Collective Analysis
Ground water Round 1	Monitoring and residential wells (68 locations)	Bailed/pumped	TCL Organics +40[2]; SAS1[3]
	Monitoring wells (S6, S12, S18, T2)	Bailed/pumped	TCL Organics +40; SAS1; SAS2[4]; TCL Inorganics
Round 2	Monitoring wells (TBD)[5]	Bailed/pumped	To be determined
Soil	On-site, Crane-Deming (24 locations)	Test pit, depth specific	TCL Organics +40; SAS1
	Exclusion Area A & B + 4 additional sites (6 locations)	Test pit, depth specific	TCL Organics +40; SAS1; SAS2; TCL Inorganics
	Railroad tracks (TBD)[5]	Test pit, depth specific	TCL Organics +40; SAS1
	5 ponds, soil/sludge area (14 borings)	Split spoon, depth specific (non-native)	TCL Organics +40; SAS1, SAS2; TCL Inorganics
		Split spoon, depth specific (native)	TCL Organics + 15; SAS1; Methoxychlor
		Split spoon, depth specific composite (native)	TCL Non-Volatile Organics + 25
		Shelby Tube (3 feet) depth specific	Physical characteristics
	Floodplains (7 locations)	Composite	TCL Non-Volatile Organics +25; SAS1
	Off-site soils (11 borings)	Split spoon	TCL Non-Volatile Organics + 25; SAS1
	Off-site soil/sludge (3 locations)	Split spoon	TCL Organics +40; SAS1
Sediment	Slanker Pond (4 locations)	Pond bottom, beach, inlet/outlet	TCL Organics +40; SAS1
	MFLBC (50 locations)	Composite (1 foot)	TCL Organics +40; SAS1
	Feeder Creek (3 locations)	Composite (1 foot)	TCL Organics +40; SAS1
	On-site drainage and Crane-Deming (4 locations)	Grab	TCL Organics +40; SAS1
Surface Water	Slanker Pond (1 location)	Grab	TCL Organics +40; SAS1
	MFLBC (21 locations)	Grab	TCL Organics +40; SAS1
	Feeder Creek (3 locations) if water present	Grab	TCL Organics +40; SAS1
	Crane-Deming (1 location)	Grab	TCL Organics +40; SAS1
Fish	Slanker Pond (1 location)		TCL Organics +40; SAS1
	MFLBC (27 locations)		TCL Organics +40; SAS1
Air	On-site, upwind downwind (6 locations)	24 hour	TCL Organics +40[6]; SAS1

[1]Based on one sampling event
[2]Target compounds list plus library searches
[3]SAS1 = Mirex, photomirex, kepone, DPS
[4]SAS2 = 3,4-DCNB, dioxins and furans
[5]To be determined
[6]Four of the ketones on the VOA TCL will not be examined.

HSP TABLE 1-5

CRITICAL SAMPLE SUMMARY
RUETGERS-NEASE SALEM SITE RI/FS

Sample Location	Matrix	Rationale	Activity
Well Cluster "I"	Ground Water	1) Determine existing background water quality at the site. 2) Provide basis for remediation levels.	1) Collect collocated sample from one well. 2) Complete two sample events. 3) 100%(1) completeness goal. 4) SAS low detection levels. (See Section 9)
Residential Wells	Ground Water	1) Ensure acceptable drinking water quality.	1) Collect two collocated samples during each sampling event. 2) 100%(1) completeness goal. 3) SAS low detection levels. (See Section 9)
MFLBC 1	Surface Water	1) Define background water quality conditions in MFLBC.	1) Collect one collocated sample at the upgradient location. 2) 100%(1) completeness goal 3) SAS low detection levels. (See Section 9)
Off-Site Soil (background)	Soil	1) Define natural or background soil conditions. 2) Provide remedial design Data	1) One collocated sample from one boring. 2) 100%(1) completeness goal.
MFLBC 1	Fish	1) Define background conditions	1) 100%(1) completeness goal.
Slanker Pond	Surface Water	1) Ensure acceptable water quality	1) 100%(1) completeness goal.

(1) Data with <100% completeness will be acceptable as long as the deficiency does not hinder project objectives.

development and validation study design and its completion will both be submitted for U.S. EPA and OEPA approval before investigation activities begin.

2.0 SCOPE AND LIMITATIONS

This HSP establishes responsibilities and procedures for the health and safety program to be followed during the remedial investigation at the Ruetgers-Nease Site in Salem, Ohio. This plan has been designed to satisfy the OSHA requirements specified in 29 CFR-1910.120.

Every potential safety hazard associated with this investigation cannot be predicted. This HSP does not attempt to establish rules to cover every contingency which may arise, but is intended to provide a basic framework for the safe completion of field activities planning for reasonable contingencies. The procedures provided herein are to be used by all contractor and subcontractor employees who will be involved in the performance of the project. All personnel are required to enforce and adhere to the established rules specified in the approved HSP which will be made available to them.

On-site subcontractors shall adopt this HSP, and shall complete all work in accordance with the plan.

3.0 ORGANIZATION AND RESPONSIBILITIES

The organization and responsibilities for implementing this HSP are described in this section. Safety responsibilities must be incorporated into project management roles to ensure proper program implementation. Additionally, all persons participating in this investigation must be aware of the potential hazards involved and assume appropriate responsibilities to protect themselves and others. A well-defined organizational structure is an important factor for instilling a strong safety ethic into remedial investigations. Figure 3-1 illustrates the organizational structure for this project. Responsibilities as they impact health and safety are described below. Other responsibilities of the project team are described in the other volumes of the Work Plan. This plan may be amended only with the approval of the U.S. EPA and OEPA.

3.1 U.S. EPA RPM and OEPA Project Coordinator

Amy Blumberg is U.S. EPA's Region 5 Remedial Project Manager and Susan MacMillan is OEPA's Project Coordinator for this RI/FS. Their responsibilities include:

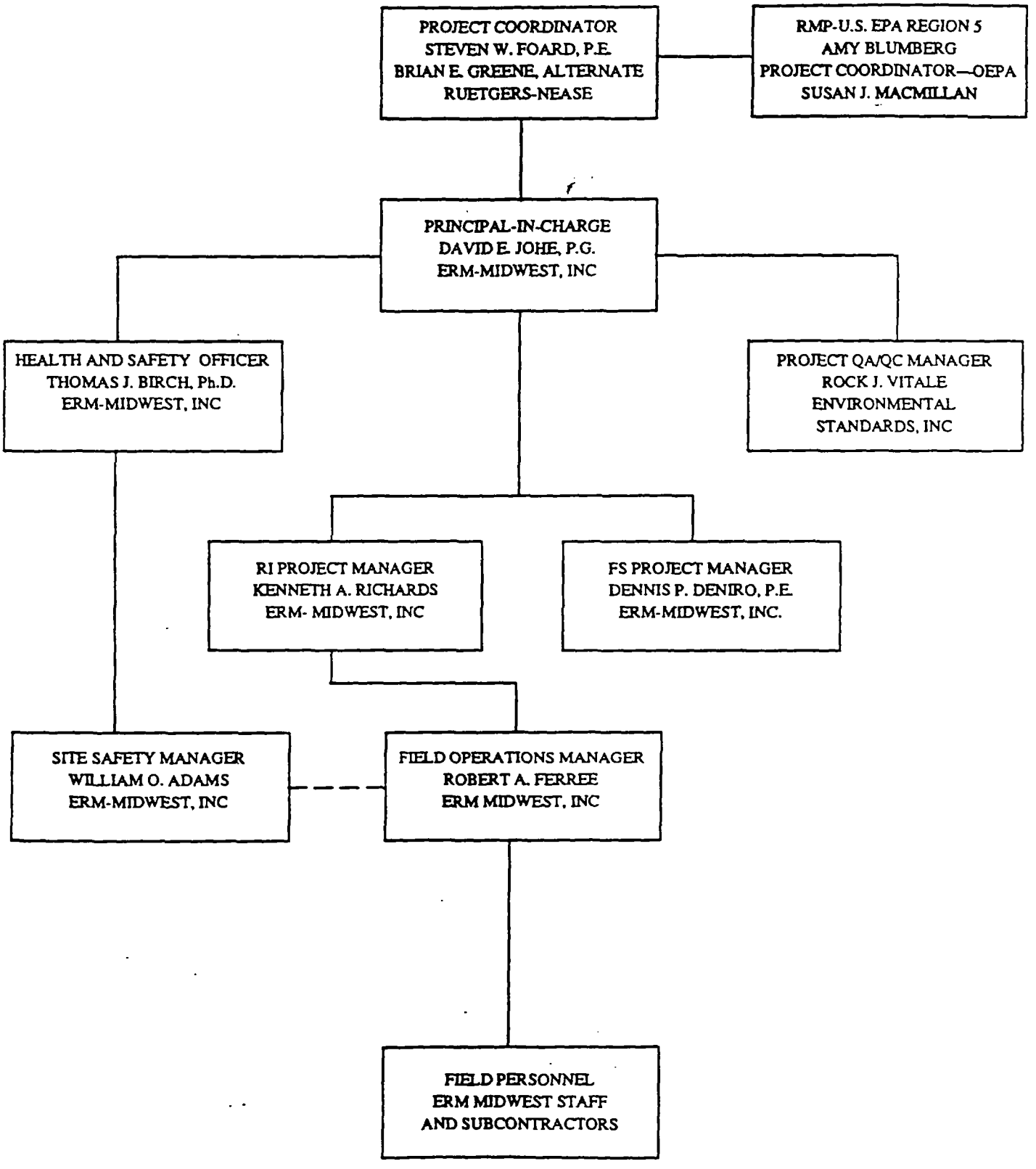
1. Technical review and approval of all plans and data submitted as part of the RI/FS.

2. Coordination of RI/FS activities with the Project Coordinator.

3. Overseeing and approving amendments to the HSP.

FIGURE HSP 3-1

HEALTH AND SAFETY ORGANIZATION
RUETGERS-NEASE SALEM SITE RI/FS



REVISED 12.89

ERM MIDWEST, INC

3.2 Project Coordinator

Steven W. Foard, P.E., of Ruetgers-Nease is the Project Coordinator for this investigation. The Project Coordinator is responsible for recommending and requesting modifications to this HSP for approval by both U.S. EPA and OEPA. Brian E. Greene of Ruetgers-Nease is the Alternate Project Coordinator.

3.3 Principal-in-Charge

The Principal-in-Charge (PIC) for the project is David E. Johe, of ERM-Midwest. The PIC is responsible for providing the ERM-Midwest resources necessary to implement the provisions of this HSP and to enforce the ERM-Midwest commitment to health and safety.

3.4 Project Manager

The RI Project Manager is Kenneth A. Richards, of ERM-Midwest. The RI Project Manager is responsible for coordinating field activities so that project objectives are met without compromising health and safety. This responsibility includes identifying the resources necessary to complete project activities in accordance with the provisions of the HSP and other project plans.

3.5 Field Operations Manager

The Field Operations Manager will be Robert A. Ferree of ERM-Midwest or designated alternate. He will be responsible for the overall implementation and monitoring of the HSP by:

1. Assuring that personnel are aware of the provisions of the HSP.

2. Assuring that appropriate personnel-protective equipment is available and properly used by all personnel.

3. Assuring that personnel are aware of potential hazards associated with the project.

4. Correcting any work practices that may result in injury or exposure to hazardous substances.

5. Maintaining communications with the Health and Safety Officer and Site Safety Manager.

6. Conducting daily safety briefings.

7. Assisting Site Safety Manager with ambient air monitoring.

3.6 ERM-Midwest Project Health and Safety Officer

ERM-Midwest's Health and Safety Officer is Thomas J. Birch, Ph.D. He is responsible for the overall coordination of safety matters at ERM-Midwest. His responsibilities include arranging safety training programs, evaluating new procedures, providing a follow-up investigation on corrective actions, and generally monitoring the hazardous waste site investigation safety program. He also is responsible for ensuring that proper safety equipment and clothing are available and in working order, and providing a liaison to field teams at the Site.

He will coordinate the health and safety program at the Site and will be responsible for the following:

1. Advising on health and safety policy issues.

2. Providing guidance on operational and logistical options.

3. Ensuring that health and safety training programs are available and that all personnel have received required training.

4. Monitoring the effectiveness of the HSP through review of field operation audits conducted by the Site Safety Manager.

5. Following up on necessary corrective actions.

6. Recommending the stoppage of work should Site conditions warrant such action.

7. Ensuring and verifying that all employees at the work site are being monitored under appropriate medical surveillance, hearing conservation, and respiratory protection programs.

8. Recommending proper and necessary precautions to take or work limitations required to reduce heat or cold stress.

3.7 Site Safety Manager

The Site Safety Manager (SSM) or designee for the project will be William O. Adams of ERM-Midwest. The SSM is responsible for the safety of field personnel at the Site.

This responsibility includes determining hazards associated with individual investigation phases, assisting the Project and Field Operations Managers in developing this HSP, reviewing safety matters during field operations, and notifying the Site Manager of any unsafe conditions or practices. The SSM will be responsible for the following:

1. Ambient air monitoring.

2. Assuring that all personal protection equipment and clothing are available and in proper working order at the Site.

3. Completing project safety briefings and reports.

4. Ensuring Health and Safety program compliance by conducting field audits.

5. Investigating accidents, implementing appropriate corrective actions, and preparing accident/incident reports.

6. Supervising and monitoring the safety performance of all personnel by conducting field audits to ensure that the required work practices are employed.

7. Verify that all personnel have received required health and safety training.

The SSM works with the Field Operations Manager to coordinate activities such that project objectives are met without compromising health and safety. On matters of health and safety, the SSM reports to the ERM-Midwest Project Health and Safety Officer.

3.8 Field Personnel

All field personnel will report directly to the Field Operations Manager and will be required to:

1. Be familiar with, and conform to, provisions of the HSP and complete the Health and Safety Plan Acceptance Form (Appendix B) prior to work in the work exclusion area at the Site.

2. Report accidents and/or hazardous conditions to the Field Operations Manager and the Site Safety Manager.

3. Have complete familiarity with their job requirements and the health and safety procedures involved.

All field personnel will be trained according to the OSHA standards set forth in 29 CFR 1910.120, and should be able to identify potential hazards if they arise.

4.0 SITE HAZARD EVALUATION

The purpose of the Hazard Evaluation is to determine the hazards that exist or potentially exist at the Site and to specify levels of protection and site boundaries necessary to limit risks to the local population, site workers, and the environment.

4.1 Health Effects

The possible presence of volatile organic and non-volatile organic compounds (Table 4-1) is the major health concern posed by the Site. Compounds possibly present include known or suspected carcinogens, mutagens, teratogens, and poisons. Protecting people from exposure to these substances by inhalation, oral ingestion, or dermal absorption is one of the primary purposes of this plan.

The potential inhalation and explosive hazards are shown in Table 4-1. Also shown in Table 4-1 are the OSHA Permissible Exposure Limit (PEL), threshold limit values (TLV) for the Time Weighted Average (TWA) and the Short-Term Exposure Limit (STEL), the Immediately Dangerous to Life or Health (IDLH) level, the Lower Explosive Limit (LEL), and the Upper Explosive Limit (UEL), for the possible volatile organic hazards. Symptoms of overexposure, possible chronic effects, and first aid treatment for the compounds found at the Site are provided in Appendix A.

The Project Health and Safety Officer and the Site Safety Manager are responsible for determining the level of personal protection equipment required using FID and PID instrumentation to compare to pre-established levels of protection. When work-site conditions warrant, the Site

TABLE HSP 4-1

HEALTH AND SAFETY LIMITS FOR POSSIBLE HAZARDOUS COMPOUNDS
RUETGERS-NEASE SALEM SITE RI/FS

VOLATILE ORGANIC COMPOUNDS

Compounds	OSHA PEL(2)	TLV(1) TWA(3)	TLV(1) STEL(4)	IDLH(5)	LEL(6)	UEL(7)	Incompatibles
1,1-Dichloroethane 75-34-3	100 ppm	200 ppm	250 ppm	4,000 ppm	6%	16%	Strong oxidizers and caustics
1,2-Dichloroethene	200 ppm	200 ppm	NA	4,000 ppm	9.7%	12.8%	Strong oxidizers and caustics
Chloroform 67-66-3	50 ppm (ceiling)	10 ppm	NA	1,000 ppm	NC	NC	Strong caustics, chemically active metals such as aluminum, magnesium, sodium, potassium
1,2-Dichloroethane 107-06-2	50 ppm	10 ppm	NA	1,000 ppm	6.2%	16%	Strong oxidizers, strong caustics chemically active metals such as aluminum, magnesium, sodium, potassium
1,1,1-Trichloroethane 71-55-6	350 ppm	350 ppm	450 ppm	500 ppm	7%	16%	Acids
1,2 Dichloropropane Method 1013 78-87-5	75 ppm	75 ppm	110 ppm	2,000 ppm	3.4%	14.5%	Incompatible with strong oxidizers and acid
* 1,3 Dichloropropene (skin) 542-75-6	NA	1 ppm	NA	NA	NA	NA	

TABLE HSP 4-1 (cont'd)

HEALTH AND SAFETY LIMITS FOR POSSIBLE HAZARDOUS COMPOUNDS
RUETGERS-NEASE SALEM SITE RI/FS

VOLATILE ORGANIC COMPOUNDS

Compounds	OSHA PEL(2)	TLV(1) TWA(3)	TLV(1) STEL(4)	IDLH(5)	LEL(6)	UEL(7)	Incompatibles
Trichloroethylene 79-01-6	100 ppm 200 ppm (c)	50 ppm	200 ppm	1,000 ppm	11%	41%	Strong caustics, chemically active metals such as aluminum, barium, lithium, sodium, magnesium, titanium
Benzene 71-43-2	1 ppm 25 ppm (c) 50 ppm (stel)	10 ppm	NA	2,000 ppm	1.3%	7.1%	Strong oxidizers, chlorine, bromine with iron
* 1,1,2,2 Tetra-chloroethene	100 ppm	50 ppm	200 ppm	500 ppm	NC(8)	NC(8)	Chemically active metals such as barium, lithium, beryllium; strong oxidizers
1,1,2,2 Tetrachloroethane (skin) 79-34-5	5 ppm	1 ppm	NA	150 ppm	NC(8)	NC(8)	Chemically active metals, strong caustics; hot iron, aluminum, zinc in presence of steam
Toluene 108-88-3	200 ppm	100 ppm	150 ppm	2,000 ppm	1.3%	7.1%	Strong oxidizers
Chlorobenzene 108-90-7	75 ppm	75 ppm	NA	2,400 ppm	1.3%	7.1%	Strong oxidizers
Ethyl benzene 100-41-4	100 ppm	100 ppm	125 ppm	2,000 ppm	1.0%	6.7%	Strong oxidizers

TABLE HSP 4-1 (cont'd)

HEALTH AND SAFETY LIMITS FOR POSSIBLE HAZARDOUS COMPOUNDS
RUETGERS-NEASE SALEM SITE RI/FS

VOLATILE ORGANIC COMPOUNDS

Compounds	OSHA PEL(2)	TLV(1) TWA(3)	TLV(1) STEL(4)	IDLH(5)	LEL(6)	UEL(7)	Incompatibles
o,m,p - xylene	100 ppm	100 ppm	150 ppm	10,000 ppm	1.1%	7.0%	Strong oxidizers
o - Dichlorobenzene 95-50-1	50 ppm(c)	50 ppm	NA	1,700 ppm	2.2%	9.2%	Strong oxidizers, hot aluminum or aluminum alloys
m,p - Dichlorobenzene 106-46-7	75 ppm	75 ppm	110 ppm	1,000 ppm	2.5%	unknown	none
NON-VOLATILE ORGANIC COMPOUNDS							
Methoxchlor	15 mg/m3	10 mg/m3	NA	7,500 mg/m3	NA	NA	Strong oxidizers
Mirex	NA	NA	NA	NA	NA	NA	Acids
3,4 Dichloronitro-benzene	NA	NA	NA	NA	NA	NA	Oxidizers, aluminum, liquid oxygen, potassium, sodium
Diphenyl sulfone DPS	NA	NA	NA	NA	NA	NA	NA

TABLE HSP 4-1 (cont'd)

HEALTH AND SAFETY LIMITS FOR POSSIBLE HAZARDOUS COMPOUNDS
RUETGERS-NEASE SALEM SITE RI/FS

VOLATILE ORGANIC COMPOUNDS

FOOTNOTES:

1. Threshold Limit Value.
2. Permissable exposure limit (from NIOSH/OSHA).
3. Time weighted average (from ACGIH).
4. Short term exposure limit (from ACGIH).
5. Immediately dangerous to life or health (from NIOSH/OSHA).
6. Lower explosive limit, in air (from NIOSH/OSHA).
7. Upper explosive limit, in air (from NIOSH/OSHA).
8. If temperatures are hot enough, compound will breakdown into new hazardous substances.

NA = Not available
NC = Noncombustible
* = Values based on a value for similarly structured compound.

Sources: NIOSH Pocket Guide to Chemical Hazards 1985
ACHIH, 6500 Glenway Avenue, Bldg. D-7, Cincinatti, OH 45211-4438
Rapid Guide to Hazardous Chemicals in the Workplace, Irving A and Richard J. Lewis, Sr. Von Notrand Reinhold Company, NY 1986
Dangerous Properties of Industrial Materials, Von Nostrand Reinhold Company, NY 1984
OSHA Region III Office, Philadelphia, PA 1987, Ms. Jones, I.H.

Safety Manager will modify the level of protection to be utilized in the field after consulting with the Project Health and Safety Officer and/or the Field Operations Manager and receiving U.S. EPA/OEPA approval. The discovery of any condition that would suggest the existence of a situation more hazardous than anticipated will result in the suspension of work until the Health and Safety Officer, Site Safety Manager and/or the Project Manager has been notified and appropriate instructions have been provided to the SSM or Field Operations Manager.

5.0 PERSONNEL PROTECTION AND HEALTH AND SAFETY GUIDANCE FOR SPECIFIC TASKS

Personnel protection and procedures for specific tasks to be conducted during this study are discussed in this section.

All personnel must wear appropriate protective equipment and follow standard operating procedures during field investigations. Protective clothing shields the skin from contact with hazardous chemicals, respirators protect the lungs and respiratory system (and eyes, when full-face respirators are worn), safety eye glasses/goggles protect the eyes, and good personal hygiene limits or prevents ingestion/absorption or inhalation of foreign materials. Standard operating procedures minimize the potential for accident and/or injury.

Levels of protection for specific field activities will be reviewed throughout this project. The levels of protection contained herein are to be considered requirements based upon FID and/or PID readings. At the Site, air quality monitoring will be conducted and will be used as supportive information in the establishment of appropriate levels of personnel protection.

5.1 Standard Operating Procedures

During this investigation, all personnel must adhere to the following rules:

1. During Site operations, the buddy system must be used to allow workers to act as safety backups to each other. Hand signals will be established prior

to the commencement of field activities. Off-site personnel are to provide emergency assistance.

2. All personnel should be aware of the dangerous situations which may develop because of the physical constraints imposed by personal protection equipment.

3. Visual or two way radio contact must be maintained between groups on-site or off-site.

4. Eating, drinking, chewing gum or tobacco, smoking, or any practice that increases the probability of hand-to-mouth transfer and ingestion of material is prohibited in the exclusion and contaminant reduction zones.

5. Medicine and alcohol can increase the effects from exposure to toxic chemicals. Prescription drugs should not be taken by personnel where the potential for contact with toxic substances exists, unless specifically approved by a qualified physician. Alcohol intake is prohibited during the work shift.

6. Facial hair (including beards and long sideburns) which interferes with the respirator to face seal is not allowed on personnel required to wear or potentially wear respiratory protection equipment. Each staff member must pass a qualitative or quantitative fit test for respirators.

7. Contact lenses will not be permitted at the Site. If glasses are required, prescription or "plano" safety glasses meeting the requirements of ANSI Z87.1-1979 standards must be worn. Employees will

be fitted with spectacle kits for use with full-face respirators when necessary.

8. Disposable clothing will be used whenever possible to minimize the risk of contamination and disposed of as noted in Section 9 of this HSP.

9. Procedures for leaving any contaminated area will be planned and implemented prior to going on-site. Exclusion areas, contamination reduction areas, and decontamination procedures will be established (see Section 9.0).

10. Contact with contaminated or potentially-contaminated surfaces should be avoided. Whenever possible, do not walk through puddles, mud, or any discolored ground surface; do not kneel on the ground, lean, sit or place equipment on drums, containers, vehicles or on the ground.

11. Air monitoring will be conducted on an on-going basis. Ambient air quality can change rapidly during subsurface excavations.

12. No personnel will be admitted to the exclusion and contamination reduction areas without the proper safety equipment, OSHA training and medical clearance.

13. Proper decontamination procedures must be followed before leaving the Site (see Section 7.0).

14. All personnel must comply with established safety procedures. Any person who does not comply with the established safety policy will be immediately dismissed from the Site.

15. Any medical emergency supersedes routine safety requirements (see Section 10).

5.2 Protective Equipment Selection and Limitation

At this time, conditions mandating Level A or B protection are not anticipated at the Site. Should such conditions arise, the immediate work area will be evacuated and the situation reviewed.

5.2.1 Level A

If VOC concentrations pose significant (IDLH or serious acute risks) health hazards and all alternatives exhausted as determined by the Project Health and Safety Officer and Project Manager, an upgrade to Level A protective equipment will be necessary. This upgrade is not anticipated and will only be used as a last resort when all other means have been exhausted. Upgrading to Level A will only be necessary when full encapsulation of the individual is required.

5.2.1.1 Protective Equipment

1. Full Face pressure-demand SCBA
2. Fully-encapsulated chemical resistant suit
3. Inner chemical-resistant gloves
4. Two way radio communication
5. Cooling/heating unit (if appropriate)
6. Coveralls

7. Long cotton underwear (if appropriate)

8. Hard hat

9. Steel-toed shoes

10. Disposable gloves and boot covers

5.2.1.2 Selection Criteria

Level A contaminant concentrations that warrant the need for total encapsulation as determined by the Project Health and Safety Officer and Site Safety Manager.

5.2.2 Level B

If VOC concentrations are greater than 20 ppm as detected by FID and/or PID instruments, Level B personal protective equipment will be required. Based on previous site investigations, Level B protection is not anticipated during remedial investigation activities.

A 20 ppm level is established based upon:

1. Chemical odor thresholds

2. Breakthrough characteristics associated with chemical absorption and penetration through an organic vapor cartridge.

3. Full face piece protection factor of 50X

4. A random 50% safety factor or margin due to unknown synergists effects of the potential chemicals identified in the sludge.

Twenty parts per million (20 ppm) is well below the OSHA and ACGIH established STEL and IDLH values for the chemicals listed in Table 4-1.

5.2.2.1 Protective Equipment

1. Full-face SCBA (self-contained breathing apparatus) or pressure-demand supplied-air respirator with 5- minute escape pack

2. Poly-coated chemical-resistant coveralls (e.g., tyvek overalls)

3. Chemical resistant outer gloves (taped to sleeves) and inner gloves

4. Outer disposable boots (taped to cuffs)

5. Hard hat and steel toe boots

6. Available 5-minute escape packs

5.2.2.2 Selection Criteria

1. Atmospheric contaminant concentrations have exceeded Level C criteria and the SSM has deemed Level B protection necessary.

2. Level A protection will be implemented should total encapsulation of personal be deemed necessary.

5.2.3 Level C

Based on preliminary air monitoring and previous field investigations at the Site, most activities conducted within the exclusion zone should be conducted in Level C.

During the performance of field activities, such as test pit excavation, well drilling, and soil boring, on-site work areas will be considered work exclusion zones. These work exclusion zones will surround the location of the field activity. Air monitoring equipment will be used to determine the level of protection needed in the work area. All personnel entering a work exclusion zone will be required to use the level of protection that has been deemed appropriate.

Level C safety equipment will be required when entering a work area in the exclusion zone if VOC concentrations are greater than background levels and less than 20 ppm as measured with FIDs or PIDs.

Level C protective equipment will be worn when conditions within the exclusion zone will present a dust hazard. Non-volatile organics are present in the soil and may pose an inhalation and skin absorption hazard when dust particles are generated. The SSM and/or Field Operations Manager will determine if upgrade from Level D is warranted under these conditions.

5.2.3.1 Protective Equipment

1. Full-face air-purifying respirator with appropriate cartridges and/or Hepa filters to provide protection from acids, volatile and non-volatile organic vapors, and particulates.

2. Poly-coated chemical-resistant coveralls (e.g., tyvek overalls)

3. Chemical resistant outer gloves (taped to sleeves) and inner gloves

4. Outer disposable boots (taped to cuffs)

5. Hard hat and steel-toed boots

6. 5-minute escape packs (available)

5.2.3.2 Selection Criteria

1. Atmospheric contaminant above background concentration and not greater than 20 ppm.

2. Conditions are such that small exposed areas about the head and neck will not be contacted by hazardous substances that pose a significant skin adsorption hazard.

3. Service limit of respiratory cartridges will not be exceeded.

4. Atmosphere contains between 19.5-25 percent oxygen.

5. Atmosphere contains between background levels and 5 ppm volatile organics using FID and/or PID, or testing for specific compounds, indicating that appropriate exposure limits are not being exceeded.

5.2.4 Level D

Level D equipment will be worn in areas where contact with highly contaminated materials is unlikely. Based on preliminary air monitoring and previous field investigations at the Site, it is anticipated that all off-site field work will be conducted in Level D.

5.2.4.1 Protective Equipment

1. Full-face air-purifying respirator (readily available) with organic vapor and/or Hepa filter canisters.

2. Cotton work coveralls or chemical resistant coveralls (e.g., tyvek)

3. Hard hat and steel-toed boots

4. Protective gloves (optional)

5. Goggles or safety glasses

6. Outer disposable boots

7. Face shield as determined by SSO and Site Manager

5.2.4.2 Selection Criteria

Level D protection is the basic work uniform. It can be worn in areas where only boots can be contaminated, no visible toxic substances are present, and VOC concentrations are equal to or less than background levels or 0 ppm.

5.3 Special Procedures for Subsurface Activities

Off-site, special precautions are required since work areas are accessible to the public. The public will be kept from entering the drilling area. Off-site activities will be conducted using Level D protection. On-site, subsurface excavations and drilling may disturb contaminated materials. Therefore, access will be restricted at all sampling locations. At each borehole or excavation site, a work-zone exclusion area of at least a 25-foot radius will be established. On-site and off-site subsurface activities will be performed using appropriate levels of protection. Appropriate personnel protection levels will be confirmed at each location based on regular air monitoring using PIDs and FIDs. Hard hats, safety glasses, and steel-toed safety shoes will be worn during all subsurface and drilling activities.

Prior to beginning any subsurface activity, local utilities (gas, electric, water, sewer, telephone, etc.) must be contacted to determine if any lines are in the subsurface activity area. Blueprints from Ruetgers-Nease may also provide on-site information. This information must be documented.

5.4 Special Procedures for Test Pit Operations

Test pit operations require special safety precautions due to the potential for collapse of the test pit walls. Shallow test pits constructed in firm ground are generally stable and do not require wall sheeting; however, pits which are excavated in unstable soil or those which extend to depths beyond the range of four to five feet may require sheeting if personnel will enter the pit. The type and extent of sheeting required will be determined, based on

field conditions, by the SSM. All test pit operations must conform to the OSHA standard 29 CFR 1926. subpart P, Excavations, Trenching and Shoring. Under no conditions should field personnel enter a test pit, or a sheeted pit unless the person has obtained a confined space entry permit from Field Operations Manager or SSM. Hard hats, safety glasses, and steel-toed shoes or boots are to be worn at all times during test pit excavations. Appropriate personal protection levels will be determined by the SSM or Field Operations Manager at each pit location based upon criteria established in Section 5.2.

Chemical vapors may become trapped at the bottom of a test pit. Therefore, personnel entering such a pit may have to follow confined space procedures. These procedures include testing the atmosphere in the pit with a combustible/toxic/O_2 meter (explosimeter) and following the buddy system (one person on top of pit to watch the person below who is attached with a safety harness and life line).

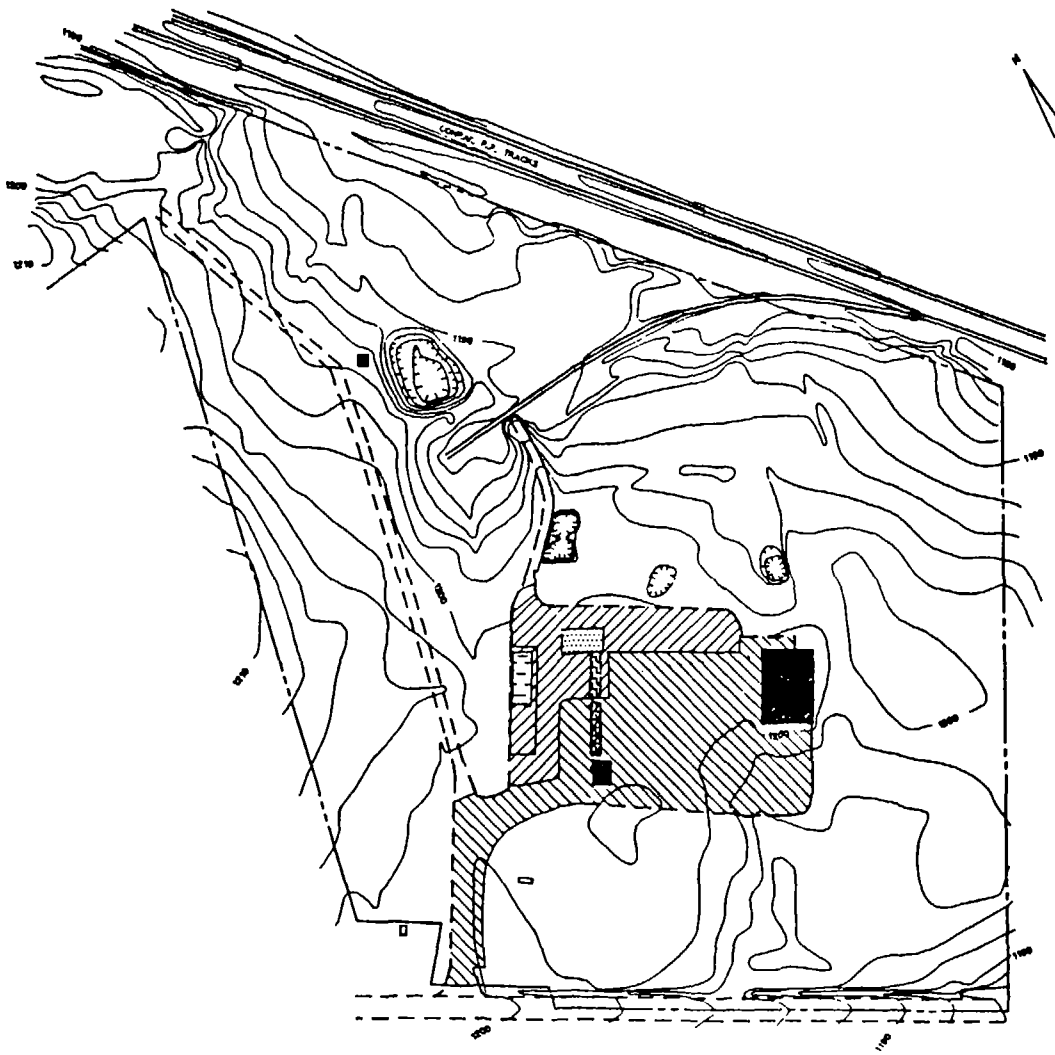
6.0 SITE CONTROL

The Site will be divided into three specific zones to control site activities: Zone 1, exclusion zone (EZ); Zone 2, contamination reduction zone (CRZ); and Zone 3, support zone (SZ) as shown in Figure 6-1. These three zones have been established on the basis of contamination potential ranging from the potentially highest levels of contamination in the exclusion zone to little or no contamination potential in the support zone. The CRZ and SZ will be located at the clear areas along the tiled cement and concrete areas. The EZ will encompass the remainder of the site. Access to the EZ will be limited. The boundaries of these zones may be modified depending on such factors as prevailing winds and organic vapor levels.

Around each field activity such as test pit excavation, well drilling, and soil boring, a work exclusion area will be defined. The boundaries of such a work exclusion area will surround the location of the field activity. Within the boundaries of the work area, air monitoring equipment will be used to determine the level of protection needed in the work exclusion area. All personnel entering a work exclusion area will be required to use the level of protection that has been deemed appropriate.

6.1 Access-Personnel

Only authorized personnel will have access into the appropriate zones as deemed necessary by the Field Operations Manager and Site Safety Manager.



LEGEND

SUPPORT ZONE

CONTAMINANT REDUCTION ZONE

DECONTAMINATION

OFFICE TRAILER

DECONTAMINATION TRAILER

PERSONNEL DECONTAMINATION ZONE

EXISTING BUILDING

NOTE: ALL OTHER ON-SITE AREAS ARE WITHIN THE EXCLUSION ZONE.

SCALE

0 60 120 240 (FEET)

CONTOUR INTERVAL = 2 FT

FIGURE HSP 6 - 1

EXCLUSION AREAS

RUETGERS-NEASE
SALEM SITE RI/FS

REVISED 10.88

ERM-Midwest, inc.

6.2 Security

A fence has been installed to deter unauthorized personnel from trespassing. Access to the EZ will be from the on-site decontamination pad and personnel decontamination trailer. In addition, signs have been placed on the Site fence stating the following:

Danger

Unauthorized Persons Keep Out

Outside the exclusion zone, there will be trailers for office support and decontamination purposes. All personnel entering the site will sign in and out in the visitor/employee log book which will be maintained in the office trailer.

6.2.1 Communications

A telephone will be installed in the office support trailer. Emergency telephone numbers contained in Section 10.0 will be posted next to the phone.

At the remote areas of the site, in the EZ or the CRZ zones,and at off-site field activities, communication will be maintained by two-way radios. The radio main base will be in the support trailer or with the Field Operations Manager.

6.2.2 Administrative Actions for Violations

Personnel who violate the provisions specified in the HSP will have their actions documented and submitted to their company's supervisor for appropriate disciplinary action.

The document generation and liaison actions will be done by the Project Manager.

If unauthorized personnel enter the area and are not willing to leave the area peacefully, the Field Operations Manager will call the police to remove the trespasser.

6.3 Training Verification

The Site Safety Manager will verify all training of individuals proposed for participation in the investigation at the Site by requesting signed copies of training certificates or official certified copies of transcripts. These transcripts will be submitted to ERM-Midwest for verification. If there are questions of invalid documents, ERM-Midwest will contact training centers by written correspondence and telephone notification. Personnel will not be permitted to enter the CRZ, EZ, or off-site work exclusion areas without the verified training.

7.0 MONITORING EQUIPMENT

The safety equipment which will be used at the Site will include: 1) air monitoring equipment to determine hazardous or potentially hazardous conditions at the Site; 2) personnel protection equipment to properly protect workers during site activities; and 3) emergency equipment in case of emergency incidents at the Site. All safety equipment calibration record forms and data records will be completed and in the document control file at ERM-Midwest.

7.1 Air Monitoring

Air monitoring equipment is used to determine if hazardous conditions exist. Air quality will be regularly monitored to determine the level of volatile organics in the air, the concentration of combustible gases, the concentration of oxygen in the atmosphere, and the presence of toxic gases. Measurements will be recorded on an ongoing basis and more frequently if fluctuations in the measured levels are observed. To ensure that all monitoring instruments are operating properly, a field calibration will be performed and documented as detailed in Section 4 in Volume 3.

7.1.1 Organic Vapors

Air quality will be monitored during site activities by the Site Safety Manager or designated personnel for the presence of volatile organics. The flame ionization detector (FID) will be used as the primary instrument to measure

organic vapors. It is important to note that FID readings should be considered approximate; these values must be used with discretion. A photoionization detector (PID) meter will be used to measure organic vapors if the FID meter is not available.

7.1.2 Oxygen, Combustible Gases, and Toxic Gases

In conjunction with the FID and/or PID meter, readings will be collected at each work site for the presence of oxygen (percentage), combustible gases, and toxic gases. These readings will be displayed and collected from an explosimeter or another appropriate piece of air monitoring equipment.

7.2 Emergency Equipment

7.2.1 Fire Extinguishers

Although the probability of fire at the site is low, fire extinguishers will be readily available throughout the investigation. All fire extinguishers will be Class ABC type. One fire extinguisher will be kept in each field vehicle during any subsurface activity such as drilling or backhoe excavations, and also in the support and decontamination trailers.

7.2.2 First Aid

An industrial first aid kit with sufficient supplies for ten (10) people will be kept in the decontamination trailer. First aid kits will be kept in all field vehicles used at the Site.

An eye wash station will be maintained at the decontamination trailer. Portable eye wash kits with hose (for emergency irrigation of other body parts) will also be kept with the field crews. All eye wash stations will be inspected for water flow and operability. In addition, portable eye wash kits will be checked on a monthly basis.

7.3 Personnel Protection Equipment

The following equipment is required to be available to all personnel at the Site:

1. Full-face respirators
2. Cover lenses and spectacle kits for full-face respirators
3. Safety Glasses and/or Chemical Splash Goggles
4. Respirator cartridges-organic vapor and HEPA filters
5. Vinyl gloves
6. Nitrile latex gloves
7. Coveralls, (e.g., Tyvek, uncoated)
8. Coveralls, (e.g., Tyvek, polycoated)
9. Disposable rubber outer boots
10. Goggles, unvented
11. Hard hats

12. Duct tape (to secure sleeves to gloves and cuffs to boots)

13. Emergency air pack (five minute escape pack e.g., ELSA)

14. Hearing protective ear muffs and/or plugs

15. Rain gear

8.0 WORK LIMITATION LEVELS

Work limitations and conditions when work will be suspended at the site are discussed in the following sections.

8.1 Work Limitation Criteria

All jobs will be carried out as per the directions of the Field Operations Manager and Site Safety Manager.

8.1.1 Air Criteria

The Site Safety Manager or alternate designee is responsible for conducting air monitoring at the Site for Health and Safety purposes. Periodic air monitoring will be conducted during all field operations to determine limitations to work activities. Air monitoring will routinely analyzed for the following parameters:

1. Volatile organic compounds - Measured by a PID (photoionization detector) such as the HNU, or FID (flame ionization detector) such as the Century OVA.

2. Combustible gases - Measured by an explosimeter such as the Gastech GX-82 or a GX-86.

3. Oxygen concentration - Measured by a direct read meter such as the Gastech GX-82 or a GX-86

4. Toxic gases - Measured by a direct read meter such as the Gastech GX-82 or GX-86.

The following action levels or work limitations will be used during the investigation.

1. Volatile Organic Gases
   - Background levels - Level D personal protection
   - Background levels ND to 20 ppm - Level C personal protection
   - Greater than 20 ppm - Level B Personal Protection
   - 500 ppm - evacuate work area until the concentrations have been determined to be at safe levels
   - Total Encapsulation - Level A personal protection

2. Oxygen
   - Less than 19.5% - evacuate work site
   - 19.5-25% - routine operations as applicable, monitor other parameters
   - Greater than 25% - evacuate work site, atmosphere may be explosive

3. Toxic Gases
   - Action levels will be determined by SSM as related to individual toxic gases (ex. Carbon monoxide, Hydrogen sulfide)

4. Combustible gases
   - 0-10% of LEL - routine operations, monitor other parameters
   - 10-20% of LEL - proceed with caution, continue monitoring
   - Greater than 20% of LEL - evacuate work area until concentrations return to safe level

8.1.2 Extreme Weather Conditions

Cold-related illness, or cold stress, can occur when working in temperatures at or below freezing. An illness such as frostbite may cause severe injury to the skin and extremities. Unless the victim is obviously contaminated, decontamination should be minimized or omitted and treatment begun immediately. First aid and emergency procedures for handling cold-related disorders are described in Section 10.4.

In order to reduce the occurrence of cold stress, the following will be observed while conducting field activities unless the SSM approves a work rule exception:

1. Drilling and related activities will only be conducted in sustained daily temperatures at or above 25°F.

2. All work involving water will cease when sustained daily temperatures are below 25°F.

Heat-related illnesses, or heat stress, can occur at any time when restrictive protective clothing is worn. An illness such as heatstroke requires prompt treatment to prevent irreversible damage or death. Protective clothing may have to be cut off and decontamination may have to be minimized or omitted to allow treatment to begin immediately. First aid and emergency procedures for handling heat related disorders are described in Section 10.4.

In order to avoid the occurrence of heat stress, continuous physical work may be conducted generally below 86°F. The SSM will consider such factors as the type of protective equipment being worn and physical work being done and will modify the temperature limitations and/or the work

schedule accordingly to further minimize the risk of heat stress.

8.2 Site Evacuation

It is anticipated at this time that necessary evacuation will only involve the immediate evacuation of a work zone area.

8.2.1 Upwind Withdrawal

The Site Safety Manager or Field Operations Manager will determine a safe upwind location for withdrawal from the work zone. Withdrawal will be necessary in the following cases:

1. Work zone air quality concentrations contain hazardous concentrations of volatile organics (greater than 500 ppm), combustible gases (greater than 20 percent LEL), or oxygen percentage above 25% or below 19.5% safety limits for the level of protection being worn (see Section 8.1.1).

2. Occurrence of a minor accident. The victim will undergo decontamination procedures and be transported to a safe upwind location. Field operations will resume after first aid and/or decontamination procedures have been administered to the affected individual and the cause of the accident is identified and corrected by the SSM or Field Operations Manager.

3. Protective clothing, SCBA, and/or respirator malfunctions.

8.2.2 Evacuation of Site

The site will be evacuated in the following cases:

1. The air quality monitoring determines that a majority of the site atmosphere contains greater than 20 percent of LEL combustible gases, or greater than 500 ppm volatile organics, and less than 19.5% or greater than 25% oxygen.

2. A major accident or injury occurs.

3. Fire and/or explosion occurs.

When any of these site evacuation conditions are met, the Field Operations Manager will notify the workers to evacuate the site through two-way radio and/or three blasts from an air horn. All equipment will be turned off and the keys will be left in the vehicle ignitions. Gross decontamination may take place on site or may be omitted at the discretion of the Field Operations Manager. The Field Operations Manager will notify the U.S. EPA and the OEPA and a joint decision will be made whether or not to notify local residents of the emergency and to request emergency assistance from local authorities, if necessary. If the U.S. EPA and/or OEPA cannot be reached and the situation is imminent, the Field Operations Manager will make the decision independently.

9.0 DECONTAMINATION AND HAZARDOUS MATERIALS HANDLING PROCEDURES

The following procedures concerning decontamination and materials handling will be implemented to ensure the health and safety of the site personnel and the general public.

9.1 Decontamination Stations

Decontamination (decon) will take place at three different types of stations:

1. The on-site decon pad (15' x 30')

2. Temporary, off-site work zone decon areas

3. The on-site personnel decon trailer and personnel decon zone. (The decon area will be equipped with changing areas and showers.)

The on-site decon pad will lie within the contaminant reduction zone (see Figure 6-1). The pad will consist of a current concrete slab, with installed curbs along the slab perimeter to retain water (approximately 4 to 6 inches high), and a drainage system. Wash water generated at the decon pad will be collected and stored in an on-site tank for proper disposal. Heavy equipment such as drilling rigs, augers and drilling stems, support vehicles, and backhoe buckets will be decontaminated at the on-site decon pad.

In each off-site work area a temporary decon station will be set up. The primary function of these stations is for personnel decon and decon of sampling equipment. These temporary stations will consist of a plastic tarp placed upon the ground and supplies needed for personnel decon.

All solids (cuttings, soils, etc.) will be collected in 55-gallon drums and stored in the warehouse building pending disposal. All wash fluids and decon related fluids will be collected and stored in an aboveground tank or drums located on-site pending disposal.

The on-site personnel decon trailer and personnel decon zone are within the contaminant reduction zone (see Figure 6-1). The facilities within the decon trailer will include showers and changing areas. Water generated by showers will be collected and stored on-site until the level of contamination is determined and disposed of in accordance with level of contamination. The personnel decon zone consists of a concrete slab adjacent to the personnel decon trailer.

9.2 Equipment Decontamination Procedures

All equipment will be decontaminated prior to and after its use on-site. The following procedures will be used for decontamination of equipment.

9.2.1 Heavy Equipment

Drilling rigs, drilling equipment such as augers and drill stems, support vehicles, backhoe buckets, and any other heavy equipment that has come in contact with potentially contaminated materials will be decontaminated at the on-site decon pad. The procedure for decontamination of heavy equipment is as follows:

1. Remove all loose soil.

2. Steam or high pressure wash (using non-phosphate detergent).

3. Potable water rinse.

9.2.2 Small Sampling Equipment

Small equipment used for sampling includes soil collection equipment, portable electronic equipment, ground water and surface water collection equipment, and soil gas probes.

Equipment used to collect soil samples such as split spoons, shelby tubes, hand augers, stainless steel scoops/trowels, and compositing containers will be decontaminated prior to their initial use, between each sample and between each sample location. The procedure for decontaminating soil sampling equipment is as follows:

Inorganics	Organics
1. Non-phosphate detergent wash	1. Non-phosphate detergent wash
2. 0.1 N HCL rinse	2. Tap water rinse
3. Tap water rinse	3. Deionized/Distilled water rinse
4. Deionized water rinse	4. Methanol rinse
5. Air dry	5. Pesticide quality hexane rinse*
	6. Methanol rinse

Inorganics	Organics
	7. Four rinses with deionized/distilled water
	8. Air dry

* - The use of hexane for decontamination is highly discouraged since hexane is detectable by the volatile analysis (as a TIC). Residual amounts could result in mandatory sample dilutions, and useless data.

All cuttings, soils and fluids generated will be collected and stored on-site until the level of contamination is determined. Disposal will be appropriate for the level of contamination. All decontaminated soil sampling equipment will be stored in clean plastic sheeting. This equipment may be decontaminated at the sampling location.

Electronic equipment such as PIDs, FIDs, explosimeters, portable GCs, and portable pumps used for air sampling will be decontaminated prior to their initial use and at the end of each working day. The procedure for decontaminating electronic equipment is as follows:

1. Brush to remove particulate contamination.

2. Wipe down with a clean, damp cloth (deionized water).

3. Air dry.

Decontaminated electronic equipment will be wrapped in plastic and stored on a clean surface in the office trailer.

This equipment may be decontaminated in the personnel decon zone.

Equipment used for ground water and surface water sampling will be decontaminated before sampling activities begin and between each sample location if dedicated equipment is not used. Water sampling equipment includes pumps, hoses, glass beakers, and bailers. The procedure for decontaminating this equipment is as follows:

Inorganics

1. Non-phosphate detergent wash
2. 0.1 N HCL rinse
3. Tap water rinse
4. Deionized water rinse
5. Air dry

Organics

1. Non-phosphate detergent wash
2. Tap water rinse
3. Deionized/Distilled water rinse
4. Methanol rinse
5. Pesticide quality hexane rinse
6. Methanol rinse
7. Four rinses with deionized/distilled water
8. Air dry

All cuttings, soils and fluids generated will be collected and stored on-site until the level of contamination is determined. Disposal will be appropriate for the level of contamination. All ground water and surface water sampling equipment will be stored in plastic sheeting after is has been decontaminated. This equipment may be decontaminated at each water sample location.

The soil gas probe used during the soil gas survey will be decontaminated using the procedures specified below:

1. Remove loose soil.

2. Non-phosphate soap wash.

3. Potable water rinse.

4. Twice rinsed with deionized water rinse.

5. Field scan with PID and FID.

All cuttings, soils and fluids generated will be collected and stored on-site until the level of contamination is determined. Disposal will be appropriate for the level of contamination. Decontaminated soil gas probes will be stored in plastic sheeting. This equipment may be decontaminated at the soil gas sampling location.

9.2.3 Well Construction Supplies

Supplies used to construct wells will be decontaminated prior to being installed. These supplies include well screens, riser pipes, and outer casings. These supplies will be decontaminated with a steam cleaner at the on-site decon

pad. Decontaminated well supplies will be stored on clean plastic sheeting in the on-site warehouse.

9.3 Personnel Decontamination Procedures

The total personnel decontamination process is divided into a number of individual steps. Each step within the decon process has specific decon tasks that must be performed. Each step is performed at a separate decon "station." Personnel will proceed from station to station until the total decon process has been completed. The process of personnel decon will begin at the on-site personnel decon zone in exclusion area and finish at the on-site personnel decon trailer exit in the support zone.

Personnel decontamination of individuals working at off-site sampling locations will begin in a temporary, off-site decon station and will finish in the on-site personnel decon trailer. Personnel decontamination procedures will be dependent upon the level of protection that has been donned by the personnel. The following sections describe decon procedures for Level A through Level D protection.

9.3.1 Level A Decontamination

Station 1: Segregated Equipment Drop

Deposit equipment used on the site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment necessary is:

1. Containers of various sizes
2. Plastic liners
3. Plastic drop cloths

Station 2: Boot Cover and Glove Wash

Scrub outer boot covers and gloves with decon solution or detergent/water solution.

Equipment necessary is:

1. Container (20 to 30 gal)
2. Decon solution
3. Detergent/water solution
4. Two or three long-handle, soft-bristle brushes

Station 3: Boot Cover and Glove Rinse

Rinse off decon solution from Station 2 using copious amounts of water. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30 to 50 gal)
2. High-pressure spray unit
3. Potable water
4. Two or three long-handle, soft-bristle scrub brushes

Station 4: Tape Removal

Remove tape around boots first and then tape around gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30 to 50 gal)
2. Plastic liners

Station 5: Boot Cover Removal

Remove boot covers and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 6: Outer Glove Removal

Remove outer gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20 to 30 gal)
2. Plastic liners

Station 7: Suit/Safety Boot Wash

Thoroughly wash fully encapsulating suit and boots. Scrub suit and boots with long-handle, soft-bristle scrub brush and copious amounts of decon solution or detergent/water solution. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30-50 gal)
2. Decon Solution
3. Detergent/water solution
4. Two or three long-handle, soft-bristle scrub brushes
5. Small buckets
6. Sponges or cloths

Station 8: Suit/SCBA/Boot/Glove Rinse

Rinse off decon solution or detergent/water solution using copious amounts of water. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30-50 gal)
2. High-pressure spray unit
3. Potable water
4. Small buckets
5. Two or three long-handle, soft-bristle scrub brushes
6. Sponges or cloths

Station 9: Tank Change

If worker leaves Exclusion Zone to change air tank, this is the last step in the decontamination procedure. Worker's air tank is exchanged, new outer glove and boot covers donned, and joints taped. Worker returns to duty.

Equipment necessary is:

1. Air tanks
2. Tape

3. Boot covers
4. Gloves

Station 10: Safety Boot Removal

Remove safety boots and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 11: Fully Encapsulating Suit and Hardhat Removal

With assistance of helper (helper will be wearing protective suit, Tyvek and gloves), remove fully encapsulating suit and hard hat. Hang suits on rack or lay out on drop cloths.

Equipment necessary is:

1. Rack
2. Drop Cloths
3. Bench or stool

Station 12: SCBA Backpack Removal

While still wearing facepiece, remove backpack and place on table. Disconnect hose from regulator valve and proceed to next station.

The only equipment necessary is a table.

Station 13: Inner-Glove Wash

Wash with decon solution or detergent/water solution that will not harm skin. Repeat as many times as necessary.

Equipment necessary is:

1. Basin or bucket
2. Decon solution
3. Detergent/water solution
4. Small table

Station 14: Inner-Glove Rinse

Rinse with water. Repeat as many times as necessary.

Equipment necessary is:

1. Potable water
2. Basin or bucket
3. Small table

Station 15: Facepiece Removal

Remove facepiece. Deposit in container with plastic liner. Avoid touching face with fingers.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners

Station 16: Inner-Glove Removal

Remove inner gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 17: Inner-Clothing Removal

Remove clothing soaked with perspiration. Place in container with plastic liner. Do not wear inner clothing off the site, since there is a possibility that small amounts of contaminants have been transferred in removing fully encapsulating suit.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners

Station 18: Field Wash

Shower if highly toxic, skin-corrosive, or skin-absorbable materials are known or suspected to be present.

Equipment necessary is:

1. Potable water
2. Soap
3. Small table
4. Basin or bucket
5. Field Showers
6. Towels

Station 19: Redress

Put on clean clothes. A dressing trailer is needed in inclement weather.

Equipment needed

1. Tables
2. Chairs
3. Lockers
4. Clothes

9.3.2 Level B Decontamination

Procedure for Full Decontamination

Station 1: Segregated Equipment Drop

Deposit equipment used on the site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment necessary is:

1. Containers of various sizes
2. Plastic liners
3. Plastic drop cloths

Station 2: Boot Cover and Glove Wash

Scrub outer boot covers and gloves with decon solution or detergent/water solution.

Equipment necessary is:

1. Container (20-30 gal)
2. Decon solution

3. Detergent/water solution
4. Two or three long-handle, soft-bristle scrub brushes

Station 3: Boot Cover and Glove Rinse

Rinse off decon solution from Station 2 using copious amounts of water. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30-50 gal)
2. High-pressure spray unit
3. Potable water
4. Two or three long-handle, soft-bristle scrub brushes

Station 4: Tape Removal

Remove tape around boots first and then remove tape around gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 5: Boot Cover Removal

Remove boot covers and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 6: Outer Glove Removal

Remove outer gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 7: Safety Boot Wash

Thoroughly wash splash suit and safety boots. Scrub with long-handle, soft-bristle scrub brush and copious amounts of decon solution or detergent/water solution. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30-50 gal)
2. Decon solution
3. Detergent/water solution
4. Two or three long-handle, soft-bristle scrub brushes

Station 8: Safety Boot Rinse

Rinse off decon solution or detergent/water solution using copious amounts of water. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30 to 50 gal)
2. High-pressure spray unit
3. Potable water
4. Two or three long-handle, soft-bristle scrub brushes

Station 9: Tank Change

If worker leaves Exclusion Zone to change air tank, this is the last step in the decontamination procedure. Worker's air tank is exchanged, new outer gloves and boot covers donned, and joints taped. Worker then returns to duty.

Equipment necessary is:

1. Air tanks
2. Tape
3. Boot covers
4. Gloves

Station 10: Safety Boot Removal

Remove safety boots and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 11: SCBA Backpack Removal

While still wearing facepiece, remove backpack and place on table. Disconnect hose from regulator valve and proceed to next station. If wearing a 5-minute escape, remove escape bottle and harness while still wearing facepiece.

The only equipment necessary is a table.

Station 12: Protective Suit Removal

With assistance of helper (helper will be wearing protective suit, Tyvek, and gloves), remove splash suit. Deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 13: Inner Glove Wash

Wash inner gloves with decon solution or detergent/water solution that will not harm skin. Repeat as many times as necessary.

Equipment necessary is:

1. Decon solution
2. Detergent/water solution
3. Basin or bucket
4. Small table

Station 14: Inner Glove Rinse

Rinse inner gloves with water. Repeat as many times as necessary.

Equipment necessary is:

1. Potable water
2. Basin or bucket
3. Small Table

Station 15: Facepiece Removal

Remove facepiece. Avoid touching face with gloves. Deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners

Station 16: Inner Glove Removal

Remove inner gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 17: Inner Clothing Removal

Remove clothing soaked with perspiration. Place in container with plastic liner. Do not wear inner clothing off the site since there is a possibility that small amounts of contaminants have been transferred in removing fully encapsulating suit.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners

Station 18: Field Wash

Shower if highly toxic, skin corrosive, or skin-absorbable materials are known or suspected to be present.

Equipment necessary is:

1. Potable Water
2. Soap
3. Small table
4. Basins or buckets
5. Field showers
6. Towels

Station 19: Redress

Put on clean clothes. A dressing trailer is needed in inclement weather.

Equipment necessary is:

1. Tables
2. Chairs
3. Lockers
4. Clothes

9.3.3 Level C Decontamination

Procedure for Full Decontamination

Station 1: Segregated Equipment Drop

Deposit equipment used on the site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different

containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment necessary is:

1. Containers of various sizes
2. Plastic liners
3. Plastic drop cloths

Station 2: Boot Cover and Glove Wash

Scrub outer boot covers and gloves with decon solution or detergent/water solution.

Equipment necessary is:

1. Container (20-30 gal)
2. Decon solution
3. Detergent/water solution
4. Two or three long-handle, soft-bristle scrub brushes

Station 3: Boot Cover and Glove Rinse

Rinse off decon solution from Station 2 using copious amounts of water. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30-50 gal)
2. High-pressure spray unit
3. Potable water
4. Two or three long-handle, soft-bristle scrub brushes

Station 4: Tape Removal

Remove tape around boots first and then remove tape around gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 5: Boot Cover Removal

Remove boot covers and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 6: Outer Glove Removal

Remove outer gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 7: Safety Boot Wash

Thoroughly wash safety boots. Scrub with long-handle, soft-bristle scrub brush and copious amounts of decon solution or detergent/water solution.

Equipment necessary is:

1. Container (30-50 gal)
2. Decon solution
3. Detergent/water solution
4. Two or three long-handle, soft-bristle scrub brushes

Station 8: Safety Boot Rinse

Rinse off decon solution or detergent/water solution using copious amounts of water. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30-50 gal)
2. High-pressure spray unit
3. Potable water
4. Two or three long-handle, soft-bristle scrub brushes

Station 9: Canister or Mask Change

If worker leaves Exclusion Zone to change canister (or mask), this is the last step in the decontamination procedure. Worker's canister is exchanged, new outer glove and boot covers donned, and joints taped. Worker returns to duty.

Equipment necessary is:

1. Canister (or mask)
2. Tape
3. Boot covers
4. Gloves
5. Container for used canisters

Station 10: Safety Boot Removal

Remove safety boots and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 11: Protective Suit Removal

With assistance of helper, (helper will be wearing protective suit, Tyvek, and glove) remove splash suit. Deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Bench or stool
3. Plastic liner

Station 12: Inner Glove Wash

Wash inner gloves with decon solution or detergent/water solution that will not harm skin. Repeat as many times as necessary.

Equipment necessary is:

1. Decon solution
2. Detergent/water solution
3. Basin or bucket

Station 13: Inner Glove Rinse

Rinse inner gloves with water. Repeat as many times as necessary.

Equipment necessary is:

1. Potable water
2. Basin or bucket
3. Small table

Station 14: Facepiece Removal and Wash

Remove facepiece. Avoid touching face with gloves. Deposit facepiece in container with plastic liner.

Equipment necessary is:

1. Decon solution
2. Potable water
3. Basin or bucket

Station 15: Face Piece Rinse

Rinse face piece with water, towel dry and place in sealable plastic bag.

Equipment necessary is:

1. Potable water
2. Basin or bucket
3. High pressure spray mist
4. Plastic bags
5. Towels
6. Used towel container

Station 16: Inner Glove Removal

Remove inner gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 17: Inner Clothing Removal

Remove clothing soaked with perspiration. Place in container with plastic liner. Do not wear inner clothing off the site since there is a possibility small amounts of contaminants have been transferred in removing fully encapsulating suit.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners

Station 18: Field Wash

Shower if highly toxic, skin-corrosive or skin-absorbable materials are known or suspected to be present. Wash hands and face if shower is not available.

Equipment necessary is:

1. Potable Water
2. Soap
3. Tables
4. Wash basins or buckets
5. Field Showers

6. Towels
7. Used towel container

Station 19: Redress

Put on clean clothes. A dressing trailer is needed in inclement weather.

Equipment necessary is:

1. Tables
2. Chairs
3. Lockers
4. Clothes

9.3.4 Level D Decontamination

Procedure for Full Decontamination

Station 1: Segregated Equipment Drop

Deposit equipment used on the site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment necessary is:

1. Containers of various sizes
2. Plastic liners
3. Plastic drop cloths

Station 2: Boot and Glove Wash

Scrub outer boot covers and gloves with decon solution or detergent/water solution.

Equipment necessary is:

1. Container (20-30 gal)
2. Decon solution
3. Detergent/water solution
4. Two or three long-handle, soft-bristle scrub brushes

Station 3: Boot and Glove Rinse

Rinse off decon solution from Station 2 using copious amounts of water. Repeat as many times as necessary.

Equipment necessary is:

1. Container (30-50 gal)
2. High-pressure spray unit
3. Potable Water
4. Two or three long-handle, soft-bristle scrub brushes

Station 4: Tape Removal

Remove tape around boots and gloves and deposit in container with plastic liner.

Equipment necessary is:

1. Container (20-30 gal)
2. Plastic liners

Station 5: Safety Boot Removal

Remove safety boots and deposit in container with plastic liner.

Equipment necessary is:

1. Container (30-50 gal)
2. Plastic liners
3. Bench or stool

Station 6: Protective Suit or Cotton Overalls Removal

With assistance of helper, remove protective suit or cotton overalls. Deposit in container with plastic liner.

Equipment necessary is:

1. Bench or stool
2. Plastic liner

Station 7: Field Wash

Shower if highly toxic, skin-corrosive, or skin-absorbable materials are known or suspected to be present. Wash hands and face if shower is not available.

Equipment necessary is:

1. Potable water
2. Soap
3. Tables
4. Wash basins or buckets
5. Field Showers
6. Towels

Station 8: Redress

Put on clean clothes. A dressing trailer is needed in inclement weather.

Equipment necessary is:

1. Tables
2. Chairs
3. Lockers
4. Clothes

9.4 Hazardous Materials Handling Procedures

This section concerns the off-site and on-site handling of hazardous materials.

9.4.1 Off-Site

Wells drilled and developed in the off-site areas will encounter potentially contaminated materials such as contaminated rock, soil, and water.

Before drilling a well, a plastic tarp will be placed around the intended drilling location to allow the cuttings to be collected once drilling has begun. These cuttings will be drummed and brought on-site to await proper disposal.

Exposed portions of drilling rigs that may contact potentially contaminated materials will be covered with plastic sheeting to prevent contamination of the vehicle's wheels during drilling. This will inhibit potentially contaminated soils from being deposited on public roadways while the rig is in transport to the on-site decon pad.

Drilling equipment such as augers may be wrapped in plastic sheeting prior to transport to the on-site decontamination area.

The water brought to the ground surface during well development procedures and Round 1 purging will be placed in containers, transported, and stored in a tank on site for proper disposal.

9.4.2 On-Site

Soil borings, test pit construction and well purging will be handled according to the following procedures.

The soil brought to ground surface during boring procedures will be replaced with grout once drilling of the boring is finished. All boring cuttings will be containerized.

Similarly, soil brought to the ground surface during test pit construction will be returned to the pit once excavation of the pit has finished. The soils removed from the test pit will be returned to the pit in the order reversed from the order in which it was removed from the pit. This will insure soils are returned to their original depth within the pit.

The water brought to the surface during the purging of on-site wells will be placed in containers, transported, and stored in a tank on site for proper disposal.

10.0 EMERGENCY PROCEDURES AND CONTACTS

The HSP for this project has been established to allow site operations to be conducted without adverse impacts on the worker or public Health and Safety.

10.1 General Medical Procedures

In the event of an emergency, appropriate corrective measures must immediately be taken to assist those who have been injured or exposed and to protect others from hazard. The Field Operations Manager should be immediately notified of the incident and, if necessary, first aid will be rendered.

Accident reports will be completed as necessary, returned to the Health and Safety officer, and kept in the document control file at ERM-Midwest's Health Services Department. These and other forms are described in Section 11.0 and can be found in Appendix B.

In life threatening situations, care must be instituted immediately without considering decontamination protocol. Outside protective clothing can be removed if it does not cause delays, interfere with treatment, or aggravate the problem. Respirators must always be removed. If outer contaminated garments cannot be safely removed, the individual should be wrapped in suitable material to help prevent contaminating ambulances and/or medical personnel. For minor medical problems or injuries, normal decontamination procedures should be followed when at all possible.

First aid or other appropriate initial action will be administered by those closest to the accident/unusual event.

This assistance will be conducted in a manner to assure that those rendering assistance are not placed in a situation of unacceptable risk. Listings of on-site Emergency Medical Technicians will be posted in prominent locations.

All OSHA-recordable injuries, accidents involving property damage, and unusual events must be reported to the Site Safety Manager or Field Operations Manager. Upon notification, the Site Safety Manager or Field Operations Manager will conduct an investigation and complete the Accident Report Form, attached in Appendix B. The Project Coordinator, Project Manager, and the Agencies' on-site representative will be informed of the accident or event within 24 hours of being reported to the Site Safety Manager or Field Operations Manager.

After reviewing the Accident Report Form, the Site Safety Manager will complete the Accident Investigation Information Report, Appendix B. The completed forms will then be filed at the site for review by regulatory personnel.

The Field Operations Manager will be responsible for insuring that all corrective actions identified by the Accident Investigation Information are implemented.

The Site Safety Manager or Field Operations Manager will decide if off-site assistance and/or medical treatment is required and he will arrange for assistance. Off-site coordination and assistance procedures will be prearranged with local ambulance companies, hospitals, and doctors or other medical specialists.

All workers on site are responsible for conducting themselves in a mature, calm manner in the event of an accident/unusual event. All personnel must conduct

themselves in a manner to avoid spreading the danger to themselves and to surrounding workers.

The following emergency equipment will be available on the site:

- First aid kit
- Fire extinguisher and blanket
- Cool wet towels or sheets
- Stretcher
- Emergency eyewash station
- Hand-held air horns
- Egress air packs
- Absorbent material
- Plentiful supply of potable water

10.2 Emergency Horn

All personnel will be informed of an emergency situation which requires suspension of site operations; egression from the work area; emergency responses; and if necessary, site evacuation via horn blasts as defined during site employee briefings. Radio communication may also be used in addition to the horn blasts.

10.3 Evacuation Plan

Although very unlikely, it is possible that a site emergency could necessitate evacuating all personnel from the site. If such a situation should arise, the Site Safety Manager or Field Operations Manager will notify the on-scene U.S. EPA/OEPA representative of this event and the appropriate horn blast will be given for site evacuation. It

is the responsibility of these individuals to evacuate personnel in a calm, controlled fashion.

All available vehicles will be used in the evacuation if they are needed. All personnel will exit the site and be taken to a rendezvous point selected by the Site Safety Manager or Field Operations Manager.

The Field Operations Manager's log of on-site personnel will be used to ensure that all individuals have been evacuated. If someone is missing, the Site Safety Manager or Field Operations Manager will alert the appropriate emergency personnel. Control of personnel at the rendezvous point is the responsibility of the Field Operations Manager or designated assistant. Appropriate emergency contact will be made when evacuation is necessary.

10.4 Cold/Hot Weather Related Emergencies

Cold weather related emergencies include frostbite, trench foot, and hypothermia. Treatment of individuals experiencing any of these injuries will be administered immediately. Employees will be monitored by the SSO and/or Field Operations Manager to detect signs of cold weather related emergencies.

Frostbite occurs when there is actual freezing of the tissues with the attendant mechanical disruption of cell structure. With increasing wind velocity, heat loss is greater and frostbite will occur more rapidly. Once started, freezing progresses rapidly. Furthermore, if the skin comes in direct contact with objects whose surface temperature is below freezing point, frostbite may develop in spite of warm environmental temperatures. The first warning of frostbite is often a sharp, pricking sensation. However, cold itself

produces numbness and anesthesia which may permit serious freezing to develop without the warning of acute discomfort. Injury produced by frostbite may range from simple superficial injury with redness of the skin, transient anesthesia and superficial bullae to deep tissue freezing with persisting ischemia, thrombosis, deep cyanosis, and gangrene.

If detected, individuals with frostbite will be transported to an emergency facility.

Trench foot or immersion foot may be caused by long continuous exposure to cold without freezing, combined with persistent dampness or actual immersion in water. This condition is due to persistent local tissue anoxia, combined with mild or severe cold, with a resultant injury to the capillary walls. Edema, tingling, itching, and severe pain occur and may be followed by blistering, superficial skin necrosis, and ulceration.

If trench foot is detected, the individual will be placed in blankets and moved to a warm, dry location. If symptoms persist, the individual will get medical attention.

General hypothermia is an extreme acute problem resulting from prolonged exposure to cold and heat loss. If an individual becomes fatigued during physical activity, he will be more prone to heat loss, and as exhaustion approaches, the vasoconstrictor mechanism is overpowered; then sudden vasodilatation occurs with a resultant rapid loss of heat, and critical cooling ensues. Sedative drugs and alcohol increase the danger of hypothermia.

If hypothermia is detected the individual will be wrapped with blankets and transported immediately to an emergency facility.

Heat related emergencies can range from heat cramps to life threatening heatstroke. Treatment of individuals suffering from any of these injuries will be administered immediately. Employees will be monitored by the Site Safety Manager to detect signs of heat related emergencies.

Heat rash (prickly heat) may be caused by unrelieved exposure to hot and humid air as may occur in warm-moist climatic zones. The orifices of the sweat ducts become plugged due to the swelling of the moist keratin layer of the skin which leads to inflammation of the glands. There are tiny red vesicles visible in the affected skin area and, if the affected area is extensive, sweating can be substantially impaired. As a consequence heat rash not only is a nuisance because of the discomfort it causes but also can greatly diminish the worker's capacity to tolerate heat.

Heat cramps may occur after prolonged exposure to heat with profuse perspiration and inadequate replacement of salt. The signs and symptoms of heat cramps consist of spasm and pain in the muscles of the abdomen and extremities. Albuminuria may be a transient finding.

Heat cramps will be treated by administration of water, removal of the individual to a cool, shaded location, and rest.

Heat exhaustion may result from physical exertion in a hot environment when vasomotor control and cardiac output are inadequate to meet the increased demand placed upon them by peripheral vasodilatation or the plasma volume is reduced by dehydration. Signs and symptoms of heat exhaustion may include palor, lassitude, dizziness, syncope, profuse sweating, and cool moist skin. There may or may not also be a mild hyperthermia.

Heat exhaustion will be treated by elevating the individual's feet, transporting the individual to a cool, shaded area, administering of water, applying of cool water compresses, and transporting the individual to an emergency facility if individual response is not rapid and full.

Heat stroke is a serious medical condition. An important predisposing factor is excessive physical exertion. Signs and symptoms may include dizziness, nausea, severe headache, hot dry skin because of cessation of sweating, very high body temperature (usually 106°F and rising), confusion, collapse, delirium, and coma. Often circulation is also compromised to the point of shock. If cooling of the victim's body is not started immediately, irreversible damage to vital organs may develop, leading to death.

Heatstroke will be treated as a true medical emergency. An ambulance will be called as soon as an individual has been diagnosed as suffering from heatstroke. An immediate attempt will be made to decrease the body temperature as rapidly as possible by applying cold wet sheets or compresses and placing the individual supine, feet elevated, in the decontamination trailer until transportation to emergency care facilities is possible.

For work limitations and precautions for avoiding heat and cold related injuries see Section 8.1.2 of the HSP.

10.5 Chemical Exposure

Exposure to chemicals can be divided into two categories:

- Injuries from direct contact, such as acid burns or inhalation of toxic chemicals.

- Potential injury due to gross contamination of clothing or equipment.

For the inhaled contaminant, treatment can only be provided by qualified physicians. If the contaminant is on the skin or in the eyes, immediate measures must be taken to counteract the substance's effect. First aid treatment usually consists of flooding the affected area with water; however, for a select few chemicals, water may cause more severe problems.

When protective clothing is grossly contaminated, contaminants may be transferred to treatment personnel or the wearer and cause injuries. Unless severe medical problems will be aggravated by splashing water, the protective clothing should be washed off as rapidly as possible and carefully removed. Portable eye washes and potable water will be available to provide a means of flushing and washing such contamination.

If the injury to the worker results from a chemical splash or uncontrolled release, the following first aid procedures are to be instituted:

- Eye Exposure - If contaminated solids or liquids get into the eyes, wash eyes immediately at the emergency eyewash station using large amounts of water and lifting the lower and upper lids occasionally. Obtain medical attention immediately. Contact lenses will not be worn when working on the site.

- Skin Exposure - If contaminated solids or liquids get on the skin, promptly wash the contaminated skin using soap or mild detergent and water. If

severely contaminated material penetrates through the clothing, remove the clothing immediately and wash the skin using soap or mild detergent and water.

Obtain medical attention immediately when exposed to such material.

- Breathing - If a person breathes in large amounts of contaminants, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration immediately. Keep the affected person warm and at rest. Obtain medical attention as soon as possible. Certified CPR personnel will be on-site at all times during field activities.

- Swallowing - When contaminants have been swallowed and the person is conscious. Attempt to obtain information to aid in identifying the substance swallowed from the person. Contact the poison control center immediately. The poison control center may direct responder to induce vomiting. Do not induce vomiting if: (1) The person is unconscious or semiconscious, or having a convulsions; (2) if a strong corrosive has been swallowed; or (3) if a petroleum product has been swallowed. Vomiting is best induced by administering one tablespoon of syrup of ipecac. Transport the person to the hospital and monitor the airway constantly.

10.6 Fires

Fire extinguishers (type ABC) will be available on site. If a small localized fire breaks out, fire extinguishers will

be used to bring the occurrence under control. If necessary and feasible, a fire blanket, soil, or other inert materials will be placed on the burning area to extinguish the flames and minimize the potential for spreading. If appropriate, local fire-fighting authorities will be contacted for assistance.

If an uncontrolled fire develops releasing potentially toxic gases, on-site personnel and the public in the immediate vicinity will be evacuated. Only personnel trained in fire fighting and outfitted with the proper protective equipment will be allowed in the immediate fire area. The Field Operations Manager or his designated assistant will alert local fire-fighting companies.

10.7 Soils

Handling procedures have been developed to limit potential problems with material spillage. In the event of a spill at the site, the area will be isolated from traffic patterns by the Site Safety Manager or Field Operations Manager. Liquid spills will be solidified with absorbent material and loaded with a front-end loader or other means into a truck for on-site storage. If the spill involves a direct or potential release from the site, local, state, or federal agencies will be notified as appropriate to protect the public.

10.8 Unusual Objects or Events

Although highly unlikely, unusual objects (e.g., buried pressurized, gas cylinders, bulging drums, fuming containers) could be encountered during boring and sampling operations. When such objects are encountered, the Field Operations

Manager will halt operations and notify the Site Safety Manager. The Field Operations Manager or Project Manager will consult the U.S. EPA and the OEPA Project Manager to decide on the next course of action.

10.9 Emergency Personnel

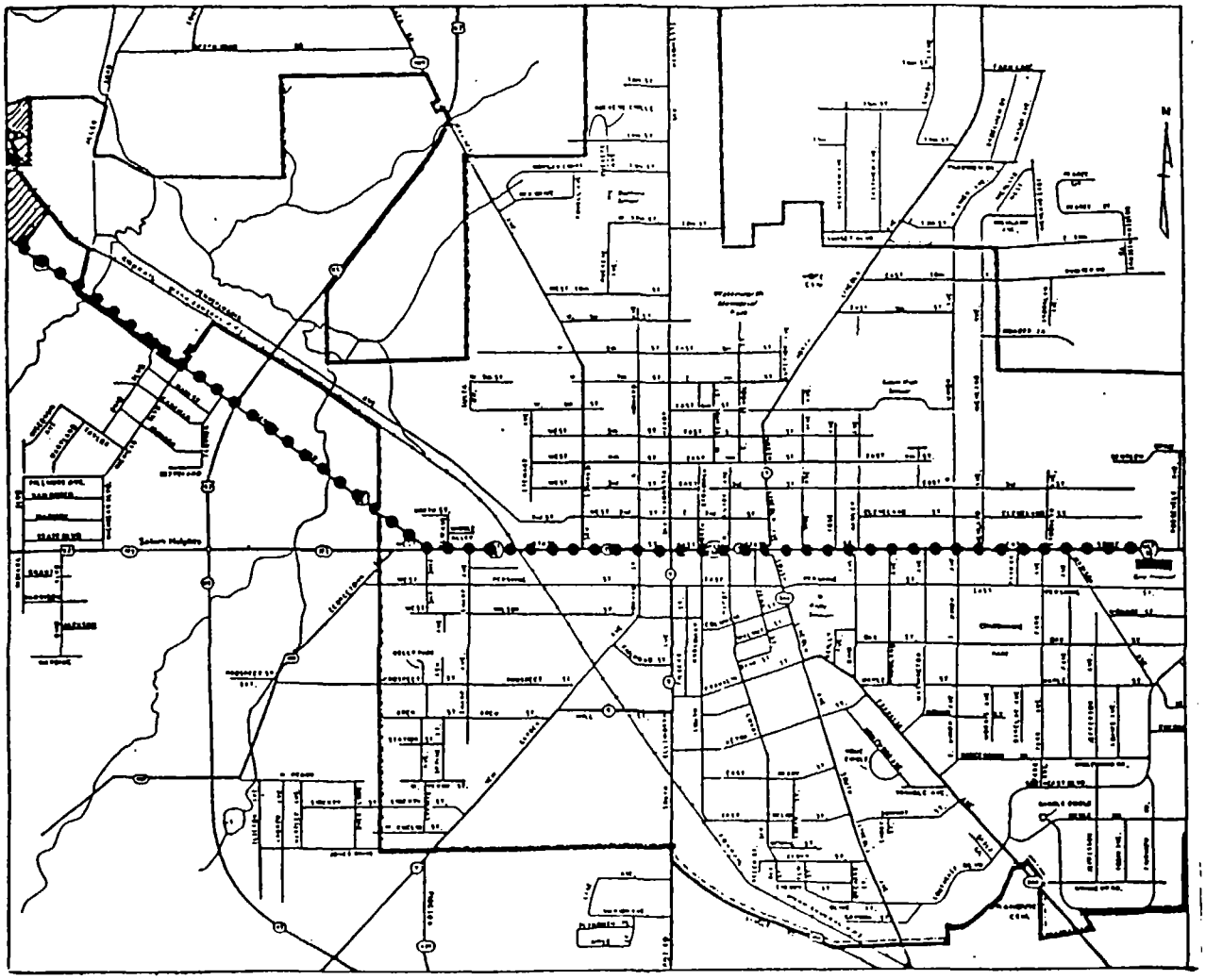
In the event of an emergency, access to the Site will be granted to emergency personnel. Ambulance/Emergency Medical Technical personnel will suit up in coveralls, tyveks, and respirators (their own, respirators at site will not be provided) before entering contaminated areas. Firemen will suit up in basic personnel protective equipment before proceeding to the emergency occurrence. In the case of an emergency in which time is critical, Health and Safety requirements may be waived.

10.10 Emergency Contacts

The closest emergency care service is the Salem Community Hospital which is located approximately 2 3/4 miles from the site. The preferred route to the Hospital is shown in Figure 10-1.

Should any emergency situation or other unplanned occurrence require outside services, the appropriate contacts from the following list will be made:

Agency	Address	Telephone No.
Fire	Salem Twp. Western Section Fire Dept.	(216) 222-1234



LEGEND

RUETGERS - NEASE SITE

ROUTE TO HOSPITAL

FIGURE HSP 10 - 1

ROUTE TO SALEM COMMUNITY HOSPITAL

SCALE

0 1/2 1/4 MILE

REVISED 10.86

ERM - Midwest, Inc.

Agency	Address	Telephone No.
Ambulance	Advanced Gold Cross Ambulance 383 N. Lincoln Salem, OH	(216) 337-3441
Poison Control Center	Mahoning Valley St. Elizabeth's Med.Cntr. 1044 Belmont Ave. Youngstown, OH	(216) 764-2222
Hospital	Salem Community Hospital 1995 E. State Street Salem, OH	(216) 332-7166
Police	Salem Township Western Section	(216) 337-8112
U.S. EPA (Ms. Amy Blumberg)	Chicago, IL	(312) 886-7341
OEPA (Ms. Susan MacMillan)	Twinsburg, OH	(216) 425-9171
Mr. Steven Foard Project Coordinator	State College, PA	(814) 238-2424
ERM-Midwest	Columbus, OH	(614) 433-7900

11.0 FIELD DOCUMENTATION OF HEALTH AND SAFETY PROCEDURES

The following forms are described in this section and can be found in Appendix B:

1. Plan Acceptance Form.
2. Health Certification Form.
3. Accident Report Form.
4. Accident Investigation Form (by SSO).
5. Respiratory Issuance Form.
6. Hearing Protection Issuance Form.
7. Sampling Equipment Calibration Form.
8. Sampling Data Form.
9. Air Monitoring Form.

The Plan Acceptance Form will be completed by all personnel working at the Site. The Accident Report Form will be completed by the Field Operations Manager or Site Safety Manager and the Accident Investigation form will be completed by the Site Safety Manager in the event that an accident occurs. The Air Monitoring Form will contain the OVA monitoring records, and the Sampling Data form will contain the records for the detector tube air and other quantifying samples from the work-zone. The respirator and hearing protection issuance forms contain information concerning the equipment issued to the personnel and will be completed by either the Site Safety Manager or the Health and Safety Officer. The Health Certification Form documents the

physician's approval of the individual for use of a respirator, hearing protection, and general health.

All completed forms will be returned to the Health & Safety Officer. Additionally, all forms and records generated as required by the HSP will be organized and stored in accordance with the guidelines contained in the Data Management Plan, as required under Federal Privacy Act, 504 Civil Rights Handicapped & Disabled Regulations & OSHA 29 CFR 1910.20 Recordkeeping.

12.0 MEDICAL SURVEILLANCE PROGRAM

To safeguard the health of response personnel, a medical surveillance program has been established.

12.1 Background

Prior to commencing work at hazardous waste sites, ERM-Midwest employees must undergo a thorough physical examination if not performed within the preceding 12 months. At present, they are conducted by Urgent Medical Care in Columbus, Ohio as required by ERM-Midwest's Medical Surveillance Program. Pre-site investigation medical examinations establish each individual's state of health, provide baseline physiological and psychological data, and assess the individual's ability to cope with the stress of hazardous waste site investigations.

Annual medical examinations are conducted to assess the health status of individual workers as to their fitness for continued assignments at hazardous waste sites. Periodic or follow-up medical evaluation of individual workers will be conducted as deemed necessary by ERM-Midwest's Health & Safety Officer and Medical Advisor per exposure to various environmental factors. The content and frequency of these examinations is influenced by the kinds of work and exposures encountered by each individual. The frequency of these follow-up examinations is established by the Health and Safety Officer in conjunction with the Medical Advisor.

12.2 Physical Examination

Each individual receives a thorough physical examination which includes an evaluation of blood counts and blood chemistry to assess blood-forming, kidney, liver, and

metabolic functions. The examining physician, or his/her immediate supervisor, will be accredited by the American Occupational Medical Association (AOMA). In addition, the physician and/or appropriate medical staff professional(s) will be certified by the Council of Accreditation in Occupational Hearing Conservation (29 CFR 1910.95 (g)(3) Noise Standard) in order to perform and interpret audiograms/audiometric tests. Pulmonary function tests will be performed by certified spirometrists who have taken the required NIOSH spirometry course. X-rays will be read by a technician with a B license. Specifically, the physical examination consists of the following elements:

Physical Examination

1. Review of personal and family health history

2. Nutritional evaluation

3. Cardiovascular risk analysis

4. Daily pressure questionnaire

5. Complete physical examination

6. History of occupational exposures

7. Eye tests:
   - Near and distant vision
   - Color vision
   - Peripheral vision
   - Depth perception

8. Hearing test (Audiometric screening)

9. Electrocardiogram, 12 lead

10. Pulmonary function study (functional lung capacity)

11. Gastrointestinal System

12. Genitourinary Disorders

13. Chest X-Ray

Laboratory Studies

1. Hematology
   - Red blood count
   - White blood count

2. Differential
   - Polys
   - Lymphs
   - Mono
   - Eos
   - Baso

3. Blood chemistries (26)
   - Calcium
   - BUN/Creat ratio
   - Creatinine
   - Total protein
   - Total Bilirubin
   - Alk. Phosphatase
   - Iron
   - Potassium
   - Triglycerides
   - Phosphorus
   - Uric Acid
   - Albumin
   - Alb/glob ratio
   - Direct Bilirubin
   - LDH
   - Total lipids
   - Chloride
   - T4
   - BUN
   - Glucose
   - Globulin
   - SGOT
   - SGPT
   - Cholesterol
   - Sodium
   - GGTP

4. Urogram:
   - Acetone
   - Glucose
   - Albumin
   - Specific gravity
   - Blood
   - pH

5. Serology - IgE
   - IgG

6. Papanicolaou (PAP) test for cervical cancer (female staff)

Summary reports of the examinations are sent to each employee at their home address and to ERM-Midwest's Personnel Department and kept on file in the individual's personnel file. Full results of each physical are sent to each employee's personal physician upon written request and/or authorization of employee/employer. The original reports are kept at Urgent Medical Care.

13.0 PERSONNEL TRAINING

Prior to beginning field work at the Salem Site, all individuals required to work in the exclusion zone and contaminant reduction zone will have completed the required 40-hour Hazardous Material & Safety training as required by 29 CFR 1910.120 for working with hazardous materials at a hazardous waste site, and an 8-hour Refresher Course or an 8-hour Supervisor Training Course, if applicable. In addition, each staff member will have completed a Hearing Conservation Program and a review of the Health & Safety Site Plan.

13.1 Training Course

Key elements of the training program include:

1. Introduction to hazardous waste site work
   - Legal considerations
   - Overview of work duties
   - 1910.120 Regulation

2. Working with hazardous materials
   - Physical/chemical properties
   - Toxicity
   - Hazards
   - Reference books
   - Sampling techniques
   - Handling, packaging, labeling
   - MSDS
   - Fire Extinguishers

3. Survey instruments
   - General monitoring equipment
     Combustible gas indicator
     Toxic gas indicator

Organic vapor analyzers
- Specific survey instruments
  Oxygen meters
  Colorimetric tubes

4. Respiratory protection
   - Respiratory hazards
   - Respirators and their selection
   - Air-purifying respirators
   - Air-supplying respirators (Self Contained Breathing Apparatus: SCBA)
   - Fit-testing of respirators

5. Protective clothing
   - Chemical resistance
   - Material selection
   - Types of clothing
   - Donning and doffing

6. Medical Surveillance Program & Record Keeping

7. Safety Planning, Principles & Work Practices

8. Engineering Controls
   - Various types of control methods

9. Site entrance and decontamination
   - General precautions
   - Personal hygiene
   - Operations
   - Emergency medical care
   - Weather conditions
   - Air monitoring
   - Levels of protection:
     Level A
     Level B

Level C
Level D

10. Drum Handling & Sampling Techniques
    - Regulations & manifesting
    - Opening drums & containers
    - Material for handling electrical equipment
    - Shock sensitive waste
    - Laboratory waste packs
    - Shipping and transport

11. Site work zones
    - Exclusion zone
    - Contamination reduction zone
    - Support area

12. Decontamination
    - Extent required
    - Equipment/solutions
    - Decontamination during medical emergencies
    - Equipment decontamination

13. Training Introduction to Sampling & Chain-of-Custody
    - Sampling procedures
    - Sampling equipment and methods
    - Documentation/Chain-of-Custody
    - Packaging, marketing, labeling & shipping

14. Emergency Response and Area Location Requirements
    - General
    - Site Emergency procedures
    - Off-site emergency procedures
    - HazMat Teams
    - Determination of Hazardous Site Logistics
    - Community Relations

The training curriculum consists of the following:

1. Overview of Hazardous Waste Operation and Response Regulations (OSHA 1910.120)

2. Development of Site Specific Health and Safety Plans

3. Criteria for selection of Personal Protective Equipment and Medical Surveillance Program

4. Site Security/Risk Management/Assessment

5. Medical and Employee Training Record Keeping

6. Management of Hazardous Waste Site Logistics

7. Health and Safety Guidelines for the Supervisor

13.4 Hearing Conservation

Training as deemed necessary by OSHA 1910.95 for Noise Exposure consists of the following:

1. The effects of noise on hearing.

2. The purpose of hearing protectors, the advantages, disadvantages, and attenuation of various types of protectors. Also, instructions on their selection, fitting, and use.

3. The purpose of audiometric testing, and an explanation of the test procedures.

4. Employer/employee responsibilities for record keeping.

13.5 Health and Safety Site Plan

All individuals will be taught the location and criteria for health and safety precautions at the Salem Site before actual work activities begin.

Health and Safety Plan Appendix A: The Health Effects and the Response to Exposure to Site Specific Compounds

Submitted by

Ruetgers-Nease
Chemical Company, Inc.
201 Struble Rd.
State College, Pennsylvania 16801

APPENDIX A: SYMPTOMS OF OVER EXPOSURE AND FIRST AID TREATMENT FOR POSSIBLE HAZARDOUS:

Volatile Organic Compounds

Non-Volatile Organic Compounds

Symptoms of Over Exposure and First Aid Treatment for Possible Hazardous

Volatile Organic Compounds

COMPOUND - 1,1 Dichloroethene (Syn: 1,1-Dichloroethylene, acetylene dichloride)

Exposure Route - Inhalation, ingestion, absorption, consumption

Symptoms - Acute: Depression, skin irritation, drowsiness, unconsciousness, liver and kidney damage,
Chronic: Headache, mental confusion and fatigue, can cause lung edema.

First Aid - Eyes: Irrigate eyes immediately
Skin: Wash with soap promptly
Breath: Artificial respirator and fresh air
Swallow: Seek medical attention immediately

Target Organ - Skin, liver, kidney

COMPOUND - 1,2 Dichlorethene

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute: Irritant eyes, respiratory system, depressant.
Chronic: Respiratory, neurological disorders

First Aid - Eyes: Irrigate immediately
Skin: Wash with soap promptly
Breath: Artificial respirator and fresh air
Swallow: Seek medical attention immediately

COMPOUND - Chloroform (Syn: trichloromethane)

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute: Dizziness, mental dullness, nausea, headache, fatigue, anesthesia, hepatomegoly, eye, skin irritant
Chronic: Neurotoxic effects, cardiac, liver, kidney damage. Group B2 suspected carcinogen

Note: Individuals who consume alcohol seem to be affected sooner and more severely from chloroform exposure.

First Aid - Eyes: Irrigate immediately
Skin: Wash with soap promptly
Breath: Artificial respirator and fresh air
Swallow: Seek medical attention immediately

Target Organ - Liver, kidney, heart, eyes, skin, Central Nervous System (CNS)*, lungs*

* Exposures should be minimized due to the structural similarity to the carcinogenic chloroethanes.

COMPOUND - 1,1,1 Trichoroethane (Syn: Methyl Chloroform)

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute:	Headache, lassitude, depression, irritated eyes, tightness of breath, dermatitis.
Chronic:	Mild conjunctivitis, incoordination increased reaction time, unconsciousness, death. Cardiac arrhythmias, may cause affects on blood pressure. Congenital birth defects and spontaneous abortions.

First Aid - Eyes:	Irrigate immediately
Skin:	Wash with soap promptly
Breath:	Artificial respirator, fresh air
Swallow:	Seek medical attention immediately

Target Organ - Reproduction, liver, kidney, respiratory, cardiac, & CNS

COMPOUND - 1,2 Dichloropropane (Syn: Propylene Dichloride)

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute:	Dermatitis, irritation of eyes, tightness of breath.
Chronic:	CNS narcosis, liver and kidney damage

First Aid - Eyes:	Irrigate immediately
Skin:	Wash surface
Breath:	Fresh air, artificial respirator
Swallow:	Seek medical attention immediately

Target Organ - CNS, liver, kidney, skin, eyes, respiratory system

COMPOUND - 1,3 Dichloropropene (Syn: 1,3-Dichloropropylene)

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute:	Respiratory and eye irritant, headache, nausea, dermatitis.
Chronic:	Liver and kidney damage. Suspect carcinogen and mutagen

First Aid - Eyes:	Irrigate immediately
Skin:	Wash surface
Breath:	Fresh air, artificial respirator
Swallow:	Seek medical attention immediately

Target Organ - Suspected carcinogen and mutagen causes liver and kidney damage.

COMPOUND - Trichloroethene (Syn: Trichloroethylene, ethylene-trichloride,tridene)

Exposure Route - Ingestion, inhalation, absorption

Symptoms - Acute:	Nausea, vomit, abdomen pain, tremors enlargement and tenderness of liver, headache, vertigo and visual disturbances.
Chronic:	Dermatitis, liver kidney, paresthesias, cancer, tumors, and CNS depressant.

First Aid - Eyes:	Irrigate immediately
Skin:	Wash with soap and water promptly
Breath:	Artificial respirator, fresh air
Swallow:	Seek medical attention immediately

Target Organ - CNS, liver, kidney, gastrointestinal, skin, respiratory system

COMPOUND - Benzene

Exposure Route - Ingestion, inhalation, absorption

Symptoms - Acute:	Irritant to skin, nose, eyes, and upper respiratory tract. Pulmonary edema and hemorrhage. Erythema, vesiculation and dermatities. Headache, nausea, giddiness.
Chronic:	Leukemia, bone marrow depressive, abdominal pain, chromosomal aberrations, bronchopneumonia, leucopenia, hemato-lympho reticular neoplasies (leukemias) and mammary carcinomas, known human carcinogen
First Aid - Eyes:	Irrigate immediately
Skin:	Wash with soap and water promptly
Breath:	Fresh air and artificial respirator
Swallow:	Seek medical attention immediately

Target Organ -Blood Mutagen, CNS, skin, bone marrow, eyes, respiratory system

COMPOUND - 1,1,2,2- Tetrachloroethane (Syn: Acetylene Tetrachloride)

Exposure Route - Ingestion, inhalation

Symptoms - Acute:	Nausea, vomit, abdominal pain, tremore fingers, jaundice, enlargement and tenderness of liver.
Chronic:	Dermatitis, liver, kidney, paresthesias and CNS.
First Aid - Eyes:	Irrigate immediately
Skin:	Wash with soap and water promptly
Breath:	Artificial respirator and fresh air
Swallow:	Seek medical attention immediately

Target Organ -CNS, liver, kidney

COMPOUND - Tetrachloroethene (Syn: Tetrachlorethylene, Perchloroethylene)

Exposure Route - Inhalation, ingestion, absorption

Symptoms - Acute:	Irritates eyes, skin, nose, and throat, nausea, flush face-neck, vertigo, dizziness, incoordination, headache, increased perspiration, staggery gait, slow mental ability.
Chronic:	Cardiac arrhythmias and renal injury, kidney, CNS, upper respiratory disease, depressive, suspected carcinogen
First Aid - Eyes:	Irrigate immediately
Skin:	Wash with soap and water promptly
Breath:	Artificial respirator, fresh air
Swallow:	Seek medical attention immediately

Target Organ - Liver, kidney, eyes, upper respiratory system, CNS

<u>COMPOUND - Toluene</u> (Syn: Phenyl methane, methyl benzene)

Exposure Route - Inhalation, ingestion, absorption

Symptoms - Acute:	Fatigue, weakness, confusion, dizziness, headache, diolated pupils, nervous, dermatitis, and insomnia
Chronic:	CNS, kidneys, and skin and neurological disorders
First Aid - Eyes:	Irrigate immediately
Skin:	Wash with soap and water promptly
Breath:	Artificial respirator, fresh air
Swallow:	Seek medical attention immediately

Target Organ - CNS, liver, kidneys, skin

<u>COMPOUND - Chlorobenzene</u> (Syn: Monochlorobenzene; chlorobenyl, phenol chloride, MCB)

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute:	Can cause somnolence, loss of consciousness, twitchings of extremities, cyanosis, deep rapid respiratory, burgundy red urine, and small irregular pulse. Irritant to eyes, nose, drowsiness incoordination and skin and conjunctivitis.
Chronic:	Kidney, liver, respiratory, skin

burns, damage, eyes, nuerological disorders/damages.

First Aid - Eyes: Irrigate immediately
Skin: wash with soap and water promptly
Breath: artificial respirator, fresh air
Swallow: **Seek medical attention immediately**

Target Organ - Respiratory system, eyes, skin, CNS, liver

<u>COMPOUND - Ethylbenzene</u> (Sny: Phenylethane, Ethylbenzol)

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute: Irritant and sensitive eyes, nose, throat, and skin dermatitis, upper respiratory tract, nose mouth, narcotic, and coma
Chronic: Upper respiratory disease with allergic reactions

First Aid - Eyes: Irrigate immediately
Skin: Water wash promptly
Breath: Artificial respirator
Swallow: **Seek medical attention immediately**

Target Organ -Eyes, immune system, upper respiratory system, skin, CNS

<u>COMPOUND - o, m, p-ylene</u> (Syn: 1,2 Dimethyl-benzene, 1,3 Dimethyl-benzene, 1,4 Dimethyl-benzene)

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute: Dizziness, excitement, drowsiness, incoordination, staggering gait, irritation to eyes, nose, throat, corneal vacuolization, anorexia, nausea, vomit, abdominal pain and pulmonary edema.
Chronic: Liver, kidney, lung disfunctions, neurotoxic effects.

First Air - Eyes: Irrigate immediately
Skin: Water wash promptly
Breath: Artificial respirator
Swallow: **Seek medical attention immediately**

Target Organ - CNS, eyes, gastrointentinal tract, blood liver, kidneys, skin, respiratory

COMPOUND - o, m, -Dichlorobenzene

Exposure Route - Inhalation, absorption, ingestion

Symptoms - Acute:	Irritant to nose, eyes, conjuction and mucus membranes of upper respiratory tract.
Chronic:	Liver, kidney and lung damage and skin blistering, drowsiness, incoordination, unconsciousness experimental carcinogen.
First Aid - Eyes:	Irrigate immediately
Skin:	Wash with soap and water promptly
Breath:	Artificial respirator
Swallow:	Seek medical attention immediately

Target Organ - Liver, kidney, skin, eyes, respiratory system

COMPOUND - p Dichlorobenzene (1,4 dichlorobenzene)

Exposure Route - Inhalation, ingestion, absorption

Symptoms - Acute:	Headache, eye irritant, swelling periorbital, profuse rhinitis, anorexia, nausea, vomit, low weight, jaundice, and circulation problems
Chronic:	Experimental Carcinogen and Mutagen, liver damage
First Aid - Eyes:	Irrigate immediately
Skin:	Soap wash
Breath:	Artificial respirator
Swallow:	Seek medical attention immediately

Target Organ - Liver, skin, bladder, lungs, GI tract

NON-VOLATILE ORGANIC COMPOUNDS:

COMPOUND - Methoxychlor (Syn: 2,2-bis(p-methoxy-phenyl, 1,1,1 trichloroethane)

Exposure Route - Inhalation, ingestion, absorption

Symptoms - Acute:	Irritatable, trembling, convulsions
Chronic:	Kidney and liver damage
First Aid - Eyes:	Irrigate immediately
Skin:	Wash skin
Breath:	Artificial respirator
Swallow:	**Seek medical attention immediately**

Target Organ - unknown

COMPOUND - Mirex

Exposure Route - Inhalation, ingestion, absorption

Symptoms - Acute:	Nausea, abdominal pain, vomiting
Chronic:	CNS depression, reproductive effects, weight loss, anorexia
First Aid - Eyes:	Irrigate with water
Skin:	Wash with soap and water promptly
Breath:	Fresh air, artificial respirator
Swallow:	**Seek medical attention immediately**

Target Organ - Reproductive System

COMPOUND - 3,4 Dichloronitrobenzene

Exposure Route - Inhalation, Ingestion, Absorption

Symptoms - Acute:	Headache, nausea, depression, tightness of chest
Chronic:	No information available
First Aid - Eyes:	Irrigate with water
Skin:	Wash with soap and water promptly
Breath:	Fresh air, aritificial respirator
Swallow:	**Seek medical attention immediately**

Target Organ - Blood, kidneys, liver, bone marrow and nervous system

COMPOUND - Diphenyl Sulfone (Syn: Phenyl Sulfone, Benzene, 1,1'-sulfonyl bis-(9cl)

Exposure Route -Ospare Inhalation, ingestion, absorption

Symptoms - Acute:	**No information available***
Chronic:	**No information available***

First Aid - Eyes:	Irrigate with water
Skin:	Wash with soap and water promptly
Breath:	Fresh air, artificial respirator
Swallow:	Seek medical attention immediately

Target Organ - Skin, liver, kidney and CNS

* EPA Chemical #399500 - out of date pesticide
  CAS#127-63-9
  Tox Net, RTECS,IRIS, and Hazardous Substance Data Bases searched

Health and Safety Plan Appendix B: Health and Safety Forms

Submitted by

Ruetgers-Nease
Chemical Company, Inc.
201 Struble Rd.
State College, Pennsylvania 16801

APPENDIX B: FIELD DOCUMENTATION

Plan Acceptance Form

Health Certification Form

Accident Report Form

Accident Investigation Form (by SSO)

Respiratory Issuance Form

Hearing Protection Issuance Form

Sampling Equipment Calibration Form

Sampling Data Form

Air Monitoring Form

HEALTH AND SAFETY PLAN ACCEPTANCE FORM

INSTRUCTION: This form is to be completed by each person to work on the Salem Site Remedial Investigation/Feasibility Study and returned to the Health and Safety Officer.

I represent that I have read and understand the contents of the Salem Site Health and Safety Plan and agree to perform my work in accordance with it.

_____
Signed

_____
Print Name

_____
Company

_____
Date

HEALTH CERTIFICATION FORM

RUETGERS-NEASE SALEM SITE RI/FS

The Salem Site Remedial Investigation/Feasibility Study may expose the field personnel to hazardous compounds during the project.

TO: Health and Safety Officer

ERM-Midwest, Inc.

450 W. Wilson Bridge Road

Worthington, OH 43085

(614) 433-7900

FROM:

From my examination I do not see any reason to preclude from performance of his/her normal work activities, including those that may require the use of protective clothing and respiratory equipment during this project.

Exceptions:

(Examining Physician) (Date)

ACCIDENT INVESTIGATION INFORMATION
RUETGERS-NEASE SALEM SITE RI/FS

To be completed by Site Health and Safety Manager

Investigator:_____Date:_____Time:_____

Date and Location of Accident_____

Reported by:_____Date/Time:_____

DESCRIPTION: (Narrate what happened in detail - use supplement sheets, if necessary)

_____

_____

_____

_____

_____

_____

_____

Identify to Whom or What:

_____

_____

Supervisor of Area where occurrance took place:_____

Supervisor of employee(s) or object(s) involved:_____

_____

Where were supervisors (each, if different) when indident occurred:_____

_____

List witnesses (name, badge, phone, mail point, supervisor) and relate where each was and what each was doing at the time of occurrance:

_____

_____

_____

_____

Describe cause of incident (identify if suspected or apparent):_____

_____

_____

SALEM SITE
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
ACCIDENT REPORT FORM

SUPERVISOR'S REPORT OF ACCIDENT

DO NOT USE FOR MOTOR VEHICLE OR AIRCRAFT ACCIDENTS

TO

FROM

TELEPHONE(include area code)

NAME OF INJURED OR ILL EMPLOYEE

DATE OF ACCIDENT TIME OF ACCIDENT EXACT LOCATION OF ACCIDENT

NARRATIVE DESCRIPTION OF ACCIDENT

NATURE OF ILLNESS OR INJURY AND PART OF BODY INVOLVED

LOST TIME
YES NO

PROBABLE DISABILITY (Circle One)

FATAL	LOST WORK DAY WITH DAYS AWAY FROM WORK	LOST WORK DAY WITH DAYS OF RESTRICTED WORK	NO LOST WORK DAY	FIRST AID ONLY

CORRECTIVE ACTION WHICH REMAINS TO BE TAKEN (By whom and by when)

List of Witnesses and their statements

NAME OF SUPERVISOR TITLE

SIGNATURE DATE

RESPIRATOR ISSUANCE FORM
RUETGERS-NEASE SALEM SITE RI/FS

I. Employee/User

Name: _____ Date: _____

S.S. #: _____

Address: _____ Phone: _____

_____ Employer: _____

_____ Supervisor: _____

Sex: _____ Race: _____ DOB: _____

Type of Work Being Done: _____

Type of Chemical Exposure: _____

II. Medical/Occupational Health

1. Pulmonary function test approved. Yes ___ No ___

Date Tested: _____ By: _____

2. Fit Test. Yes ___ No ___ Type performed: _____

III. Industrial Hygiene

1. Received respiratory training as deemed by OSHA Std. 1910.134.

Date: _____ By: _____

2. Any restrictions or special precautions. (Ex. beard, long side burns, etc.)

3. Type of respirator issued and by whom: _____

_____

HEARING PROTECTION ISSUANCE FORM
RUETGERS-NEASE SALEM SITE RI/FS

I. Employee/User

Name: _____ Date: _____

S.S. #: _____

Address: _____ Phone: _____

_____ Employer: _____

_____ Address: _____

_____ _____

Supervisor: _____

Sex: _____ Race: _____ DOB: _____

II. Medical/Occupational Health

1. Audiometric exam results/restrictions. _____

_____

_____

2. Type of protection employee/user can use._____

3. Additional comments: _____

_____

III. Industrial Hygiene

1. Has Industrial Hygiene performed any tests (noise) for employee/user under hearing conservation program at any location.

   a. Yes ___ No ___

   b. Where: _____ Date: _____

2. Type of protection issued: _____

3. Employee/User trained as deemed by OSHA 1910.95 Std.

   a. Yes ___ No ___

   b. By Whom: _____ Date: _____

SAMPLING EQUIPME[illegible]RATION FORM

RUTGERS-NEASE SALEM SITE RI/FS

1 DATE/TIME	2 TYPE PUMP AND SN	3 LOCATION T/ALT	4 VOLTAGE CHECKED	5. FLOWRATE MEASURED	6. METHOD OF CALIBRATION	7. PRE-SURVEY	8. POST SURVEY	9. I.H. INITIALS	10 REMARKS/CALCULATIONS/CORRECTIONS

SAMPLING DATA FORM
RUETGERS-NEASE SALEM SITE RI/FS

1. ESTABLISHMENT

INSPECTION NO.
2. CONSULTANT NO.

3. REPORT NO

N PERFORMING SAMPLING

5. SAMPLING DATE

6. WEATHER CONDITIONS
TEMP HUM. WIND

LOYEE NAME

8. JOB TITLE

9. FREQ OF EXPOSURE

ADDRESS

11. CITY

12. PHONE NUMBER

PPE TYPE & EFFECTIVENESS

14. PHOTO
Y N
ROLL NO
FRAME NO

JOB DESCRIPTION, OPERATION, WORK LOCATIONS, ENG. CONTROLS, VENTILATION

CONTD

PUMP NO. SAMPLING DATA DATE SUBMITTED TO LAB

SAMPLE TYPE/MEDIA						
SAMPLE NUMBER						
TIME ON						
TIME OFF						
AL TIME (MIN.)						
FLOW RATE ☐ l/min ☐ cc/min						
VOLUME (LITERS)						
NET SAMPLE WGT (MG.)						
LAB SAMPLE NO. →						
SUBSTANCE / RESULTS →						

INTERFERENCES AND L.H. COMMENTS TO LAB

27. SUPPORTING SAMPLES

a. BLANKS

b. BULKS.

c. OTHER

ANALYST'S COMMENTS/ADDITIONAL ANALYSIS

PAGE OF

AIR MONITORING FORM
RUETGERS-NEASE SALEM SITE RI/FS

GENERAL INFORMATION

Name(s):_____ Background:_____

Date:_____ Weather Conditions:_____

Time:_____ _____

Project:_____ _____

Job No.:_____

Estimated Wind Direction:_____

Estimated Wind Speed (i.e., calm, moderate, strong, etc.):_____

Location Where Background Level Was Obtained:_____

OVA EQUIPMENT SETTINGS

Range:_____

Calibration Gas:_____

FIELD ACTIVITIES

TIME	ppm	TIME	ppm	TIME	ppm

Verification of the required minimum three days of actual field experience under the direct supervision of a trained, experienced supervisor will be conducted by ERM-Midwest. Workers who may be exposed to unique or special hazards will be provided with additional training as needed.

13.2 Refresher Courses

An 8-hour refresher training course will be attended annually by individuals who have completed the 40-hour training course.

The training curriculum consists of the following:

1. Revisions in OSHA 1910.120.

2. Review of Personal Protection Equipment and Medical Surveillance.

3. Respiratory Protection

4. Site Assessment

5. Health & Safety Guidelines

6. Decontamination and QA/QC Updates

13.3 Supervisor Training Course

All individuals who will act in a supervisory role on-site will have received the required additional eight hours of training for supervisors as stated in 29 CFR 1910.120.